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FINAL SAMPLING AND ANALYSIS PLAN FOR REMEDIAL INVESTIGATION UXO 1 NAS
CORPUS CHRISTI TX
10/01/2010
TETRA TECH NUS, INC.

Comprehensive Long-term Environmental Action Navy

CONTRACT NUMBER N62467-04-D-0055



Rev. 0
10/22/10

Final Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan) for Remedial Investigation of the Skeet Range

Naval Auxiliary Landing Field Cabaniss
Corpus Christi, Texas

Contract Task Order 0135

October 2010



NAS Jacksonville
Jacksonville, Florida 32212-0030

SAP Worksheet No. 1 -- Title and Approval Page
(UFP-QAPP Manual Section 2.1)

**FINAL SAMPLING AND ANALYSIS PLAN
FOR
MUNITIONS RESPONSE PROGRAM
SKEET RANGE
REMEDIAL INVESTIGATION
NAVAL AUXILIARY LANDING FIELD CABANISS
CORPUS CHRISTI, TEXAS**

OCTOBER 2010

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Prepared under:
Comprehensive Long-Term Environmental Action Navy Contract
No. N62467-04-D-0055
Contract Task Order 0135

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ACRONYMS

bgs	Below ground surface
CA	Corrective Action
CAS	Chemical Abstract Service
CCC	Calibration Check Compound
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-Term Environmental Action Navy
COC	Contaminants of Concern
COD	Coefficient of Determination
CSM	Conceptual Site Model
CTO	Contract Task Order
DDESB	DoD Explosive Safety Board
DFTPP	Decafluorotriphenylphosphine
DoD	Department of Defense
DoE	Department of Energy
DQI	Data Quality Indicators
DQO	Data Quality Objective
DVM	Data Validation Manager
e.g.	For example
EDD	Electronic Data Deliverable
EOD	Explosive Ordnance Disposal
ELAP	Environmental Laboratory Accreditation
ESS	Explosive Safety Submission
FM	Farm-to-Market
FOL	Field Operations Leader
FS	Feasibility Study
FSP	Field Sampling Plan
FTMR	Field Task Modification Request
GC/MS	Gas Chromatograph/Mass Spectrometer
GPS	Global Positioning System
GSA	General Services Administration

HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HDOP	Horizontal Dilution of Precision
HSM	Health and Safety Manager
IDQTF	Intergovernmental Data Quality Task Force
ICV	Initial Calibration Verification
IDW	Investigation-Derived Waste
IRCDQM	Navy Installation Restoration Chemical Data Quality Manual
Katahdin	Katahdin Analytical Services, Inc.
LCS	Laboratory Control Sample
LOD	Limit of Detection
LOQ	Limit of Quantitation
MC	Munitions Constituents
MDL	Method detection limit
MEC	Munitions and Explosives of Concern
mg/kg	Milligram per kilogram
mg/L	Milligram per Liter
mm	Millimeter
MMRP	Military Munitions Response Program
MPC	Measurement Performance Criteria
MRP	Munitions Response Program
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NA	Not Available/Not Applicable
NAD	North American Datum
NALF	Naval Auxiliary Landing Field
NASCC	Naval Air Station Corpus Christi
NAVFAC SE	Naval Facilities Engineering Command Southeast
NCP	National Oil and Hazardous Substances Pollution Contingency Plan
NELAP	National Environmental Laboratory Accreditation Program
NFA	No Further Action
NOSSA	Naval Ordnance Safety and Security Activity
OLF	Outlying Field
OSHA	Occupational Safety and Health Administration
PA	Preliminary Assessment
PAH	Polycyclic Aromatic Hydrocarbon
PAL	Project Action Limit

PCL	Protective Concentration Level
PG	Professional Geologist
PM	Project Manager
PMO	Project Management Office
POC	Point of Contact
QA	Quality Assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
QSM	Quality Systems Manual
RBEL	Risk Based Exposure Limit
RCI	Reactivity, Corrosivity, Ignitability
RF	Response factor
RI	Remedial Investigation
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RSD	Relative Standard Deviation
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SI	Site Inspection
SIM	Selective Ion Monitoring
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SQL	Structured Query Language
SSO	Site Safety Officer
SVOC	Semivolatile Organic Compound
TAL	Target Analyte List
TBD	To Be Determined
TCEQ	Texas Commission on Environmental Quality
TCLP	Toxicity Characteristic Leaching Procedure
TDS	Total Dissolved Solids
TOM	Task Order Manager
TP	Technical Paper
TRRP	Texas Risk Reduction Program
TtNUS	Tetra Tech NUS, Inc.

USEPA	United States Environmental Protection Agency
UFP	Uniform Federal Policy
UXO	Unexploded Ordnance
VOC	Volatile Organic Compound
WWII	World War II

EXECUTIVE SUMMARY

Tetra Tech NUS, Inc. (TtNUS) has prepared this Uniform Federal Policy Sampling and Analysis Plan (UFP-SAP) under the Comprehensive Long-Term Environmental Action Navy (CLEAN) Contract No. N62467-04-D-0055 Contract Task Order (CTO) 135. This plan has been prepared for a Remedial Investigation (RI) at the former Skeet Range located at Naval Auxiliary Landing Field (NALF) Cabaniss, Corpus Christi, Texas.

NALF Cabaniss is an outlying field (OLF) with the current primary role of supporting Naval air training operations originating from Naval Air Station Corpus Christi (NASCC). NALF Cabaniss is located eight miles west of NASCC. The installation occupies 923 acres and was originally constructed with four 5,000-foot runways. Only two runways, oriented in north/south and northwest/southeast directions, are presently active and maintained. The airfield is lighted, to allow for night flight training, and daylight training is also conducted.

The Skeet Range was located in the southeastern corner of the installation, 1,230 feet southeast of Runway 31 and 400 feet north of Oso Creek. A former drainage ditch lies to the west of the former range, while another drainage canal currently intersects the eastern end of the former range area. The area surrounding the former range is covered in vegetation. The former Skeet Range was originally constructed in 1943 and was generally used for small arms qualification and moving target orientation training for Naval aviators, although the range may have also been used for recreational purposes. The Skeet Range was demolished between 1958 and 1964.

A Site Inspection (SI) was conducted in 2008 to determine the presence and approximate lateral extent of munitions constituents (MC) contamination present in surface water, surface soil, and sediment at the Skeet Range. The SI consisted of the collection of surface soil, surface water, and sediment samples; laboratory analysis of surface soil samples, surface water and sediment samples; land surveying of sample locations; and reporting of results. Two soil borings were drilled to determine subsurface lithology and depth to groundwater. Subsurface soil samples were not collected for laboratory analysis. Temporary monitoring wells were installed to collect groundwater samples to determine the groundwater resource classification. However, due to heavy silting of the temporary monitoring wells, groundwater samples were unable to be collected.

Analytical results from the SI indicated that MC (specifically polycyclic aromatic hydrocarbons [PAHs]) were present in surface soil at concentrations exceeding risk based regulatory screening criteria (i.e.,

Texas Risk Reduction Program [TRRP] human health criteria). Analytical results for surface water and sediments were less than the applicable TRRP human health or ecological criteria.

The Department of Defense (DoD) has established the Military Munitions Response Program (MMRP) to address MC and Munitions and Explosives of Concern (MEC) at closed ranges. The DoD is following the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) process for the investigation and remediation of these sites. The Navy is responsible for implementing the MMRP at NALF Cabaniss. Of note, one MEC item, a smoke grenade, was encountered during the SI and so it is possible that MEC may again be encountered during the RI.

The primary objective of this RI is to determine the nature and extent of MC in soil and groundwater at the Skeet Range.

This UFP-SAP has been prepared in accordance with the DoD, Department of Energy (DoE), and United States Environmental Protection Agency (USEPA) Intergovernmental Data Quality Task Force (IDQTF) environmental requirements for federal facilities. To comply with IDQTF requirements, the SAP is presented in the format of 37 standard worksheets as specified in the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP, aka UFP-SAP), Parts 1, 2A, and 2B (USEPA, 2005), along with Navy-developed template to combine the required elements of the UFP-QAPP and a Field Sampling Plan (FSP) to create a complete UFP-SAP.

The information provided in the worksheets was developed based on the results of two project scoping meetings among the planning team, which consists of representatives of the Navy, USEPA Region 6, Texas Commission on Environmental Quality (TCEQ), and TtNUS (see Worksheet No. 9 for attendees). Worksheet No. 10 contains a summary of the site-specific Conceptual Site Model (CSM) for the site. The CSM was used as the basis for the development of the site-specific project Data Quality Objectives (DQOs), which are contained in Worksheet No. 11. The remainder of the worksheets describe the sampling, analytical, and data evaluation procedures including quality requirements.

SAP Worksheet No. 2 -- SAP Identifying Information

Site Name/Number: Former Skeet Range at Naval Auxiliary Landing Field (NALF) Cabaniss, Corpus Christi, Texas
Operable Unit: Not Applicable (NA)
Contractor Name: Tetra Tech NUS, Inc. (TtNUS)
Contract Number: N62467-04-D-0055
Contract Title: Comprehensive Long-Term Environmental Action Navy (CLEAN) IV
Work Assignment Number (optional): Contract Task Order (CTO) 0135

1. This Sampling and Analysis Plan (SAP) was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)* (U.S. EPA 2005) and *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, QAMS (U.S. EPA 2002)*.

2. Identify regulatory program: Military Munitions Response Program (MMRP); National Contingency Plan (NCP); Comprehensive Environmental Response Compensation, and Liability Act of 1980 (CERCLA)

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
<u>Kick-off meeting for development of Conceptual Site Model (CSM) and Data Quality Objectives (DQOs)</u>	<u>August 12, 2009</u>
<u>Kick-off meeting for development of CSM and DQOs</u>	<u>April 2, 2009</u>

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

Title	Date
<u>Work Plan for the Skeet and Pistol Range</u>	<u>March 2008</u>

6. List organizational partners (stakeholders) and connection with lead organization:

USEPA Region 6 – Regulatory Oversight
TCEQ – Regulatory Oversight
Naval Facilities Engineering Command Southeast (NAVFAC SE) – Property Owner

7. Lead organization Naval Facilities Engineering Command Southeast

8. 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:
NA, as there are no exclusions, all worksheets are applicable.

UFP-SAP Worksheet No.	Required Information	Crosswalk to Related Information
A. Project Management		
<i>Documentation</i>		
1	Title and Approval Page	NA
2	Table of Contents SAP Identifying Information	NA
3	Distribution List	NA
4	Project Personnel Sign-Off Sheet	NA
<i>Project Organization</i>		
5	Project Organizational Chart	NA
6	Communication Pathways	NA
7	Personnel Responsibilities and Qualifications Table	NA
8	Special Personnel Training Requirements Table	NA
<i>Project Planning/ Problem Definition</i>		
9	Project Planning Session Documentation (including Data Needs tables) Project Scoping Session Participants Sheet	NA
10	Problem Definition, Site History, and Background. Site Maps (historical and present)	NA
11	Site-Specific Project Quality Objectives	NA
12	Measurement Performance Criteria Table	NA
13	Sources of Secondary Data and Information Secondary Data Criteria and Limitations Table	NA
14	Summary of Project Tasks	NA
15	Reference Limits and Evaluation Table	NA
16	Project Schedule/Timeline Table	NA
B. Measurement Data Acquisition		
<i>Sampling Tasks</i>		
17	Sampling Design and Rationale	NA
18	Sampling Locations and Methods/ SOP Requirements Table Sample Location Map(s)	NA
19	Analytical Methods/SOP Requirements Table	NA
20	Field Quality Control Sample Summary Table	NA
21	Project Sampling Standard Operating Procedure (SOP) References Table Sampling SOPs	NA
22	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	NA
<i>Analytical Tasks</i>		
23	Analytical SOPs Analytical SOP References Table	NA
24	Analytical Instrument Calibration Table	NA
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	NA

<i>Sample Collection</i>		
26	Sample Handling System, Documentation Collection, Tracking, Archiving and Disposal Sample Handling Flow Diagram	NA
27	Sample Custody Requirements, Procedures/SOPs Sample Container Identification Example Chain-of-Custody Form and Seal	NA
<i>Quality Control Samples</i>		
28	QC Samples Table Screening/Confirmatory Analysis Decision Tree	NA
<i>Data Management Tasks</i>		
29	Project Documents and Records Table	NA
30	Analytical Services Table Analytical and Data Management SOPs	NA
C. Assessment Oversight		
31	Planned Project Assessments Table Audit Checklists	NA
32	Assessment Findings and Corrective Action Responses Table	NA
33	QA Management Reports Table	NA
D. Data Review		
34	Verification (Step I) Process Table	NA
35	Validation (Steps IIa and IIb) Process Table	NA
36	Validation (Steps IIa and IIb) Summary Table	NA
37	Usability Assessment	NA

SAP Worksheet No. 3 -- Distribution List

(UFP-QAAP Manual Section 2.3.1)

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-mail Address or Mailing Address	Document Control Number (Optional)
Leanna Woods Poon	Remedial Project Manager (RPM)	NAVFAC SE	904-542-6961	leanna.woodspoon@navy.mil	NA
Gary LeFlore	Base Coordinator	NASCC	361-961-3704	gary.leflore@navy.mil	NA
Bonnie Capito	Administrative Record Librarian	NAVFAC	757 322-4785	bonnie.capito@navy.mil	NA
Chris Siegel	TCEQ Project Manager	TCEQ	512-239-2992	csiegel@tceq.state.tx.us	NA
Tara Hubner	EPA Region 6 Project Manager	EPA	214-665-7246	hubner.tara@epa.gov	NA
Debra Humbert	Program Manager/Manages the Navy CLEAN Program	TtNUS	412-921-8698	debra.humbert@tetrattech.com	NA
G. Kenneth Grim, Jr	Task Order Manager (TOM)	TtNUS	832-251-6023	kenneth.grim@tetrattech.com	NA
Larry Basilio	Field Operations Leader (FOL)/Site Safety Officer (SSO)	TtNUS	832-251-6018	larry.basilio@tetrattech.com	NA
Matt Soltis (Health and Safety Plan [HASP] only)	Health and Safety Manager (HSM)/Manages Corporate Health and Safety Program	TtNUS	421-921-8912	matt.soltis@tetrattech.com	NA
Kelly Carper	TtNUS Quality Assurance Manager (QAM)/Manages	TtNUS	421-921-7273	Kelly.carper@tetrattech.com	NA

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-mail Address or Mailing Address	Document Control Number (Optional)
	Corporate Quality Assurance (QA) Program and Implementation				
Matthew Kraus (shared copy with Joe Samchuck)	Project Chemist	TtNUS	412-921-8729	matthew.kraus@tetrattech.com	NA
Joe Samchuck (shared copy with Matthew Kraus)	TtNUS Data Validation Manager (DVM)/Manages Data Validation	TtNUS	412-921-8510	joseph.samchuck@tetrattech.com	NA
Ralph Brooks	Unexploded Ordnance (UXO)/Munitions and Explosives of Concern (MEC) Manager/Manages Corporate MEC Hazards and Risks	TtNUS	770-413-0965 x231	ralph.brooks@tetrattech.com	NA
TBD	UXO Technician III/Manages Project MEC Hazards and Risks	TtNUS	TBD	TBD	NA
Kate Zaleski	Laboratory Project Manager (PM)	Katahdin Analytical Services, Inc. (Katahdin)	207-874-2400	kzaleski@katahdinlab.com	NA
TBD	Drilling Subcontractor/ Provides Drilling Services	TBD	TBD	TBD	NA

SAP Worksheet No.4 -- Project Personnel Sign-Off Sheet

[\(UFP-QAAP Manual Section 2.3.2\)](#)

Certification that project personnel have read the text will be obtained by one of the following methods as applicable:

1. In the case of regulatory agency personnel with oversight authority approval letters or e-mails will constitute verification that applicable sections of the SAP have been reviewed. Copies of regulatory agency approval letters / e-mails will be retained in the project files and are listed in Worksheet No. 29 as project records.
2. E-mails will be sent to Navy, TtNUS, and subcontractor project personnel whom will be requested to verify by e-mail that they have read the applicable SAP / sections and the date on which they were reviewed. Copies of the verification e-mail will be included in the project files and is identified in Worksheet No. 29.

A copy of the signed Worksheet No. 4 will be retained in the project files and is identified as a project document in Worksheet No. 29.

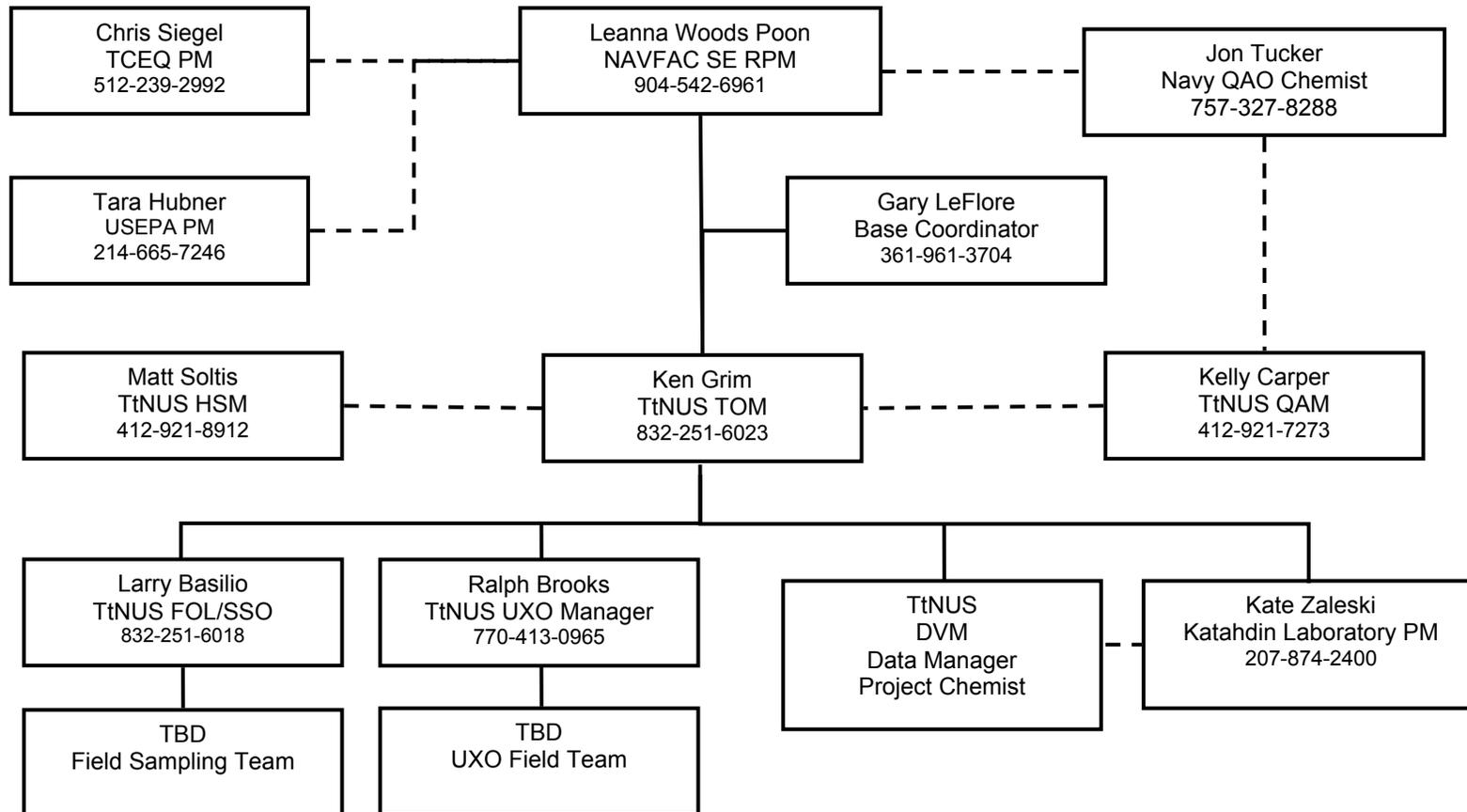
Name	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
TtNUS Project Team Personnel					
G. Kenneth Grim, Jr.	TtNUS//TOM	832-251-6023	See Worksheet No. 1 for signature	All	
Larry Basilio	TtNUS/FOL/SSO/Senior Geologist	832-251-6018		All	
Kelly Carper	TtNUS/Program Quality Assurance Manager (QAM)	412-921-7273	See Worksheet No. 1 for signature	All	

Name	Organization/Title/Role	Telephone Number	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Ralph Brooks	TtNUS/UXO/MEC Manager	770-413-0965 x231		Worksheet Nos. 10, 11, 14, 17, 18	
TBD	TtNUS/UXO Technician III	TBD		Worksheets Nos. 11, 14, 17, 18	
Joseph Samchuck	TtNUS/ DVM	412-921-8510		Worksheet Nos. 10, 11, 14, 17, 18	
Matthew Kraus	TtNUS/Project Chemist	412-921-8729		Worksheet Nos. 12, 14, 15, 19, 20, 23-28, 30, 34- 37	
Subcontractor Personnel					
Kate Zaleski	Katahdin/Laboratory PM	207-874-2400		Worksheet Nos. 6, 12, 14, 15, 19, 20, 23-28, 30, 34- 36	
TBD	Drilling Subcontractor/ Subcontractor PM/ Provides Drilling Services	TBD		Worksheet Nos. 6, 10, 11, 14, 17, and 18	

SAP Worksheet No. 5 -- Project Organizational Chart

(UFP-QAPP Manual Section 2.4.1)

Lines of Authority _____ - - - - - Lines of Communication



SAP Worksheet No. 6 -- Communication Pathways

(UFP-QAPP Manual Section 2.4.2)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure (timing, pathway to & from, etc.)
SAP amendments	TtNUS FOL TtNUS TOM Navy RPM	Larry Basilio G. Kenneth Grim, Jr. Leanna Woods Poon	832-251-6018 832-251-6023 904-542-6961	TtNUS FOL will verbally inform TtNUS TOM within 24 hours of realizing a need for an amendment. TtNUS TOM will document the proposed changes via a Field Task Modification Request (FTMR) form within five days and send Navy RPM a concurrence letter within seven days of identifying the need for change. Navy RPM will sign the concurrence letter within 5 days. SAP amendments will be submitted by TtNUS TOM to NAVFAC SE Program Management Office (PMO) for review and approval. TtNUS TOM will send scope changes to Project Team via e-mail within one business day.
Changes in schedule	TtNUS TOM Navy RPM	G. Kenneth Grim, Jr. Leanna Woods Poon	832-251-6023 904-542-6961	When impact is realized, send NAVFAC a schedule concurrence letter within 7 days or prior to the first affected deliverable date.
Issues in the field that result in changes in scope of field work	TtNUS FOL TtNUS TOM Navy RPM	Larry Basilio G. Kenneth Grim, Jr. Leanna Woods Poon	832-251-6018 832-251-6023 904-542-6961	TtNUS FOL will verbally inform TtNUS TOM on the day the issue is discovered. TtNUS TOM will verbally inform Navy RPM within one business day of the FOLs notification. TtNUS TOM also sends a concurrence letter to the Navy RPM within 7 days, if project scope is affected. The Navy RPM will sign the letter within 5 days of receipt. Document the change via FTMR form within two days of identifying the need for change within five days of initiating form.

Recommendations to stop work and initiate work upon corrective action	TtNUS FOL TtNUS TOM TtNUS QAM TtNUS HSM Navy RPM Base Coordinator	Larry Basilio G. Kenneth Grim, Jr. Kelly Carper Matt Soltis Leanna Woods Poon Gary LeFlore	832-251-6018 832-251-6023 412-921-7273 412-921-8912 904-542-6961 361-961-3704	If TtNUS is the responsible party for a stop work command, the TtNUS FOL will inform onsite personnel, subcontractor(s), the Navy RPM and Base Coordinator, and the identified Project Team members within 1 hour (verbally or by e-mail). If a subcontractor is the responsible party, the subcontractor PM must inform the TtNUS FOL within 15 minutes, and the TtNUS FOL will then follow the procedure listed above.
Corrective action for field program	TtNUS QAM TtNUS TOM Navy RPM	Kelly Carper G. Kenneth Grim, Jr. Leanna Woods Poon	412-921-7273 832-251-6023 904-542-6961	TtNUS QAM will notify (verbally or via e-mail) TtNUS TOM within one business day that the corrective action has been completed. The TtNUS TOM will then notify (verbally or via e-mail) the Navy RPM within one business day.
Field quality data issues	TtNUS FOL TtNUS TOM	Larry Basilio G. Kenneth Grim, Jr.	832-251-6018 832-251-6023	When a quality issue is related to field data, TtNUS FOL will verbally inform the TtNUS TOM on the same day.
Analytical data quality issues	Katahdin Laboratory PM TtNUS Project Chemist	Kate Zaleski Matt Kraus	207-874-2400 412-921-8729	Within one day of discovery, the Katahdin Laboratory PM will notify the TtNUS Project Chemist when a quality issue is related to laboratory data. TtNUS Project Chemist will notify (verbally or via e-mail) data validation staff and TtNUS TOM within one business day, if appropriate.
MEC encountered	TtNUS FOL TtNUS UXO/MEC Manager TtNUS TOM Navy RPM Base Coordinator Field Team Staff	Larry Basilio Ralph Brooks Ken Grim Leanna Woods Poon Gary LeFlore TBD (To Be Determined)	832-251-6018 770-413-0965 x231 832-251-6023 904-542-6961 361-961-3704 TBD	Within 30 minutes, TtNUS FOL will notify field staff, secure area, and contact TtNUS TOM, Base Coordinator and TtNUS UXO Manager. TtNUS UXO Manager will also verbally inform TtNUS TOM the same day. TtNUS TOM will verbally inform Navy RPM and Base Coordinator on the same day. Base Coordinator will make base emergency notifications. Navy RPM will inform Naval Ordnance Safety and Security Activity (NOSSA).

SAP Worksheet No. 7 -- Personnel Responsibilities and Qualifications Table

([UFP-QAPP Manual Section 2.4.3](#))

Name	Title/Role	Organizational Affiliation	Responsibilities
Leanna Woods Poon	RPM	NAVFAC SE	Oversees project, scoping, data review, and evaluation. Approves the SAP and other project deliverables.
Gary LeFlore	Base Coordinator	NASCC	Serves as the on-site point of contact (POC).
Chris Siegel	TCEQ Representative	TCEQ	Participates in scoping, data review, and evaluation and serves as TCEQ PM.
Tara Hubner	EPA Region 6 Representative	USEPA Region 6	Participates in scoping, data review, and evaluation and serves as USEPA Region 6 PM.
Kenneth Grim	TOM	TtNUS	<p>Oversees project, financial, schedule, and technical day to day management of the project, including the following:</p> <ul style="list-style-type: none"> • Ensures timely resolution of project-related technical, quality, and safety questions associated with TtNUS operations. • Functions as the primary TtNUS interface with the Navy RPM, NALF Cabaniss POC, TtNUS field and office personnel. • Ensures that TtNUS health and safety issues related to this project are communicated effectively to all personnel and off-site laboratory. • Monitors and evaluates all TtNUS subcontractor performance. • Coordinates and oversees work performed by TtNUS field and office technical staff (including data validation, data interpretation, and report preparation). • Coordinates and oversees maintenance of all TtNUS project records. • Coordinates and oversees review of TtNUS project deliverables. • Prepares and issues final TtNUS deliverables to the Navy.

Larry Basilio	FOL, SSO	TtNUS	<p>Supervises, coordinates, and performs field sampling activities, including the following:</p> <ul style="list-style-type: none"> • Ensures that all health and safety requirements unique to the RI are implemented. • Functions as the on-site communications link between field staff members, the Navy RPM and Base Coordinator, and the TtNUS TOM. • Alerts off-site analytical laboratory of any special health and safety hazards associated with environmental samples. • Oversees the mobilization and demobilization of all field equipment and subcontractors. • Coordinates and manages the field technical staff. • Adheres to the work schedules provided by the TtNUS TOM. • Ensures the proper maintenance of site logbooks, field logbooks, and field recordkeeping. • Initiates FTMRs (field change orders) when necessary. • Identifies and resolves problems in the field, resolving difficulties via consultation with the Navy RPM and Base Coordinator, implementing and documenting corrective action procedures, and providing communication between the field team and project management. • As the SSO, is responsible for training and monitoring site conditions. The SSO reports to the HSM and to the TtNUS TOM. Details of the SSO's responsibilities are presented in the HASP.
Matt Soltis	HSM	TtNUS	<p>Oversees CLEAN Program Health and Safety Program, including the following:</p> <ul style="list-style-type: none"> • Provides technical advice to the TtNUS TOM on matters of health and safety. • Oversees the development and review of the HASP. • Conducts health and safety audits. • Prepares health and safety reports for management.

Kelly Carper	QAM	TtNUS	<p>Reviews UFP-SAP, oversees preparation of laboratory scope, coordinates with laboratory, and conducts data quality reviews. Ensures quality aspects of the CLEAN program, including the following:</p> <ul style="list-style-type: none"> • Develops, maintains, and monitors QA policies and procedures. • Provides training to TtNUS staff in quality assurance/quality control (QA/QC) policies and procedures. • Conducts systems and performance audits to monitor compliance with environmental regulations, contractual requirements, UFP-SAP requirements, and corporate policies and procedures. • Audits project records. • Monitors subcontractor quality controls and records. • Assists in the development of corrective action plans and ensuring correction of non-conformances reported in internal or external audits. • Ensures that this SAP meets TtNUS, Navy, and TCEQ requirements. • Prepares QA reports for management.
Matt Kraus	Project Chemist	TtNUS	<p>Coordinates analyses with laboratory chemists, ensures the scope is followed, QA data packages, and communicates with TtNUS staff.</p> <ul style="list-style-type: none"> • Ensures that the project meets objectives from the standpoint of laboratory performance • Provides technical advice to the Project Team on matters of project chemistry. • Monitors and evaluates subcontractor laboratory performance. • Ensures timely resolution of laboratory-related technical, quality, or other issues effecting project goals. • Functions as the primary interface with the subcontracted laboratory and the TtNUS TOM. • Coordinates and oversees work performed by the subcontracted laboratory. • Oversees the completion of TtNUS data validation. • Coordinates and oversees review of laboratory deliverables. • Recommends appropriate laboratory corrective actions.
Joseph Samchuck	DVM	TtNUS	<p>Ensures the QA of data validation deliverables, including the following:</p> <ul style="list-style-type: none"> • Oversees data validation activities. • Serves as communication link between TtNUS and laboratory on data validation and electronic data positing activities. • Establishes TtNUS data validation protocols in support of projects.

Ralph Brooks	UXO/MEC Manager	TtNUS	Oversees selection of qualified UXO personnel, establishes overall quality control program for UXO activities, addresses UXO-related issues identified by field personnel.
TBD	UXO Technician III	TtNUS	Provides anomaly avoidance services. Will have a minimum of 8 years prior military Explosive Ordnance Disposal (EOD) and/or commercial UXO experience in munitions response actions or range clearance activities.
Kate Zaleski	Laboratory PM	Katahdin	Coordinates analyses with laboratory chemists, ensures that scope is followed, performs QA of data packages, communicates with TtNUS staff.
TBD	Drilling PM	TBD	Coordinates drilling with FOL, ensures that scope is followed, communicates with TtNUS staff.

SAP Worksheet No. 8 -- Special Personnel Training Requirements Table

([UFP-QAPP Manual Section 2.4.4](#))

Project Function	Specialized Training by Title or Description of Course	Training Provider	Training Date	Personnel / Groups Receiving Training	Personnel Titles / Organizational Affiliation	Location of Training Records / Certificates
Field Technicians	40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER); 8-hour HAZWOPER Refresher	Various	Current	Field sampling personnel	All field staff / TtNUS	TtNUS project office and field office
FOL	Same as field technician HAZWOPER requirements, plus Supervisor training	Various	Current	FOL	FOL / TtNUS	TtNUS project office and field office
Health and Safety Officer	First Aid / Cardiopulmonary Resuscitation Training	Red Cross	Current	Field Personnel	SSO / TtNUS	TtNUS project office and field office
UXO Avoidance	Various training elements, as required in DoD Explosive Safety Board (DDESB) Technical Paper (TP)-18	DoD or other approved formal course	Current	UXO Technicians supporting UXO avoidance	UXO Technician / TtNUS	TtNUS project office and field office

All Field personnel will have appropriate training to conduct the field activities to which they are assigned. Additionally, each site worker will be required to have completed a 40-hour course (and 8-hour refresher, if applicable) in Health and Safety Training as described under Occupational Safety and Health Administration (OSHA) 29 Code of Federal Regulations (CFR) 1910.120(b)(4).

SAP Worksheet No. 9 -- Project Scoping Session Participants Sheet

(UFP-QAPP Manual Section 2.5.1)

Project Name: NALF Cabaniss Projected Date(s) of Sampling: <u>Spring 2010</u> Task Order Manager: G. Kenneth Grim, Jr.		Site Name: Skeet Range, NALF Cabaniss Site Location: Corpus Christi, Texas			
Date of Session: August 12, 2009					
Scoping Session Purpose: Data Quality Objective Session					
Name	Title	Affiliation	Phone No.	E-mail Address	Project Role
G. Kenneth Grim, Jr.	TOM	TtNUS	832-251-6023	kenneth.grim@tetrattech.com	Management
Larry Basilio	FOL/Sr. Geologist	TtNUS	832-251-6018	larry.basilio@tetrattech.com	Task Management/ Technical
Bridget Twigg	Geologist	TtNUS	832-251-5195	Bridget.Twigg@tetrattech.com	Technical
Ralph Basinski	MRP Manager	TtNUS	412-921-8308	Ralph.basinski@tetrattech.com	Task Management/ Technical
Peggy Churchill	DQO Specialist	TtNUS	321-636-6470	Peggy.churchill@tetrattech.com	Technical
Helen Lockard	RPM	NAVFAC SE	904-542-6858	helen.lockard@navy.mil	Management
Chris Siegel	TCEQ Representative	TCEQ	512-239-2992	csiegel@tceq.state.tx.us	Regulatory
Scott Settemeyer	TCEQ Representative	TCEQ	512-239-3429	ssetteme@tceq.state.tx.us	Regulatory
Jim Pastorick	TCEQ Consultant	UXOPro	703-548-5300	jim@uxopro.com	Technical
Gary LeFlore	Base Coordinator	NASCC	361-961-3704	gary.lefore@navy.mil	Support
Dalton Shaughnessy	RPM	NAVFAC SE	904-542-6970	Dalton.shaughnessy@navy.mil	Management
Felix Hernandez	Base Safety	NASCC Safety	361-961-4470	Felix.hernandez@navy.mil	Support

Comments/Decisions: A site walk was conducted by session participants. Once the site walk was completed, the DQO session was held. Conceptual Site Model was reviewed and the 7-step DQO process was then conducted for the site.

Consensus Decisions:

- Delineate to Texas Risk Reduction Program criteria.
- Use ½-acre decision units

Project Name: NALF Cabaniss Projected Date(s) of Sampling: <u>March 2010</u> Task Order Manager: G. Kenneth Grim, Jr.		Site Name: Skeet Range, NALF Cabaniss Site Location: Corpus Christi, Texas			
Date of Session: April 2, 2009					
Scoping Session Purpose: Data Quality Objective Session					
Name	Title	Affiliation	Phone No.	E-mail Address	Project Role
G. Kenneth Grim, Jr.	TOM	TtNUS	832-251-6023	kenneth.grim@tetrattech.com	Management
Larry Basilio	FOL/Sr. Geologist	TtNUS	832-251-6018	larry.basilio@tetrattech.com	Task Management/ Technical
Ralph Basinski	MRP Manager	TtNUS	412-921-8308	Ralph.basinski@tetrattech.com	Task Management/ Technical
Helen Lockard	RPM	NAVFAC SE	904-542-6858	helen.lockard@navy.mil	Management
Chris Siegel	TCEQ Representative	TCEQ	512-239-2992	csiegel@tceq.state.tx.us	Regulatory
Tara Hubner	EPA Representative	EPA	214-665-7246	Hubner.tara@epa.gov	Regulatory
Gary LeFlore	Base Coordinator	NASCC	361-961-3704	gary.lefore@navy.mil	Support
Dalton Shaughnessy	RPM	NAVFAC SE	904-542-6970	Dalton.shaughnessy@navy.mil	Management
Felix Hernandez	Base Safety	NASCC Safety	361-961-4470	Felix.hernandez@navy.mil	Support

Comments/Decisions: A formal agenda was prepared by Ralph Basinski (TtNUS). Participants were asked to provide a “plan of attack” they would like to discuss in regards to the Skeet Range. The team agreed to discuss the following items:

- Review results of SI
- Establish RI objectives
- Outline preliminary planning steps

Consensus Decisions:

- PAHs are the only contaminants of concern (COCs)
- Delineate PAHs in surface soil, subsurface soil, and groundwater

SAP Worksheet No. 10 -- Conceptual Site Model

([UFP-QAPP Manual Section 2.5.2](#))

10.1 INTRODUCTION

The Skeet Range RI is the subject of this MC UFP-SAP. General information for NALF Cabaniss is presented below. Site-specific information related to the Skeet Range is presented in Section 10.2. The CSM is presented in Section 10.3.

NALF Cabaniss

NALF Cabaniss encompasses a total of 923 acres and is located on the eastern side of Nueces County, Texas, and lies approximately eight miles west of NASCC. Figure 10-1 shows the general location of NALF Cabaniss and the location of the Skeet Range. The installation is immediately bounded on the east by Brezina Road, on the north by Ayers Street and Farm-to-Market (FM) 286, to the west by Saratoga Road, and to the south by Oso Creek, a perennial water body that ultimately flows into Oso Bay. Beyond Oso Creek are agricultural and industrial properties. The area east of the installation is comprised of mixed agricultural, industrial, and residential areas. North of the current boundary are former buildings and recreational areas that were once a part of the installation. These areas were transferred to the General Services Administration (GSA) for disposal in 1958, and are now the property of the local school district. Residential zones lie beyond these buildings to the north. A former landfill is located directly west of the installation.

NALF Cabaniss is an OLF with the current primary role of supporting naval air training operations originating from NASCC. The installation was originally constructed with four 5,000-foot runways. Only two runways, oriented in north/south and northwest/southeast directions, are presently active and maintained. The airfield is lighted, to allow for night flight training, and daylight training is also conducted.

There are no currently operating ordnance/munitions storage facilities at NALF Cabaniss.

10.2 SITE LOCATION, HISTORY, AND PHYSICAL FEATURES OF THE FORMER SKEET RANGE

Location

The former Skeet Range is located in the southeastern corner of the installation, 1,230 feet southeast of Runway 31 and 400 feet north of Oso Creek. Figure 10-2 shows the Skeet Range at NALF Cabaniss. A former drainage ditch lies to the west of the former range, while another drainage canal currently

intersects the eastern end of the former range area. The area surrounding the former range is covered in vegetation.

History and Physical Features

The former Skeet Range was originally constructed in 1943. Initially, the site contained one skeet range firing area, comprised of two large firing arcs for skeet shooting, three smaller firing arcs for trap shooting, and an armory. Wood-frame “high” and “low” skeet houses were positioned at the end of each skeet firing arc, which measured approximately 148 feet in length. The trap firing arcs present on the east side of the range were smaller in size than the skeet firing arcs (approximately 82 feet in length) and had trap houses centered in the middle of each firing arc. By January 1944, an additional skeet firing arc was added on the western side of the skeet range. All firing arcs faced to the southwest toward the installation boundary and Oso Creek. World War II (WWII)-era skeet and trap ranges were typically constructed with five firing positions per firing arc.

The Skeet Range was generally used for small arms qualification and moving target orientation training for naval aviators, although the range may have also been used for recreational purposes. Ammunition used at the site likely included 12-, 16-, and 20-gage and .410 caliber shotgun munitions; other small caliber ammunition (e.g., .22 caliber, .38 caliber, .45 caliber, 9-millimeter [mm]) were likely used at the range for pistol training purposes. The armory associated with the former Skeet Range is no longer present at the installation, and the date of decommissioning is not known. The former small arms magazine remains in place in an open field east of a drainage canal on property that is no longer owned by the installation. The Skeet Range was demolished between 1958 and 1964.

Historical documentation (station documents and drawings) and NASCC personnel indicated that no other explosives or munitions were used at the site and that the site was not used for any other purpose.

Site Inspection Results

A SI was conducted at the former Skeet Range in April and May 2008. The SI consisted of the collection of surface soil, surface water, and sediment samples; laboratory analysis (antimony, arsenic, copper, lead, zinc, and PAHs) of surface soil samples, surface water and sediment samples; land surveying of sample locations; and reporting of results. Two soil borings were drilled to determine subsurface lithology. Subsurface soil samples were not collected for laboratory analysis. Temporary monitoring wells were installed to collect groundwater samples to determine the groundwater resource classification. However, due to heavy silting of the temporary monitoring wells, groundwater samples were unable to be collected.

The SI consisted of the drilling of soil borings; collection and laboratory analysis of surface and subsurface soil samples; installation of temporary groundwater monitoring wells; collection and laboratory analysis of surface water and sediment samples; Global Positioning System (GPS) survey of sample locations; and reporting of results. Surface soil and sediment samples were analyzed for metals (antimony, arsenic, copper, lead, and zinc) and PAHs. Surface water samples were analyzed for metals (antimony, arsenic, copper, lead and zinc). One surface soil sample at the location of the MEC item was analyzed for Target Analyte List (TAL) metals, explosives and perchlorate. However, due to heavy silting of the temporary monitoring wells, groundwater samples were unable to be collected.

Analytical results of soil samples were compared to risk-based regulatory screening values to determine if potential impacts to human health or the environment are present. Measured surface soil concentrations were compared to TRRP Tier 1 Residential Protective Concentration Levels (PCLs) for a 0.5-acre source area. Measured surface water concentrations were compared to Saltwater Aquatic Life Surface Water Risk Based Exposure Limits (RBELs), Saltwater Human Health Surface Water RBELs and TRRP Tier 1 Contact Recreational Water PCLs. Measured sediment concentrations were compared to criteria presented in Table 3-3, Ecological Benchmarks for Sediments, of TCEQ guidance document RG-263, Guidance for Conducting Ecological Risk Assessments at Remediation Sites in Texas (January 2006). Analytical results for sediments were also compared to TRRP Tier 1 Direct Human Contact Sediment PCLs.

The analytical results included four PAHs that were detected at concentrations greater than the TRRP Tier 1 Residential PCL criteria in surface soil samples. Benzo(a)anthracene was detected in six soil samples at concentrations greater than the TRRP Tier 1 Residential PCL criteria of 5.65 milligrams per kilogram (mg/kg) at concentrations ranging from 7.45 mg/kg to 158 mg/kg. Benzo(a)pyrene was detected in nine soil samples (8 normal and 1 duplicate) at concentrations greater than the TRRP Tier 1 Residential PCL criteria of 0.56 mg/kg at concentrations ranging from 0.615 mg/kg to 187 mg/kg. Benzo(b)fluoranthene was detected in eight soil samples at concentrations greater than the TRRP Tier 1 Residential PCL of 5.71 mg/kg at concentrations ranging from 8.25 mg/kg to 323 mg/kg. Indeno(1,2,3-cd)pyrene was detected in three soil samples at concentrations greater than the TRRP Tier 1 Residential PCL of 5.72 mg/kg at concentrations ranging from 7.76 mg/kg to 98.2 mg/kg. Skeet fragments were also noted in Grids 1, 4, 7, 8, 9, 11, 12, and 13. Figure 10-3 depicts the distribution of the PAH exceedances.

Metals and propellants were either not detected or were detected at concentrations above the reporting limit but less than the TRRP Tier 1 Residential PCLs. Analytical results for surface water and sediment were less than the TRRP human health or ecological criteria for all target analytes.

The site-specific geological setting of the Skeet Range consists of an upper fine-grained unit and a lower coarse-grained unit. The upper fine-grained unit consisted of a gray to tan with depth, lean clay with a varying amount of admixed silt. The silt content generally increased with depth. Caliche nodules were present in the upper portions of the section. The thickness of the upper fine-grained unit was between 17 and 18 feet. The lower coarse-grained unit was the first unit in which saturated soils were encountered. The contact between the two units was generally well defined. The lower coarse-grained unit consisted of a gray to tan very fine grained silty sand.

Groundwater at the site is under semi-confined conditions. Depth to static groundwater was measured at approximately 15 feet and 18.5 feet below ground surface (bgs) in the two temporary monitoring wells installed at the site. The actual water-bearing unit was encountered between 17 and 18 feet bgs. Due to heavy silting of the temporary monitoring wells, groundwater samples were unable to be collected.

During brush clearing operations to allow for surface soil sampling at the Skeet Range during the SI, one MEC item was discovered. The item, a smoke cartridge, was inspected by UXO technicians, left in place, and reported to NASCC and NOSSA personnel. The discovery of the MEC item lead to a change in the Explosive Safety Submission (ESS) Determination for the site. UXO Avoidance was added to that site for the safety of sampling crews. UXO technicians were on site during the MC SI investigation and sampling to conduct UXO avoidance activities.

10.3 CONCEPTUAL SITE MODEL FOR THE FORMER SKEET RANGE

The CSM indicates that potentially complete exposure pathways exist for both human and ecological receptors under both current and hypothetical future land uses. Figure 10-4 presents a general graphical depiction of the CSM for the Skeet Range.

10.3.1 Potential or Known Contaminants

Analytical results from the SI indicate that concentrations of MC (PAHs) in surface soil at eight locations are greater than TRRP Tier 1 Residential PCLs. Metals and propellants in surface soil were either not detected or were detected at concentrations above the reporting limit but less than the TRRP Tier 1 Residential PCLs. Analytical results for metals and PAHs in surface water and sediment are less than the applicable TRRP human health or ecological criteria that are identified in Section 10.2.

10.3.2 Contaminant Migration Pathways

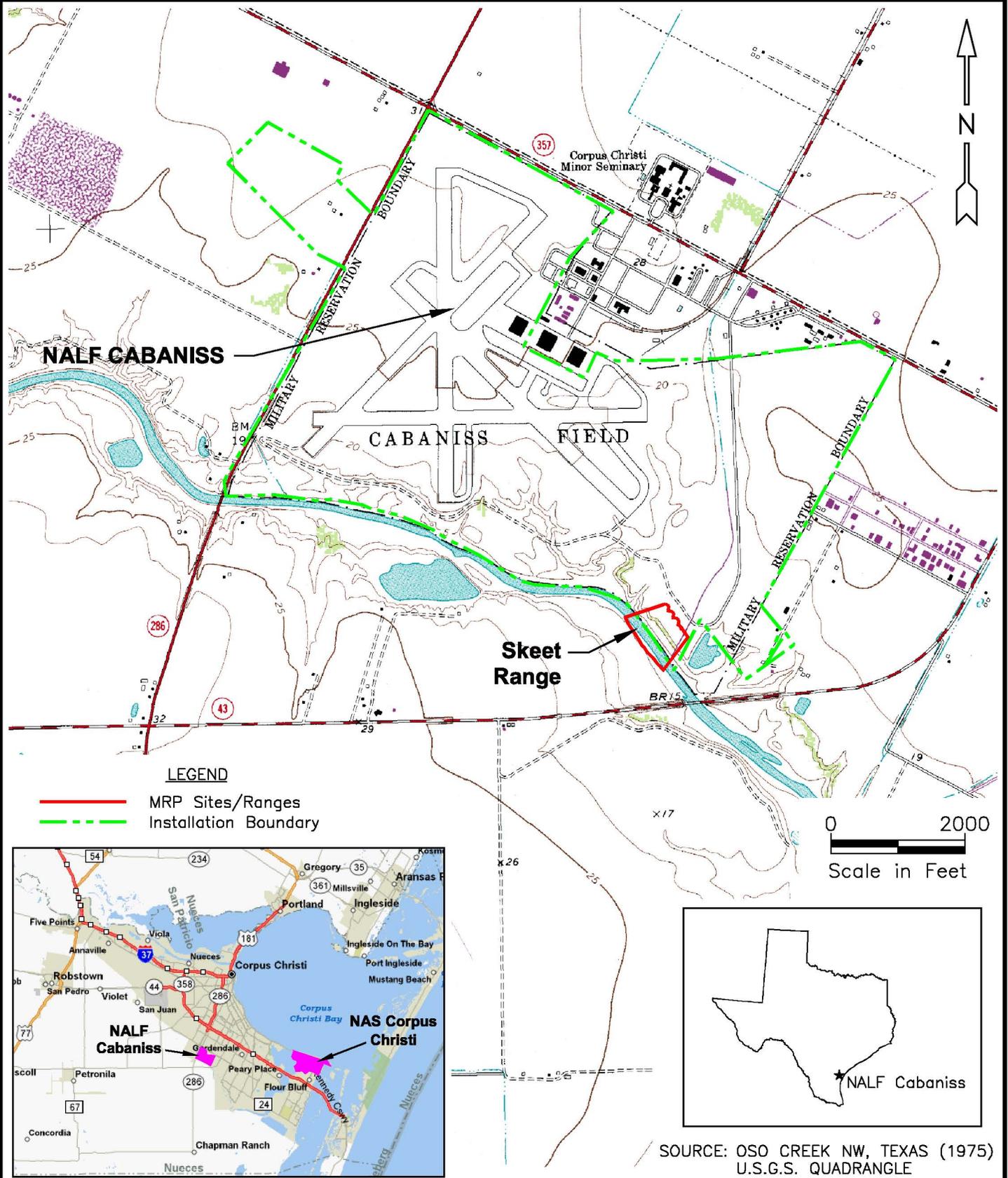
PAHs in surface soil may have migrated to subsurface soil and subsequently infiltrated to underlying groundwater. However, PAHs present in the clay targets tend to be tightly bound in the petroleum pitch and limestone matrix of the target and are not readily available to the environment. In addition, clay targets contain low solubility, high molecular weight PAHs that are not likely to leach into the surrounding soils. The upper fine-grained clay unit would tend to bind any PAHs and limit downward migration of PAHs to underlying soil and groundwater. Future construction, excavation, and maintenance at the site could be a release mechanism.

10.3.3 Exposure Pathways

Potentially complete exposure pathways exist for MC in surface and subsurface soil (direct contact, ingestion, and inhalation) for potential human receptors. However, it is not anticipated that trespassers would come in contact with subsurface soil. Confining layers and slow migration rates are expected to limit the migration of MC to the lower aquifers used for water supplies. Pathways of exposure for sediment and surface water in Oso Creek are considered incomplete.

10.3.4 Receptors

Possible current day receptors include: human and ecological. Human receptors may be Navy personnel patrolling the area and Public Works personnel, contractors, trespassers, and visitors. Ecological receptors may include common flora/fauna, predominantly grassland species, large mammals such as deer, small mammals such as rabbits, reptiles/amphibians, and bird species. No threatened or endangered species are known to occur on or near the site. Future use of the site is not expected to change.

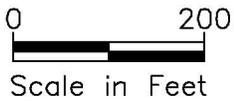
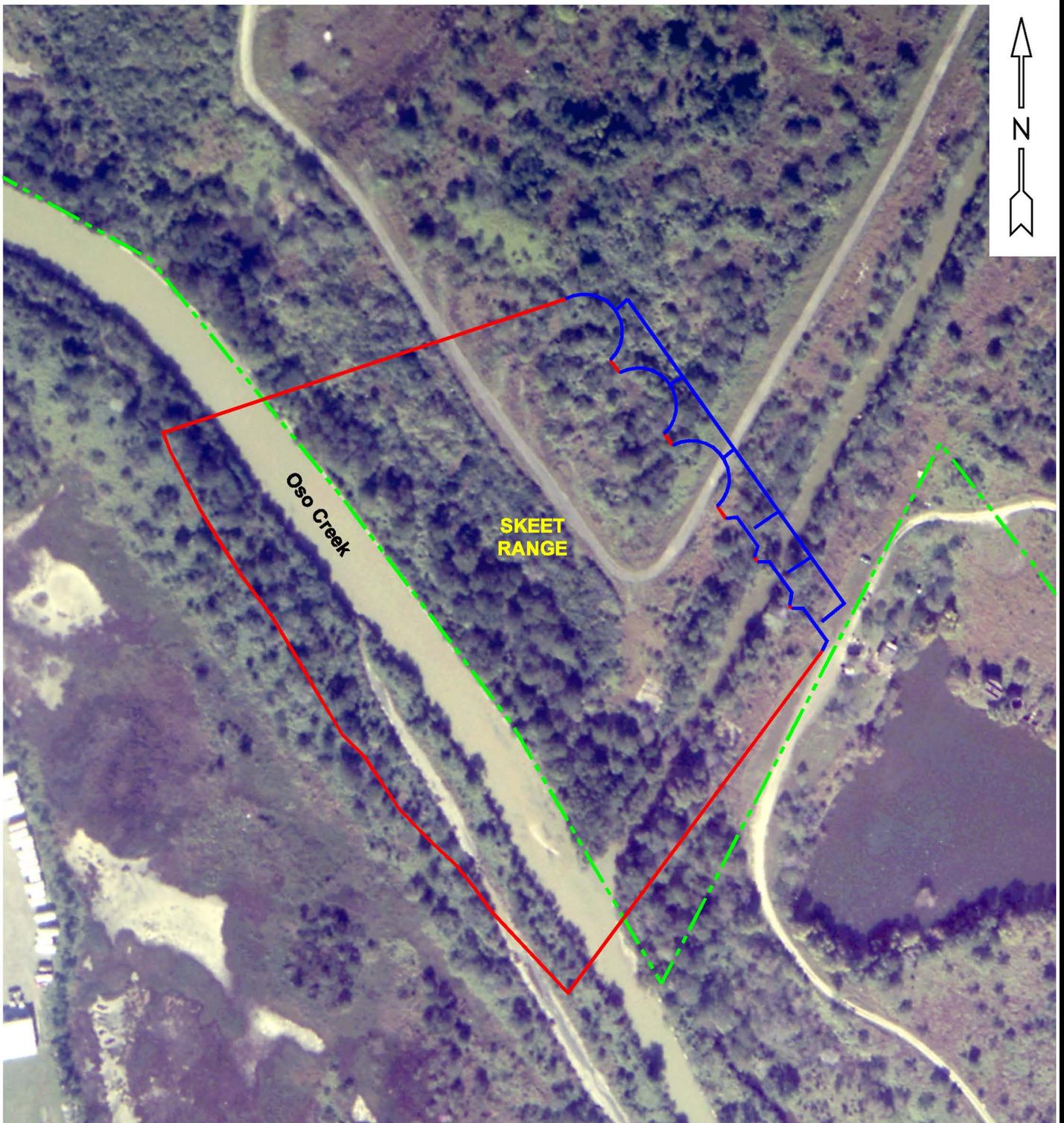


DRAWN BY GS	DATE 10/19/10
CHECKED BY LB	DATE 10/19/10
REVISED BY	DATE
SCALE AS NOTED	



AREA LOCATION MAP
SKEET RANGE
NALF CABANISS, TEXAS

PROJECT NO. 112G01821	
CTO NO. 0135	
APPROVED BY	DATE
DRAWING NO. FIGURE 10-1	REV. 0



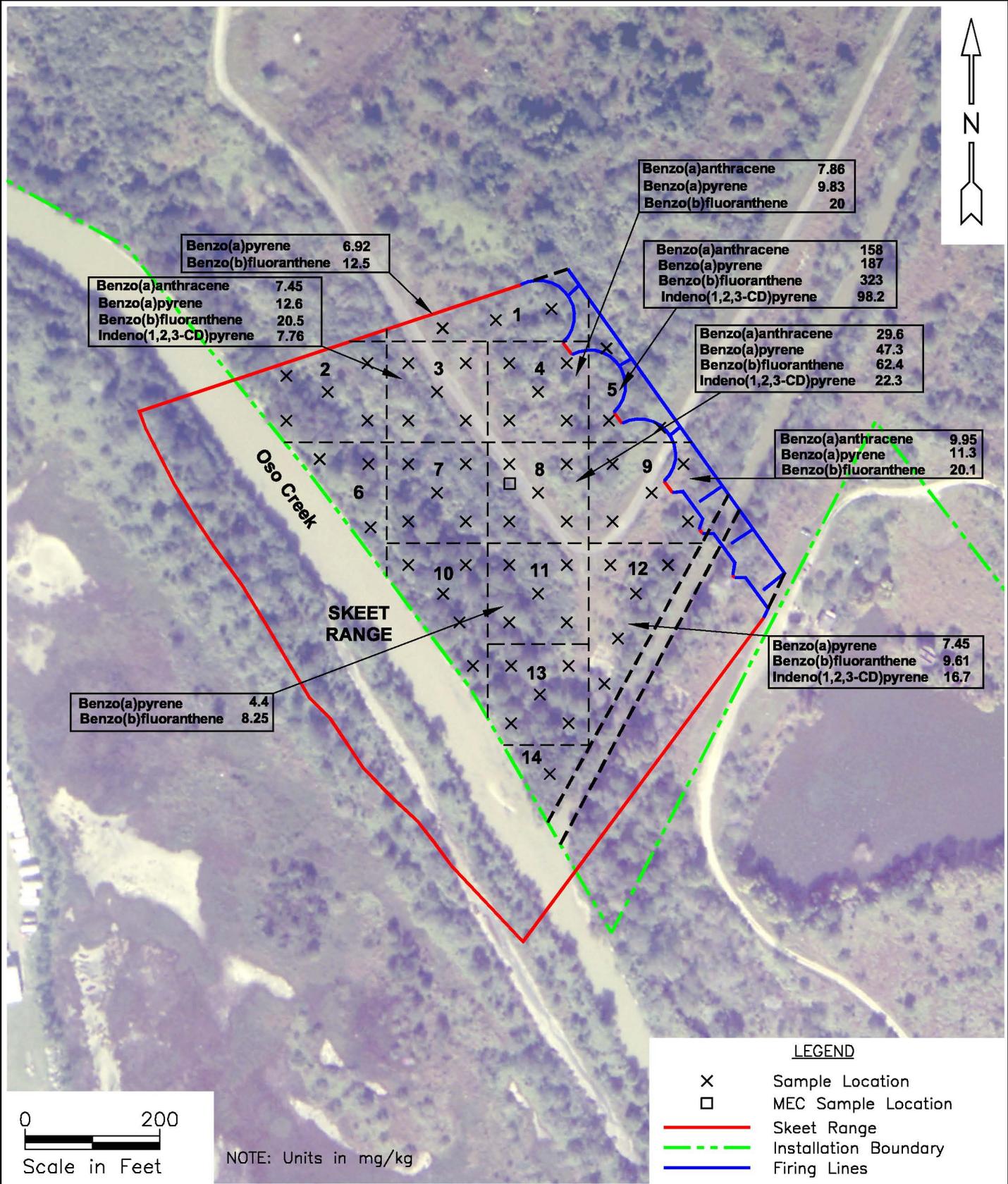
LEGEND	
	Skeet Range
	Installation Boundary
	Firing Lines

DRAWN BY	DATE
GS	10/15/10
CHECKED BY	DATE
LB	10/15/10
REVISED BY	DATE
SCALE AS NOTED	



SITE MAP
SKEET RANGE
NALF CABANISS, TEXAS

PROJECT NO. 112G01821	
CTO NO. 0135	
APPROVED BY	DATE
DRAWING NO. FIGURE 10-2	REV. 0



Benzo(a)anthracene 7.45 Benzo(a)pyrene 12.6 Benzo(b)fluoranthene 20.5 Indeno(1,2,3-CD)pyrene 7.76	Benzo(a)pyrene 6.92 Benzo(b)fluoranthene 12.5	Benzo(a)anthracene 7.86 Benzo(a)pyrene 9.83 Benzo(b)fluoranthene 20	Benzo(a)anthracene 158 Benzo(a)pyrene 187 Benzo(b)fluoranthene 323 Indeno(1,2,3-CD)pyrene 98.2	Benzo(a)anthracene 29.6 Benzo(a)pyrene 47.3 Benzo(b)fluoranthene 62.4 Indeno(1,2,3-CD)pyrene 22.3	Benzo(a)anthracene 9.95 Benzo(a)pyrene 11.3 Benzo(b)fluoranthene 20.1
Benzo(a)pyrene 4.4 Benzo(b)fluoranthene 8.25					Benzo(a)pyrene 7.45 Benzo(b)fluoranthene 9.61 Indeno(1,2,3-CD)pyrene 16.7



NOTE: Units in mg/kg

LEGEND	
x	Sample Location
□	MEC Sample Location
— (Red)	Skeet Range
- - - (Green)	Installation Boundary
— (Blue)	Firing Lines

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SCALE AS NOTED	

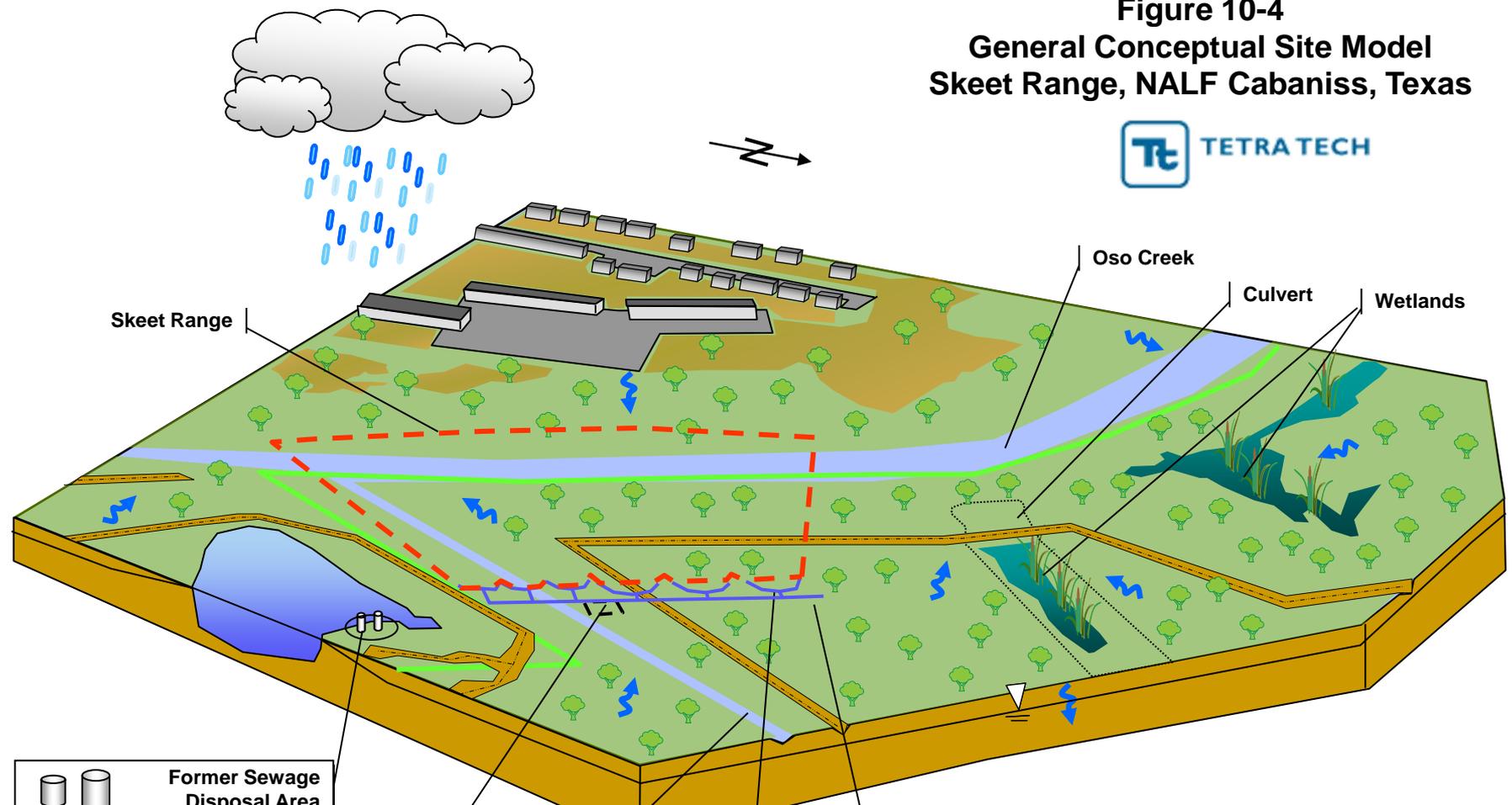


SURFACE SOIL SAMPLE EXCEEDANCES – SKEET RANGE

NALF CABANISS, TEXAS

PROJECT NO. 112G01821	
CTO NO. 0135	
APPROVED BY	DATE
DRAWING NO. FIGURE 10-3	REV. 1

Figure 10-4
General Conceptual Site Model
Skeet Range, NALF Cabaniss, Texas



Former Sewage Disposal Area

Former Armory

Drainage Channel

Former Firing Arcs
for the Skeet/Trap Range

Sidewalk

Feature Key	
	Skeet Range Boundary
	Installation Boundary
	Dirt Road
	Industrial Buildings
	Vegetation
	Precipitation
	Infiltration and Runoff
	Water Table

SAP Worksheet No. 11 -- Project Quality Objectives/Systematic Planning Process Statements

(UFP-QAPP Manual Section 2.6.1)

11.1 PROBLEM STATEMENTS

This site has been identified as a former Skeet Range where shotguns were used to fire lead shot at clay pigeons. As a result, PAHs are present in surface soil at concentrations that exceed TRRP risk-based PCLs. In order to delineate PAH contamination in soil, data regarding the horizontal and vertical extent of PAH contamination is needed. Surface soil, sediment, and surface water were the only media investigated during the SI; therefore, groundwater data must be collected during the RI in order to determine if the PAHs in surface soil have migrated to subsurface soil or groundwater. Once the extent of PAH contamination in soil and groundwater has been determined during the RI, the Project Team will decide if a response action is necessary. The RI is being conducted according to the TRRP rule (30 Texas Administrative Code Chapter 350) process. The TRRP rule specifies the assessment, monitoring, cleanup, reporting, and other requirements for regulated sites in Texas. PCLs are the regulatory standard for a concentration of a COC in a source medium in order to protect a receptor at the point of exposure to that PCL. PCLs are back calculated by determining what concentration of a COC could remain at the source and still yield protective concentrations at the point of exposure. The PCL development process is different from traditional baseline risk assessment process that starts with a known concentration in a source area and assesses the risk to the receptor at the point of exposure. As such, under TRRP, a baseline risk assessment is not required. Figure 11-1 depicts the Study Goal and Decision Rule Flow Chart for the Skeet Range.

11.2 IDENTIFY INFORMATION INPUTS

Data required for making the decisions include the following:

1. **Previously Collected Data:** Data from the SI will be used to select areas for horizontal and vertical delineation. Horizontal delineation will use a grid pattern to expand outward from those grid areas identified during the SI as impacted with PAHs. The grid areas with the highest surface soil PAH concentrations identified during the SI will be selected for vertical delineation.
2. **Chemical Data:** Chemical data will be acquired using methods identified in Worksheet No. 19 and will be used to determine the presence and concentrations of PAHs in soil and groundwater. In addition to laboratory data, field measurements of groundwater parameters (temperature, turbidity, pH, water levels) will be collected.

3. Project Action Limits: This investigation requires PAH data that can be used to determine whether further investigation or a response action is necessary. To conduct comparisons of site data to TRRP PCLs and to background concentrations, the limits of quantitation (LOQ) provided by Katahdin must be low enough to measure target analyte concentrations to regulatory or other stringent and conservative values. The following risk-based human health and ecological criteria were used to select the most stringent screening value for the particular analyte and media being investigated:
 - TRRP Tier 1 Residential PCL for soil (surface and subsurface)
 - TRRP Tier 1 Residential PCL for groundwater
 - TRRP Ecological Screening Levels for soil
 - USEPA Ecological Soil Screening Levels
4. Field identification/classification of soil types (i.e., lithology). Worksheet No. 21 contains relevant SOPs that will be followed.

11.3 DEFINE THE BOUNDARIES OF THE STUDY

The current RI is limited to the evaluation of surface and subsurface soil and groundwater at the Skeet Range. Establishing the nature and extent of contamination will require that both contaminated and non-contaminated soil be sampled (i.e., the perimeter of the impacted area must be established). Decision units are ½-acre in size, which corresponds to the TCEQ definition of a soil exposure area for a commercial/industrial site. The following items describe the horizontal and vertical boundaries as well as the temporal boundaries for the study:

1. The populations of interest include soil and groundwater that have or may have been contaminated directly by site operations or by subsequent migration of contaminants. Data that is representative of both types of populations for both types of media will be collected.
2. The horizontal study boundary will encompass the area that, based on historical information and SI sampling results, are impacted with PAHs in excess of the TRRP Tier 1 Residential PCL. Lateral expansion (i.e. “step-out”) of this horizontal study boundary may be necessary if PAH

concentrations detected in the samples collected along the study area boundary exceed screening levels. A maximum of 2 “step-out” grids will be sampled. If samples at the 2nd “step-out” grid exceed screening levels, the Project Team will re-evaluate the CSM for the Skeet Range RI.

3. The vertical study boundary is the soil column from ground surface to the top of the water table, which is estimated to be approximately 18 feet bgs. The groundwater interval of interest is the first water bearing zone encountered.
4. The temporal boundary is not a significant consideration in this study because PAH concentrations are anticipated to be relatively unchanged (stable) over the course of time needed to conduct the environmental investigations.

The study area is limited to NALF Cabaniss proper. The Project Team decided that though the estimated shotfall area of the Skeet Range extended slightly on to property to the southwest of Oso Creek and southeast of the drainage ditch, analytical results from the SI indicated that the possibility of impacts to these areas was minimal. There were no MC impacts detected in the surface water or sediments samples separating NALF Cabaniss from these two areas and these areas are at the extreme edges of the shotfall zone. One item of MEC, a smoke cartridge, was found during the SI. The discovery of the MEC is believed to be an anomaly as no documentation exists to support use of MEC at the Skeet Range. However as a safety precaution, UXO Technicians will be onsite to provide UXO avoidance support during the RI. The scope of the RI is the investigation of MC not MEC. Should additional MEC be found, the Project Team will notify the Tetra Tech PM, Navy RPM, and NOSSA personnel in accordance with the existing Explosive Safety Submission (ESS) waiver. In addition, the detection of additional MEC at the Skeet Range will require the project team to reevaluate the conceptual model for the site to consider additional dumping or disposal activities that may have occurred after the closure of the Skeet Range.

11.4 DEVELOP THE ANALYTIC APPROACH

There are two main aspects of this investigation. One is field delineation of contamination. The other is comparison of data to action levels (TRRP PCLs and ecological screening levels) to determine an appropriate course of action to mitigate potential unacceptable risks. Decision making will be conducted using the following approaches for determining the nature and extent of contamination at the site.

Delineation Approach:

Individual PAH analyte concentrations will be determined in surface and subsurface soil and groundwater through analysis by Katahdin. These concentrations will be compared to the TRRP Tier 1 PCLs listed in Worksheet No. 15.

- If any target analyte concentrations in the receptor exposure units defined for soil or groundwater exceed the screening value, which is also the Project Action Limit (PAL), continue delineation according to TRRP-12 guidance until delineated or a maximum of two step out grids have been completed.
- If all target analyte concentrations in the receptor exposure units defined for soil or groundwater are less than the PAL, then delineation is complete and no more data collection is required.

Comparison to Human Health TRRP Criteria:

Individual PAH concentrations will be determined in surface and subsurface soil and groundwater through laboratory analysis. These concentrations will be compared to the TRRP Tier 1 PCLs listed in Worksheet No. 15.

- If any target analyte concentrations in the receptor exposure units defined for soil or groundwater exceed the PAL, proceed to a Feasibility Study (FS).
- If all target analyte concentrations in the receptor exposure units defined for soil or groundwater, are less than the PAL, recommend no further action (NFA).

Comparison to Ecological Criteria:

To evaluate the potential for ecological impact, a SERA will be completed. The goal of the SERA is to determine whether adverse ecological impacts are present. The methodology for the SERA is presented in Appendix A.

Individual target analyte concentrations will be determined in surface soil (0 - 1 ft) through laboratory analysis by Katahdin. These concentrations will be compared to the ecological PALs listed in Worksheet No. 15.

- If all target analyte concentrations within the individual sample results from in the receptor exposure units defined for soil are less than the PAL, recommend NFA for ecological receptors.
- If any target analyte concentrations within the individual sample results from the receptor exposure units defined for soil exceed the PAL, evaluate the data in accordance with the Screening Level Ecological Risk Assessment (SERA) presented in Appendix A. If the SERA does not indicate risk, recommend NFA for ecological receptors. If the SERA does indicate a risk, proceed to a Baseline Ecological Risk Assessment (BERA) in the FS.

11.5 SPECIFY PERFORMANCE OR ACCEPTANCE CRITERIA

The surface and subsurface soil samples will be collected from areas known to be contaminated and from areas suspected to be uncontaminated in order to delineate the lateral and vertical extent of contamination. The intent is to bound the contamination through collection of a limited number of step-out samples based on comparison of individual sample results to the PALs. The Project Team selected locations and numbers of samples which, based on their experience and judgment, will support the attainment of the stated project objectives. The decision unit size, ½-acre, is based on the TCEQ definition of an exposure area for a commercial/industrial site. The Project Team will evaluate contaminant concentrations and concentration patterns to ensure that contaminants are likely to have been detected if present, and that data are of appropriate quality to support the site investigation.

11.6 DEVELOP THE PLAN FOR OBTAINING DATA

The proposed RI field data collection program for the Skeet Range is presented in detail on Worksheet No. 17.

Figure 11-1
MC Study Goal and Decision Rule Flow Chart for Skeet Range

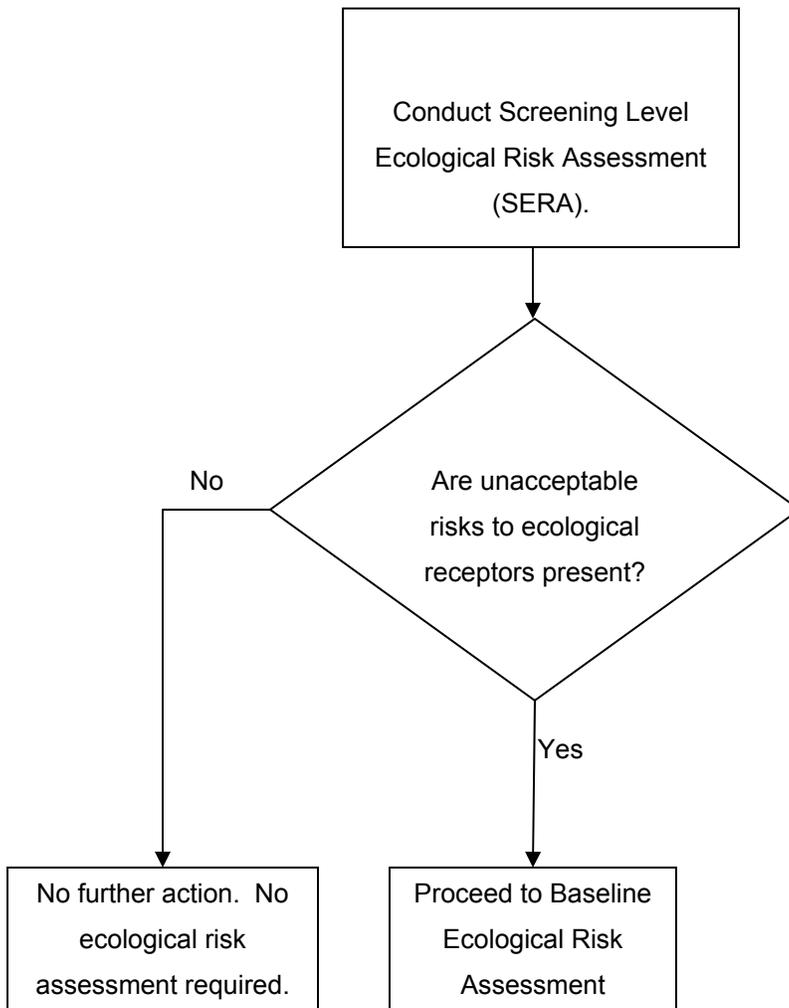
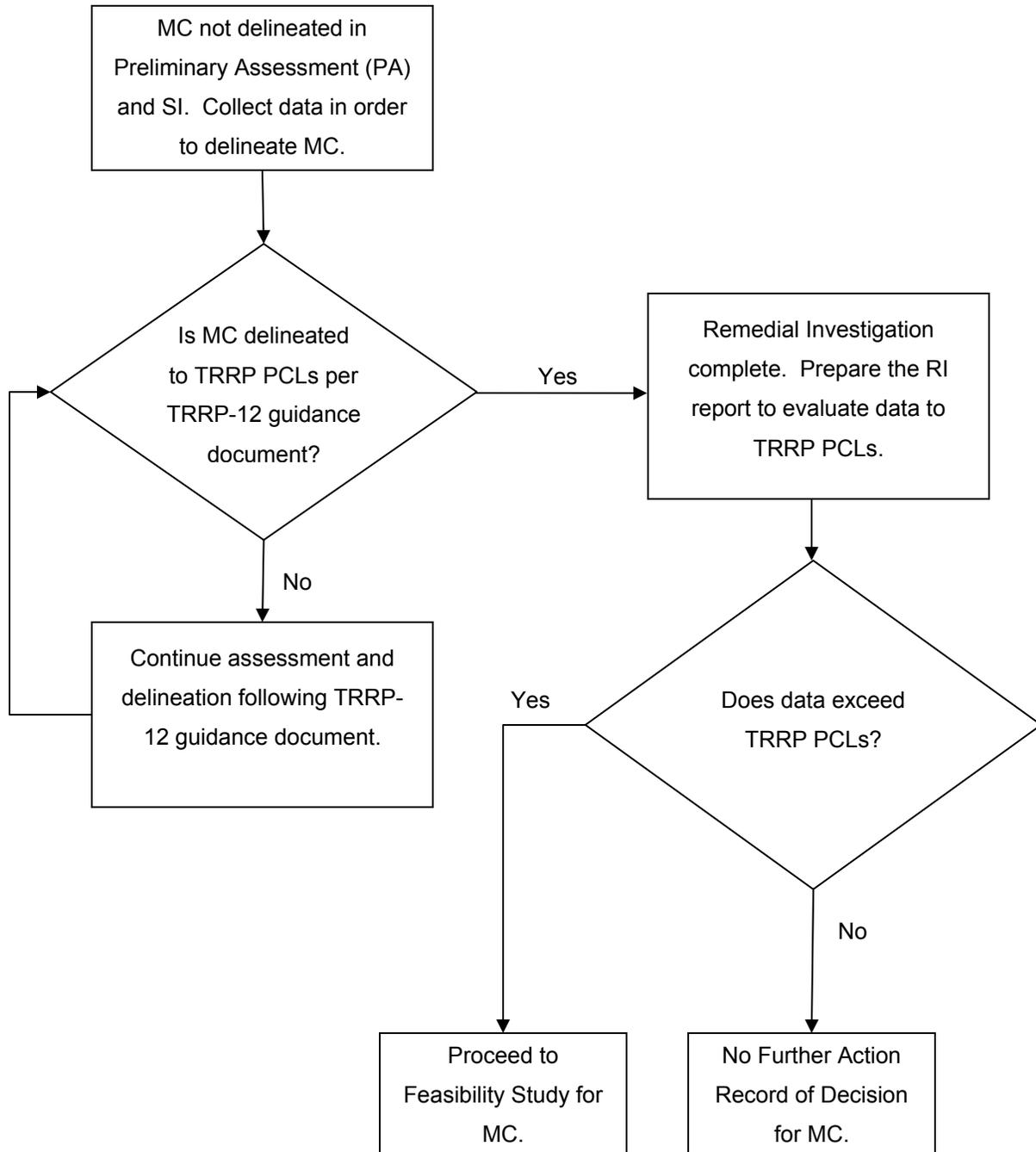


Figure 11-1 (continued)
MC Study Goal and Decision Rule Flow Chart for Skeet Range



SAP Worksheet No. 12 -- Measurement Performance Criteria Table - Field Quality Control Samples

[\(UFP-QAPP Manual Section 2.6.2\)](#)

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 Blank	All Fractions	One per source water	Bias/Contamination	No detections \geq LOQ	S&A
Equipment Rinsate Blanks	All Fractions	One per 20 field samples per matrix per reusable sampling equipment	Bias/Contamination	No analytes \geq $\frac{1}{2}$ LOQ, except common lab contaminants, which must be $<$ LOQ.	S&A
Field Duplicate	All Fractions	One per 10 field samples collected for fixed base laboratory analysis	Precision	Aqueous samples Relative Percent Difference (RPD) of ≤ 30 ; Solid samples RPD of ≤ 50	S
Cooler Temperature Blank	All Fractions	One per cooler	Accuracy / Representativeness	Between 2 and 6 ° C	S

1) Field quality control samples will not be collected for Toxicity Characteristic Leaching procedure (TCLP) metals/organics, or Reactivity, Corrosivity, Ignitability (RCI).

SAP Worksheet No. 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
Site Histories, Initial CSM	Preliminary Assessment, Naval Auxiliary Landing Field Cabaniss, Texas, April 2005	Malcolm Pirnie, Inc	Basis for UFP-SAP, Site Histories, and CSMs	The information is qualitative and no quantitative (site-specific nature and extent of contamination) information is available. The information was used to establish the field work program for the SI and identify areas most likely to be contaminated.
SI Report	Site Inspection Report for the Skeet Range, NALF Cabaniss; Sept 2009.	TtNUS	Basis for UFP-SAP, Site Histories, and CSMs	The information is quantitative. The information was used to establish the field work program for the RI and identify areas most likely to be contaminated.

SAP Worksheet No. 14 -- Summary of Project Tasks

([UFP-QAPP Manual Section 2.8.1](#))

FIELD TASKS

The Skeet Range RI project activities consist of the following tasks:

- Field Tasks, including:
 - Mobilization/demobilization and utility clearance
 - Drilling tasks
 - Sample collection tasks
 - Quality control tasks
 - Field Instrument calibration
 - Equipment decontamination
 - Investigation-derived waste tasks
 - Global Positioning System
- Analytical Tasks
- Data Management
 - Data Tracking
 - Data Storage, Archiving, and Retrieval
 - Data Security
- Assessment and Oversight
- Data Review
- Project Report

These tasks are summarized below. The SOPs and field documents referenced below and in other worksheets are included in Appendix B.

Field Tasks

- **Mobilization/Demobilization:** Mobilization/demobilization activities include field equipment procurement and transport, subcontractor procurement and coordination, utility awareness and clearance, location and setup of areas for decontamination and waste storage, acquisition of vehicles, and establishment of an on-site staging area.

Equipment requirements will be finalized by the FOL following the acceptance of the SAP. The FOL will review the scope of work and assemble equipment (e.g., vehicles, sampling, personal protection, and decontamination equipment) to implement and complete the field investigations. The FOL will be responsible for packaging and loading of equipment, and ensuring that all equipment is operable and calibrated. The FOL will be responsible for receiving and unpacking the equipment and ensuring that all equipment is operable and calibrated.

The FOL will be responsible for tracking equipment used in the field. Analytical laboratory services have been subcontracted. Following the procurement of these services, the FOL will be responsible for coordinating these activities. The TtNUS Project Chemist will be responsible for coordinating the analytical services, as well as the acquisition and delivery of sample containers to the Site.

During mobilization, the FOL will review the roles and responsibilities of each member, and review the requirements of the various field activities. A series of meetings will be conducted to review the sampling and analytical requirements. Upon mobilization, an on-site meeting will be conducted to review health and safety requirements. The SSO will be responsible for reviewing the HASP with the field team members and subcontractors.

- **Utility Clearance Tasks:** Prior to conducting any subsurface intrusive investigations, NASCC personnel will be contacted and a Digging permit will be obtained. Texas One Call will also be notified for subsurface utility clearance, in accordance with State law. The FOL will be responsible for coordinating these activities.
- **Drilling Tasks:** Soil borings and monitoring wells will be installed in accordance with SOP-16 (Temporary Monitoring Well Installation). Monitoring wells will be developed in accordance with SOP-15 (Monitoring Well Development).
- **Sample Collection Tasks:** Soil sampling activities will be conducted in accordance with SOP-05 (Soil Coring and Sampling Using Hand Augering Techniques). Soil samples will be logged in accordance with SOP-10 (Borehole and Soil Sample Logging). Groundwater samples will be collected in accordance with SOP-17 (Low-Flow Well Purging and Stabilization) and SOP-18 (Groundwater Sampling). The sample numbering scheme will be in accordance with SOP-02 (Sample Identification and Nomenclature). Methods for recording data are included in each SOP and SOP-03 (Sample Custody and Documentation of Field Activities). Sample labeling will be in accordance with SOP-01 (Sample Labeling), and sample containers, preservation, packaging, and shipping will be in accordance with SOP-11 (Sample Preservation, Packaging, and Shipping).

Surface soil samples (considered to be within 0-1 feet bgs) will be collected using a hand auger, hand trowel, or disposal sampler. Subsurface soil samples (considered to be greater than 1 feet bgs) will be collected using a hand auger or drilling rig. All subsurface soil samples from ground surface to depth will be a composite of one sample over the given sample's depth interval. The sample will be composited in one container. Groundwater samples will be collected from the temporary monitoring wells using low-flow techniques.

The numbers and types of samples to be collected along with associated analytical programs are presented in Worksheet No. 18. Equipment blanks, field blanks, field duplicates, matrix spikes, and matrix spike duplicates will be collected as presented in Worksheet No. 20.

- **Quality Control Tasks:** Equipment blanks, field blanks, field duplicates, matrix spikes, and matrix spike duplicates will be collected as presented in Worksheet No. 20.

Initial and continuing calibrations, tuning, reagent blanks, surrogates, duplicates, laboratory control samples, and all other applicable QC for all analytical methods is presented in Worksheet Nos. 24 and 28.

- **Field Instrument Calibration:** These procedures are described in Worksheet No. 22.
- **Equipment Decontamination:** All reusable sampling equipment (e.g., stainless steel trowels, hand auger, etc.) will be decontaminated prior to sampling and between samples, according to the sequence established in SOP-04 (Decontamination of Field Sampling Equipment).

Decontamination will generally consist of a tap water rinse to remove gross contamination, followed by a non-phosphate detergent (e.g., Alconox) water rinse, a tap water rinse, and finally a deionized water rinse. If equipment is to be stored or transported, it should be wrapped in aluminum foil after air-drying. Disposable sampling equipment will be used where practical to minimize the need for decontamination.

- **Investigation-Derived Waste Tasks:** It is anticipated that waste materials will be generated during the field investigation. Investigation-derived waste (IDW) generated during the RI will be stored on site and will remain there until waste characterization results are available and disposal is implemented. Drums of IDW will be stored at NALF Cabaniss at a location designated by the Navy.

Waste profiling will be completed as follows:

- Liquids - One IDW aliquot will be collected from each drum of liquids and composited into one liquid sample. The liquid sample will be analyzed for TCLP metals, TCLP volatile organic compounds (VOCs), and RCI.

- Soil - One soil aliquot will be collected from each drum and composited into one soil sample. The soil sample will be analyzed for TCLP metals, TCLP VOCs, and RCI.

Upon completion of waste profiling activities, the results will be transmitted to NASCC personnel who will be responsible for the manifesting and disposal of the IDW. SOP-09 (Management of Investigation-Derived Waste) provides information on the handling of investigation-derived waste.

Global Positioning System (GPS): A hand-held GPS unit capable of sub-meter accuracy (i.e., Trimble GeoXH) will be used to locate sampling points. The GPS coordinate system will be set up so all data points are collected in North American Datum of 1983 (NAD83) Texas State Plane coordinates in US survey feet. A surveyor licensed in the State of Texas will be used to obtain vertical and horizontal locations of the temporary monitoring wells.

Analytical Tasks - Chemical analysis for PAHs will be performed by the subcontracted laboratory, Katahdin Analytical Services of Scarborough, Maine. Katahdin Analytical Services is DoD Environmental Laboratory Accreditation Program (ELAP) certified and National Environmental Laboratory Accreditation Program (NELAP) accredited. Analyses will be performed in accordance with the analytical methods identified in Worksheet No. 30. Katahdin will meet the PALs specified in Worksheet No. 15. Katahdin will perform the chemical analyses following laboratory-specific SOPs (Worksheet Nos. 19 and 23) developed based on the methods listed in Worksheet Nos. 19 and 30. Copies of the laboratory SOPs are included in Appendix C.

Data Management

- Project documentation and records
 - Field sample collection and field measurement records are described in Worksheet Nos. 27 and 29.
 - Laboratory data package deliverables are described in the analytical specifications.

- Data assessment documents and records are listed in Worksheet No. 29.
- Data recording formats are described in Worksheet No. 27.

Data Handling and Management - After the field investigation is completed, the field sampling log sheets will be organized by date and media and filed in the project files. The field logbooks for this project will be used only for this site, and will also be categorized and maintained in the project files after the completion of the field program. Project personnel completing concurrent field sampling activities may maintain multiple field logbooks. When possible, logbooks will be segregated by sampling activity. The field logbooks will be titled based on date and activity. The data handling procedures to be followed by the laboratory will meet the requirements of the technical specification. The electronic data results will be automatically downloaded into the TtNUS database in accordance with proprietary TtNUS processes.

Data Tracking and Control. The TtNUS TOM (or designee) is responsible for the overall tracking and control of data generated for the project.

- **Data Tracking.** Data is tracked from its generation to its archiving in the TtNUS project-specific files. The TtNUS Project Chemist (or designee) is responsible for tracking the samples collected and shipped to the contract laboratory. Upon receipt of the data packages from the analytical laboratory, the Project Chemist will oversee the data validation effort, which includes verifying that the data packages are complete and results for all samples have been delivered by the analytical laboratory.
- **Data Storage, Archiving, and Retrieval.** The data packages received from the subcontract laboratory are tracked in the data validation log book. After the data are validated, the data packages are entered into the TtNUS CLEAN file system and archived in secure files. The field records including field log books, sample logs, chain-of-custody records, and field calibration logs will be submitted by the FOL to be entered into the CLEAN file system prior to archiving in secure project files. The project files are audited for accuracy and completeness. At the completion of the Navy contract, the records will be stored by TtNUS.
- **Data Security.** The TtNUS project files are restricted to designated personnel only. Records can only be borrowed temporarily from the project file using a sign-out system. The TtNUS Data Manager maintains the electronic data files. Access to the data files is restricted to qualified personnel only. File and data backup procedures are routinely performed.

Assessment and Oversight – Refer to Worksheet No. 32 for assessment findings and corrective actions and Worksheet No. 33 for QA management reports.

Data Review

- Data verification is described in Worksheet No. 34.
- Data validation is described in Worksheet Nos. 35 and 36.
- Usability assessment is described in Worksheet No. 37.

Project Report - Draft and Final versions of the project report will be prepared and submitted to the Navy, EPA, and TCEQ for review. The report will be prepared following TCEQ format for Affected Property Assessment Reports to the extent practicable for reporting the data. The reports will include the following sections:

- Executive Summary – a very brief description of the work conducted and the findings.
- Introduction and Background – includes a description of the history of operations and activities at the Site and a summary of any previous investigations and removal actions.
- Description of Field Investigations – includes a summary of the RI work performed in the approved SAP and any field modifications as documented by the FOL. This section will include maps showing the sampling locations and tables summarizing the RI data collected.
- Data quality – includes a summary of quantitative analytical performance indicators such as completeness, precision, bias and sensitivity, as well as qualitative indicators such as representativeness and comparability. Includes a reconciliation of project data with the DQOs and an identification of deviations from this SAP.

A data usability assessment will be used to identify significant deviations in analytical performance that could affect the ability to meet project objectives. The elements of this review are presented in Worksheet No. 37.

- Nature and Extent of Contamination – includes the contamination previously found in each medium sampled in relation to the conceptual model of the site. This section will address any additional contaminants found during this effort compared to the SI.

- Detected contaminant concentrations will be tabulated for each medium and depicted on maps.
- Contaminant Fate and Transport – includes a description of the contaminants detected and their behavior in the soil, particularly with emphasis on the future migration of these contaminants to any possible exposure areas.
- Summary and Conclusions – this section will summarize the findings, conclude whether delineation of contamination is adequate, and recommend further sampling if needed.

TtNUS will respond to comments received on the draft report. The final version of the report will be submitted in hardcopy and electronic format to the project stakeholders.

SAP Worksheet No. 15 -- Reference Limits and Evaluation Table

(UFP-QAPP Manual Section 2.8.1)

Matrix: Surface Soil (0 – 1 ft bgs)

Analytical Group: PAHs (scan)

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit ⁽¹⁾ (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Katahdin ⁽²⁾		
					LOQ (mg/kg)	LOD ⁽³⁾ (mg/kg)	Method Detection Limit (MDL) (mg/kg)
PAHs							
1-Methylnaphthalene	90-12-0	29	Ecological Screening Level	10	0.33	0.16	0.12
2-Methylnaphthalene	91-57-6	29	Ecological Screening Level	10	0.33	0.16	0.092
Acenaphthene	83-32-9	20	Ecological Screening Level	7	0.33	0.16	0.065
Acenaphthylene	208-96-8	29	Ecological Screening Level	10	0.33	0.16	0.070
Anthracene	120-12-7	29	Ecological Screening Level	10	0.33	0.16	0.084
Benzo(a)anthracene	56-55-3	5.7	TRRP Soil PCL	1.9	0.33	0.16	0.086
Benzo(a)pyrene	50-32-8	0.56	TRRP Soil PCL	0.18	0.33	0.16	0.093
Benzo(b)fluoranthene	205-99-2	5.7	TRRP Soil PCL	1.9	0.33	0.16	0.134
Benzo(g,h,i)perylene	191-24-2	18	Ecological Screening Level	6	0.33	0.16	0.10
Benzo(k)fluoranthene	207-08-9	18	Ecological Screening Level	6	0.33	0.16	0.083

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit ⁽¹⁾ (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Katahdin ⁽²⁾		
					LOQ (mg/kg)	LOD ⁽³⁾ (mg/kg)	Method Detection Limit (MDL) (mg/kg)
Chrysene	218-01-9	18	Ecological Screening Level	6	0.33	0.16	0.095
Dibenzo(a,h)anthracene	53-70-3	0.55	TRRP Soil PCL	0.18	0.33	0.16	0.13
Fluoranthene	206-44-0	29	Ecological Screening Level	10	0.33	0.16	0.11
Flourene	86-73-7	29	Ecological Screening Level	10	0.33	0.16	0.081
Indeno(1,2,3-cd)pyrene	193-39-5	5.7	TRRP Soil PCL	1.9	0.33	0.16	0.12
Naphthalene	91-20-3	29	Ecological Screening Level	10	0.33	0.16	0.087
Phenanthrene	85-01-8	29	Ecological Screening Level	10	0.33	0.16	0.083
Pyrene	129-00-0	18	Ecological Screening Level	6	0.33	0.16	0.10

Matrix: Subsurface Soil (>1 ft bgs)

Analytical Group: PAHs (scan)

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit ⁽¹⁾ (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Katahdin ⁽²⁾		
					LOQ (mg/kg)	LOD ⁽³⁾ (mg/kg)	Method Detection Limit (MDL) (mg/kg)
PAHs							
1-Methylnaphthalene	90-12-0	150	TRRP Soil PCL	50	0.33	0.16	0.12
2-Methylnaphthalene	91-57-6	250	TRRP Soil PCL	83	0.33	0.16	0.092
Acenaphthene	83-32-9	3000	TRRP Soil PCL	1000	0.33	0.16	0.065
Acenaphthylene	208-96-8	3800	TRRP Soil PCL	1254	0.33	0.16	0.070
Anthracene	120-12-7	18000	TRRP Soil PCL	5940	0.33	0.16	0.084
Benzo(a)anthracene	56-55-3	5.7	TRRP Soil PCL	1.9	0.33	0.16	0.086
Benzo(a)pyrene	50-32-8	0.56	TRRP Soil PCL	0.18	0.33	0.16	0.093
Benzo(b)fluoranthene	205-99-2	5.7	TRRP Soil PCL	1.9	0.33	0.16	0.134
Benzo(g,h,i)perylene	191-24-2	1800	TRRP Soil PCL	594	0.33	0.16	0.10
Benzo(k)fluoranthene	207-08-9	57	TRRP Soil PCL	18.8	0.33	0.16	0.083
Chrysene	218-01-9	560	TRRP Soil PCL	185	0.33	0.16	0.095

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit ⁽¹⁾ (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Katahdin ⁽²⁾		
					LOQ (mg/kg)	LOD ⁽³⁾ (mg/kg)	Method Detection Limit (MDL) (mg/kg)
Dibenzo(a,h)anthracene	53-70-3	0.55	TRRP Soil PCL	0.18	0.33	0.16	0.13
Fluoranthene	206-44-0	2300	TRRP Soil PCL	759	0.33	0.16	0.11
Flourene	86-73-7	2300	TRRP Soil PCL	759	0.33	0.16	0.081
Indeno(1,2,3-cd)pyrene	193-39-5	5.7	TRRP Soil PCL	1.9	0.33	0.16	0.12
Naphthalene	91-20-3	220	TRRP Soil PCL	72.6	0.33	0.16	0.087
Phenanthrene	85-01-8	1700	TRRP Soil PCL	561	0.33	0.16	0.083
Pyrene	129-00-0	1700	TRRP Soil PCL	561	0.33	0.16	0.10

Matrix: Groundwater

Analytical Group: PAHs (SIM)

Analyte	CAS Number	Project Action Limit ⁽¹⁾ (mg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/L)	Katahdin ⁽²⁾		
					LOQ (mg/L)	LOD ⁽³⁾ (mg/L)	MDL (mg/L)
PAHs							
1-Methylnaphthalene	90-12-0	0.031	TRRP Groundwater PCL	0.010	0.0002	0.00010	0.000068
2-Methylnaphthalene	91-57-6	0.098	TRRP Groundwater PCL	0.032	0.0002	0.00010	0.000077
Acenaphthene	83-32-9	1.5	TRRP Groundwater PCL	0.5	0.0002	0.00010	0.000064
Acenaphthylene	208-96-8	1.5	TRRP Groundwater PCL	0.5	0.0002	0.00010	0.000054
Anthracene	120-12-7	7.3	TRRP Groundwater PCL	2.4	0.0002	0.00010	0.000044
Benzo(a)anthracene	56-55-3	0.0013	TRRP Groundwater PCL	0.00042	0.0002	0.00010	0.000046
Benzo(a)pyrene	50-32-8	0.0002	TRRP Groundwater PCL	0.000066	0.0002	0.00010	0.000066
Benzo(b)fluoranthene	205-99-2	0.0013	TRRP Groundwater PCL	0.00042	0.0002	0.00010	0.000089
Benzo(g,h,i)perylene	191-24-2	0.73	TRRP Groundwater PCL	0.24	0.0002	0.00010	0.000065
Benzo(k)fluoranthene	207-08-9	0.013	TRRP Groundwater PCL	0.0042	0.0002	0.00010	0.000049
Chrysene	218-01-9	0.13	TRRP Groundwater PCL	0.042	0.0002	0.00010	0.000036
Dibenzo(a,h)anthracene	53-70-3	0.0002	TRRP Groundwater PCL	0.000066	0.0002	0.00010	0.00007
Fluoranthene	206-44-0	0.98	TRRP Groundwater PCL	0.323	0.0002	0.00010	0.000073

Analyte	CAS Number	Project Action Limit ⁽¹⁾ (mg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/L)	Katahdin ⁽²⁾		
					LOQ (mg/L)	LOD ⁽³⁾ (mg/L)	MDL (mg/L)
Flourene	86-73-7	0.98	TRRP Groundwater PCL	0.323	0.0002	0.00010	0.000061
Indeno(1,2,3-cd)pyrene	193-39-5	0.0013	TRRP Groundwater PCL	0.000429	0.0002	0.00010	0.000052
Naphthalene	91-20-3	0.49	TRRP Groundwater PCL	0.161	0.0002	0.00010	0.000064
Phenanthrene	85-01-8	0.73	TRRP Groundwater PCL	0.240	0.0002	0.00010	0.000051
Pyrene	129-00-0	0.73	TRRP Groundwater PCL	0.240	0.0002	0.00010	0.000059

Abbreviations:

CAS = Chemical Abstracts Service
 LOD = Limits of Detection
 MDL = Method Detection Limit
 mg/kg = milligrams per kilograms
 mg/L = milligrams per liter
 PCL = Protective Concentration Level
 LOQ = Limit of Quantitation
 TRRP = Texas Risk Reduction Program

Footnotes:

- 1 Project Action Limits (PALs) are from TRRP Table 1, Tier 1 Residential Soil PCLs for total soil combined pathway or TRRP Table 3, Tier 1 Residential Groundwater PCLs for groundwater ingestion pathway, March 31, 2010. PALs for ecological are from Table 1 of Appendix A.
- 2 The Laboratory LOQs, LODs and MDLs for Katahdin Analytical Services are presented.
- 3 Limits of detection are provided for informational purposes as TCEQ requires that data be reported to the LOQ and MDL.

SAP Worksheet No. 16 -- Project Schedule/Timeline Table (optional format)

(UFP-QAPP Manual Section 2.8.2)

Activity	Organization	Dates (MM/YY)	
		Anticipated Date(s) of Initiation	Anticipated Date of Completion
Submit Final RI Work Plan and Appendices	TtNUS	--	10/10
Field Investigation	TtNUS	11/10	01/11
Laboratory Analysis	Katahdin	11/10	03/11
Data Validation	TtNUS	12/10	05/11
Database Entry	TtNUS	04/11	06/11
Prepare Rough Draft RI Report	TtNUS	02/11	07/11
Submit Rough Draft RI Report and Appendices	TtNUS	--	07/11
Navy Review	Navy	07/11	08/11
Receive Comments/Comment Resolution	TtNUS	08/11	08/11
Prepare Draft RI Report	TtNUS	08/11	09/11
Submit Draft RI Report	TtNUS	--	09/11
Navy and Regulator Review	Navy, USEPA, and TCEQ	09/11	10/11
Receive Comments/Comment Resolution	TtNUS	10/11	10/11
Prepare Final RI Report	TtNUS	10/11	11/11
Submit Final RI Report	TtNUS	--	11/11

Note: Field activities will be coordinated with RI activities at the Incinerator Disposal Site.

SAP Worksheet No. 17 -- Sampling Design and Rationale

[\(UFP-QAPP Manual Section 3.1.1\)](#)

SKEET RANGE SAMPLING LOCATIONS, ANALYSES, AND RATIONALES

This section describes sampling locations, analytical methods, and rationale for the sampling activities to be conducted in support of the Skeet Range RI at NALF Cabaniss.

During brush clearing operations in the SI, one MEC item, a smoke cartridge, was discovered. Because of this discovery, a UXO technician will be onsite during RI MC sampling to conduct UXO avoidance activities. Although additional MEC is not expected within the Skeet Range, if MEC is observed in or around any work area, work must be temporarily halted. The presence of MEC must be communicated to the FOL, and the FOL will then communicate with the NASCC Personnel so the appropriate action may be taken before proceeding.

The sampling design consists of samples within grids as shown on Figure 17-1. The sampling objective is to gather the necessary information to determine the extent of site-specific MC present in soil and groundwater. The RI will be conducted in accordance with the TRRP-12 guidance document, Affected Property Assessment Requirements. This document outlines the requirements for defining the three-dimensional extent of the affected property and PCL exceedance zone pursuant to the TRRP rule.

All soil sample locations will be marked with a stake or a brightly colored pin flag indicating the sample location. Coordinates will be determined by a handheld sub-meter accuracy GPS at each individual sample location, which will allow for future studies or guide in any removal action. Pre-determined GPS sample coordinates may be utilized in locating proposed sample locations.

Soil Sampling Strategy – Skeet Range

The chosen sampling strategy employs grid sampling to target those areas that were identified during the SI as being potentially impacted with MC. A grid pattern for soil sampling will be utilized as shown on Figure 17-1. Actual sample locations may vary from the proposed locations based upon field conditions observed during an initial site walkover by the TtNUS FOL, provided that the locations remain in the estimated site areas as depicted on the figure. The MC sampling and analytical program for soil samples is as follows.

Horizontal Delineation

The chosen sampling strategy employs a grid pattern to target and expand outward from those areas that were identified during the SI as being impacted with MC and to determine the extent of MC. The grid size will be approximately 150 feet by 150 feet (0.5-acres). Due to the geometry of the site, some grids may be smaller or larger in size and irregularly shaped. Figure 17-1 depicts the sample grid. A total of 7 grids will be sampled. The grid pattern used is an extension of the fourteen grids used during the SI.

Up to five surface soil samples will be collected within each grid. Surface soil samples will be collected from 0- to 1-foot bgs. The surface soil samples will be placed into individual plastic bags. The surface soil samples within each grid will be composited into one sample. The composite sample will be prepared by mixing a portion of each subsample in a plastic bag. The composite soil sample from each grid will be submitted to the fixed-base laboratory for analysis. The composite samples will be analyzed for PAHs. The subsamples from each grid will also be submitted to the laboratory but will be held pending results of the composite sample. Any excess soil will be returned to the sample location.

Based on the analytical results, up to two additional step out samples may be required to determine the horizontal extent of contamination.

Vertical Delineation

The chosen sampling strategy employs a judgmental design to target those areas that were identified during the SI as being impacted with PAHs. Based on the SI results, the three areas with the highest PAH concentrations will be delineated vertically.

At the selected locations, a drilling rig will be used to advance a soil boring to the top of groundwater and temporary monitoring wells will be installed. The locations of the soil borings are shown on Figure 17-1. The soil boring/monitoring well will allow for the collection of soil and groundwater samples to determine the vertical extent of PAH contamination and determine if groundwater has been impacted by PAHs. In addition, water level measurements will allow for the construction of groundwater gradient maps.

Subsurface soil samples will be collected from 2 to 4, 5 to 7 and 10 to 12 feet bgs for analysis by Katahdin. Soil samples collected will be analyzed for PAHs. Subsurface soil samples will be discrete samples and will not be composited. A temporary groundwater monitoring well will be installed in each soil boring to collect a groundwater sample. Groundwater samples will be analyzed for PAHs and total dissolved solids (TDS). TDS results will be used to classify the groundwater resource category according to the TRRP rule.

Based on the analytical results, up to two additional step out samples may be required to determine the vertical extent of contamination.

Sampling and Analytical Program - Skeet Range

The MC sampling and analytical program is as follows:

Soil Sampling

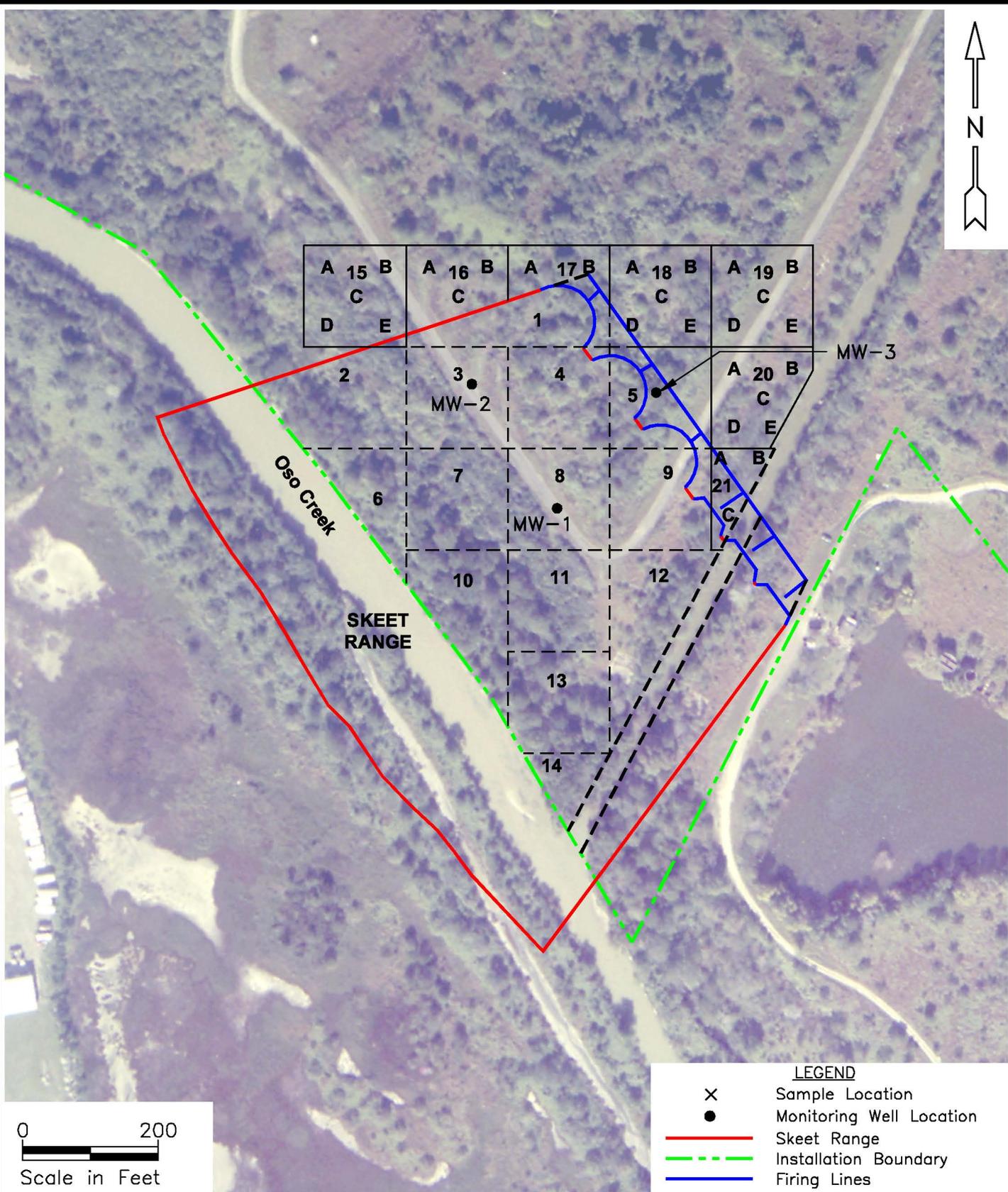
Surface soil samples will be collected in the areas to be sampled as shown on Figure 17-1. Additionally a site walk over by the TtNUS FOL will be conducted to identify any obvious areas of skeet fragment accumulation. Up to 10 additional discretionary surface soil samples may be collected based on visual observation of accumulated skeet fragments in the areas to be sampled. Emphasis will be placed on collecting samples required to delineate contaminated areas.

Groundwater Sampling

Groundwater samples will be collected from temporary monitoring wells in the areas to be sampled as shown on Figure 17-1.

Analytical Program

- Seven composite surface soil samples (one from each grid) will be analyzed for PAHs (8270C).
- Up to 10 discretionary surface soil samples may also be collected and analyzed for PAHs (8270C).
- Three discrete subsurface soil samples will be collected from each of three soil borings at the three new temporary monitoring well locations and analyzed for PAHs (8270C).
- Three groundwater samples (one from each temporary monitoring well) will be analyzed for PAHs (8270 SIM) and TDS.



LEGEND	
X	Sample Location
●	Monitoring Well Location
— (Red)	Skeet Range
- - - (Green)	Installation Boundary
— (Blue)	Firing Lines

DRAWN BY GS	DATE 10/15/10
CHECKED BY LB	DATE 10/15/10
REVISED BY	DATE
SCALE AS NOTED	



SAMPLE LOCATIONS
SKEET RANGE
NALF CABANISS, TEXAS

PROJECT NO. 112G01821	
CTO NO. 0135	
APPROVED BY	DATE
DRAWING NO. FIGURE 17-1	REV. 0

SAP Worksheet No. 18 -- Sampling Locations and Methods/SOP Requirements Table for the Skeet Range

(UFP-QAPP Manual Section 3.1.1)

Grid Number	Subsample Identification	Sample Identification ¹	Depth ^{2,3} (feet bgs)	PAHs
				SW-846 8270C (PAHs)
SURFACE SOIL SAMPLES				
15	SR-SS015a	SR-SS015	0.0 – 1.0	X
15	SR-SS015b		0.0 – 1.0	
15	SR-SS015c		0.0 – 1.0	
15	SR-SS015d		0.0 – 1.0	
15	SR-SS015e		0.0 – 1.0	
16	SR-SS016a	SR-SS016	0.0 – 1.0	X
16	SR-SS016b		0.0 – 1.0	
16	SR-SS016c		0.0 – 1.0	
17	SR-SS017a	SR-SS017	0.0 – 1.0	X
17	SR-SS017b		0.0 – 1.0	
18	SR-SS018a	SR-SS018	0.0 – 1.0	X
18	SR-SS018b		0.0 – 1.0	
18	SR-SS018c		0.0 – 1.0	
18	SR-SS018d		0.0 – 1.0	
18	SR-SS018e		0.0 – 1.0	
19	SR-SS019a	SR-SS019	0.0 – 1.0	X
19	SR-SS019b		0.0 – 1.0	
19	SR-SS019c		0.0 – 1.0	
19	SR-SS019d		0.0 – 1.0	
19	SR-SS019e		0.0 – 1.0	

Grid Number	Subsample Identification	Sample Identification ¹	Depth ^{2,3} (feet bgs)	PAHs
				SW-846 8270C (PAHs)
20	SR-SS020a	SR-SS020	0.0 – 1.0	X
20	SR-SS020b		0.0 – 1.0	
20	SR-SS020c		0.0 – 1.0	
20	SR-SS020d		0.0 – 1.0	
20	SR-SS020e		0.0 – 1.0	
21	SR-SS021a	SR-SS021	0.0 – 1.0	X
21	SR-SS021b		0.0 – 1.0	
21	SR-SS021c		0.0 – 1.0	
SUBSURFACE SOIL SAMPLES				
3	SR-SB003-0204	SR-SB003	2 - 4	X
3	SR-SB003-0507	SR-SB003	5 - 7	X
3	SR-SB003-1012	SR-SB003	10 - 12	X
5	SR-SB005-0204	SR-SB005	2 - 4	X
5	SR-SB005-0507	SR-SB005	5 - 7	X
5	SR-SB005-1012	SR-SB005	10 - 12	X
8	SR-SB008-0204	SR-SB008	2 - 4	X
8	SR-SB008-0507	SR-SB008	5 - 7	X
8	SR-SB008-1012	SR-SB008	10 - 12	X

Grid Number	Subsample Identification	Sample Identification ¹	Depth ^{2,3} (feet bgs)	PAHs
				SW-846 8270C (PAHs)
GROUNDWATER SAMPLES ⁴				
3	NA	SR MW01	NA	X
5	NA	SR MW02	NA	X
8	NA	SR MW03	NA	X

SR = Skeet Range

PAHs = Polycyclic aromatic hydrocarbons

SS = Surface Soil Sample

SB = Subsurface Soil Sample

MW = Monitoring Well Sample

NA = Not Applicable

TBD = To be determined

- 1 Surface soil samples will be collected over a grid interval. Proposed sample locations are shown on Figure 17-1.
- 2 Surface soil sample depth to a maximum of 12 inches bgs for soils. All samples will be collected in accordance with the respective SOP (Soil SOP-05).
- 3 Subsurface soil samples will be collected using a drilling rig. Proposed sample locations are the same as for the temporary monitoring wells shown on Figure 17-1.
- 4 Groundwater will also be analyzed for total dissolved solids.

Note: Two duplicates and one Matrix Spike/Matrix Spike Duplicate (MS/MSD) samples will be collected. Locations for these samples will be selected in the field by the FOL.

Additional samples collected (discretionary and/or step out) will follow the convention listed in the worksheet and will be determined in the field by the FOL.

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 (Groundwater and Field QA Samples)	SVOCs (PAHs only)	SW-846 3510C/3520/8270C SIM, CA-502/CA-213	Two 1-liter glass amber bottles	1,000 milliliters	Cool to 4 (±2) °C	7 days to extraction 40 days to analysis
Solid (Soil)	SVOCs (PAHs only)	SW-846 3540C/3550/8270C, CA- 526/CA-512/CA-204	One 4-ounce glass	15 grams	Cool to 4 (±2) °C	7 days to extraction, 40 days to analysis
Solid IDW	TCLP VOCs ⁽²⁾	SW-846 1311/3510C/8260B/8270C /8081A/ 8151B, CA-209/CA-510/CA- 502/CA-515/CA-202/CA- 204/CA-302/CA-305	4-ounce glass	100 grams	Cool to 4 (±2) °C	14 days to TCLP extraction/7 days to extraction for SVOC, 40 days analysis/ 14 days for volatiles to analysis.
Solid IDW	TCLP Metals ⁽²⁾	SW-846 1311/3010/6010B/ 7470A, CA-510/CA-604/CA-615	4-ounce glass	100 grams	Cool to 4 (±2) °C	14 days to TCLP extraction/180 days to analysis, 28 days for mercury
Aqueous IDW	RCI ⁽²⁾	SW-846 Ch. 7.7.3.2, 7.3.4.2, 9040C, 1010A / CA-708, CA-733, CA-734, CA-736	250 milliliter plastic	100 milliliters - Cyanide and Sulfide, 25 milliliters - pH,	Cool to 4 (±2) °C	Cyanide-14 days to analysis, sulfide- 7 days

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)
				50 milliliters - Ignitability		to analysis, Ignitability and pH immediately
Solid IDW	RCI ⁽²⁾	SW-846 Ch. 7.7.3.2, 7.3.4.2, 9045D, 1010A / CA-733, CA-734, CA-709, CA-736	4-8 oz glass soil jar	10 grams - Cyanide and Sulfide, 20 grams - pH, 50 grams - Ignitability	Cool to 4 (±2) °C	14 days Cyanide/ to analysis 7 days Sulfide analysis /immediately for Ignitability. & pH
Groundwater	TDS	SM2540C	250 milliliter plastic	100 milliliters	Cool to 4 (±2) °C	7 days to analysis

- 1 Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.
- 2 Quality control information is not presented in the following worksheets as these are IDW analyses. These analyses are presented in this worksheet to aid in field sampling.

SOP = Standard Operating Procedure
 QA = Quality Assurance
 IDW = Investigation-derived waste
 SVOCs = Semivolatile Organic Compounds
 PAH = Polycyclic Aromatic Hydrocarbon
 VOC = Volatile Organic Compound
 TCLP = Toxicity Characteristic Leaching Procedure
 RCI = Reactivity, Corrosivity, Ignitability
 TDS = Total Dissolved Solids

SAP Worksheet No. 20 -- Field Quality Control Sample Summary Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Concentration Level	No. of Samples ²	No. of Field Duplicates ³	No. of MS/MSDs ¹	No. of Equip. Blanks	Total No. of Samples to Lab ⁴
Solid	PAHs	Low to Moderate	28	3	2	1	32
Groundwater	PAHs	Low to Moderate	3	1	1	1	6
Solid IDW	TCLP Organics	Low to Moderate	1	NA	NA	NA	1
Solid IDW	TCLP Metals	Low to Moderate	1	NA	NA	NA	1
Solid IDW	RCI	Low to Moderate	1	NA	NA	NA	1
Aqueous IDW	TCLP Organics	Low to Moderate	1	NA	NA	NA	1
Aqueous IDW	TCLP Metals	Low to Moderate	1	NA	NA	NA	1
Aqueous IDW	RCI	Low to Moderate	1	NA	NA	NA	1

Matrix Spike/Matrix Spike Duplicates (MS/MSDs) will be collected at a frequency of 1 per 20 samples.

¹ Although the MS/MSD is not a field QC it is included here because location determination is often established in the field.

² Minimum number of samples. Additional samples may be collected at different depths at the same location, with each discrete sampling depth counting as a separate sampling location or station.

³ Duplicates will be collected at a frequency of 1 per 10 samples.

⁴ Total number of samples does not include MS/MSD samples.

Note: Field sample identifications are provided in Worksheet No. 18. QC sample identifications will be in accordance with SOP-02 (Sample Identification Nomenclature).

IDW = Investigation-derived waste

PAH = Polycyclic Aromatic Hydrocarbon

TCLP = Toxicity Characteristic Leaching Procedure

RCI = Reactivity, Corrosivity, Ignitability

SAP Worksheet No. 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
SOP-01	Sample Labeling	TtNUS	NA	N	SOP Contained in Appendix B
SOP-02	Sample Identification Nomenclature	TtNUS	NA	N	SOP Contained in Appendix B
SOP-03	Sample Custody and Documentation of Field Activities	TtNUS	NA	N	SOP Contained in Appendix B
SOP-04	Decontamination of Field Sampling Equipment	TtNUS	NA	N	SOP Contained in Appendix B
SOP-05	Soil Coring and Sampling Using Hand Auger Techniques	TtNUS	NA	N	SOP Contained in Appendix B
SOP-09	Management of Investigation-Derived Waste	TtNUS	NA	N	SOP Contained in Appendix B
SOP-10	Borehole and Soil Sample Logging	TtNUS	NA	N	SOP Contained in Appendix B
SOP-11	Sample Preservation, Packaging, and Shipping	TtNUS	NA	N	SOP Contained in Appendix B
SOP-15	Monitoring Well Development	TtNUS	NA	N	SOP Contained in Appendix B
SOP-16	Temporary Monitoring Well Installation	TtNUS	NA	N	SOP Contained in Appendix B
SOP-17	Low-Flow Well Purging and Stabilization	TtNUS	NA	N	SOP Contained in Appendix B
SOP-18	Groundwater Sampling	TtNUS	NA	N	SOP Contained in Appendix B

SOP = Standard Operating Procedure
 NA = Not applicable

SAP Worksheet No. 22 -- Field Equipment Calibration, Maintenance, Testing, and Inspection Table

[\(UFP-QAPP Manual Section 3.1.2.4\)](#)

Field Equipment	Activity¹	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference	Comments
GPS	Positioning	Beginning and end of each day used	Accuracy: sub-meter horizontal dilution of precision (HDOP)<3, number of satellites at least six	Wait for better signal, replace unit, or choose alternate location technique	FOL	NA	Follow manufacturers guidance
Multi-Parameter Water Quality Meter	Calibrated in accordance with manufacturer's specifications	Prior to daily use	Prepared standards	Operator correction or replacement	FOL	NA	Follow manufacturers guidance
Water Level Indicator	Testing	Once upon receipt from vendor	0.01-foot accuracy	Operator correction or replacement	FOL	NA	Follow manufacturers guidance

¹ Activities may include: calibration, verification, testing, maintenance.

GPS = Global Positioning

SOP = Standard Operating Procedure

SAP Worksheet No. 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)
CA-101	Equipment Maintenance, 08/09, Revision 8.	Definitive	Various	Various	Katahdin Analytical Services, Inc.	N
CA-103	Balance Calibration, 08/09, Revision 6.	Definitive	Various	Various	Katahdin Analytical Services, Inc.	N
CA-213	Analysis of semivolatile organic compounds by: SW-846 8270 – Modified for Selective Ion Monitoring (SIM) 08/09, Revision 7.	Definitive	Water PAHs	Gas Chromatography/ Mass Spectroscopy (GC/MS)	Katahdin Analytical Services, Inc.	N
CA-204	Analysis of semivolatile organic compounds by Capillary Column GC/MS: SW-846 Method 8270, 08/09, Revision 11.	Definitive	Soil PAHs	GC/MS	Katahdin Analytical Services, Inc.	N
CA-502	Preparation Of Aqueous Samples For Extractable Semivolatile Analysis, 10/09, Revision 6.	Definitive	Water PAH Extraction	Separatory Funnel Extractor	Katahdin Analytical Services, Inc.	N
CA-512	Preparation Of Sediment/Soil Samples By Sonication Using Method 3550 For Subsequent Extractable Semi-Volatiles Analysis, 02/09, Revision 7.	Definitive	Soil PAH Extraction	Ultrasonic Extractor	Katahdin Analytical Services, Inc.	N
CA-526	Preparation Of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For Subsequent Extractable Semivolatile Analysis, 08/09, Revision 6.	Definitive	Soil PAH Extraction	Soxhlet Extractor	Katahdin Analytical Services, Inc.	N
CA-719	Total Dissolved Solids (Filterable Residue) by EPA Method 160.1 and Standard Methods 2540 C	Definitive	Water TDS	Drying Oven	Katahdin Analytical Services, Inc.	N
Katahdin SD-902	Sample Receipt and Internal Control, 08/09, Revision 8.	Definitive	Various	NA	Katahdin Analytical Services, Inc.	N
Katahdin SD-903	Sample Disposal, 05/09, Revision 4.	Definitive	Various	NA	Katahdin Analytical Services, Inc.	N

SAP Worksheet No. 24 -- Analytical Instrument Calibration Table

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
GC/MS PAHs (full scan)	Initial Calibration - A minimum 5-point calibration is required.	Instrument receipt, instrument change (new column, source cleaning, etc.), when continuing calibration verification (CCV) is out of criteria. Six-point initial calibration for all analytes.	System Performance Check Compound (SPCC) average response factors (RFs) must be ≥ 0.050 ; Percent relative standard deviation (%RSD) 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 (6 points for second order).	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/Supervisor	CA-204
	Initial Calibration Verification (ICV)	Once after each initial calibration.	Percent recoveries (%Rs) of individual compounds must be within 75 -125%.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst/Supervisor	
	CCV	Analyze a standard at the beginning of each 12-hour shift after a decafluorotriphenylphosphine (DFTPP) tune.	SPCC RFs must be ≥ 0.050 . %Ds for all target compounds and surrogates must be $\leq 20\%$.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/Supervisor	
	Tune verification - DFTPP	Every 12 hours	Criteria listed in section 7.4 current revision of SOP CA-213.	Retune and/or clean source.	Analyst, Supervisor	
GC/MS PAHs (SIM)	Initial Calibration - A minimum 5-point calibration is required.	Instrument receipt, instrument change (new column, source cleaning, etc.), when CCV is out of criteria. Six-point initial calibration for all analytes.	SPCC average RFs must be ≥ 0.010 ; %RSD must be ≤ 30 for the CCCs; %RSD must be $< 15\%$ for all other compounds. If not met: Option 1) Linear least squares regression: r must be ≥ 0.995	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/Supervisor	CA-213

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
			Option 2) Non-linear regression: COD r^2 must be ≥ 0.99 (6 points for second order).			
	ICV	Once after each initial calibration.	%Rs of individual compounds must be within 75 -125%.	Identify source of problem, correct, repeat calibration, rerun samples.	Analyst/Supervisor	
	CCV	Analyze a standard at the beginning of each 12-hour shift after a DFTPP tune.	SPCC RFs must be ≥ 0.010 . %Ds for all target compounds and surrogates must be $\leq 30\%$.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/Supervisor	
	Tune verification-DFTPP	Every 12 hours	Criteria listed in section 7.4 current revision of SOP CA-213.	Retune and/or clean source.	Analyst, Supervisor	
Total Dissolved Solids	Balance Verification	Every day before use.	Within criteria specified in SOP CA-102.	Investigate problem. Do not use balance until verification has passed.	Analyst, Supervisor	CA-719

SOP = Standard Operating Procedure
 SIM = Selective Ion Monitoring
 PAH = Polycyclic Aromatic Hydrocarbon
 TDS = Total Dissolved Solids
 GC/MS = Gas Chromatography/Mass Spectrometer
 NA = Not applicable

SAP Worksheet No. 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	SOP Reference
GC/MS scan and SIM	Check pressure and gas supply daily. Manual tune if DFTPP not in criteria, change septa as needed, change liner as needed, cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP	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/ Supervisor	Katahdin SOP CA-204 and CA- 213

SOP = Standard Operating Procedure
 SIM = Selective Ion Monitoring
 PAH = Polycyclic Aromatic Hydrocarbon
 GC/MS = Gas Chromatography/Mass Spectrometer
 DFTPP = Decafluorotriphenylphosphine
 NA = Not applicable

SAP Worksheet No. 26 -- Sample Handling System

[\(UFP-QAPP Manual Appendix A\)](#)

SAMPLE HANDLING SYSTEM

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): FOL or designee/TtNUS
Sample Packaging (Personnel/Organization): FOL or designee / TtNUS
Coordination of Shipment (Personnel/Organization): FOL or designee / TtNUS
Type of Shipment/Carrier: Federal Express
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Custodians/Katahdin Analytical Services
Sample Custody and Storage (Personnel/Organization): Sample Custodians/ Katahdin Analytical Services
Sample Preparation (Personnel/Organization): Extraction Lab, Katahdin Analytical Services
Sample Determinative Analysis (Personnel/Organization): Gas Chromatography/Mass Spectrometry Lab, Katahdin Analytical Services
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 60 days from receipt of samples.
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 Analytical Services

SAP Worksheet No. 27 -- Sample Custody Requirements Table

[\(UFP-QAPP Manual Section 3.3.3\)](#)

SAMPLE CUSTODY REQUIREMENTS

Field Chain of Custody

To ensure the integrity of a sample from collection through analysis, an accurate, written record that traces the possession and handling of the sample is necessary. This documentation is referred to as the chain-of-custody form. Chain-of-custody begins at the time of sample collection. A sample is under custody if any of the following conditions apply:

- It is in the owner's actual possession
- It is in the owner's view, after being in his/her physical possession,
- It was in the owner's possession and was locked or sealed up to prevent tampering,
- It is in a secure area.

Custody documentation is designed to provide documentation of preparation, handling, storage, and shipping of all samples collected. A multi-part chain-of-custody form is used with each page of the form signed and dated by the recipient of a sample or portion of sample. The person releasing the sample and the person receiving the sample each will retain a copy of the chain-of-custody form each time a sample transfer occurs.

Preservation of the integrity of the samples collected during the RI will be the responsibility of identified persons from the time the samples are collected until the samples, or their derived data, are incorporated into the final report. Sample custody is described in Worksheet No. 27.

The FOL is responsible for the care and custody of the samples collected until they are delivered to the laboratory or are entrusted to a carrier. When transferring samples, the individuals relinquishing and receiving them will sign, date, and note the time on the chain-of-custody form. This form documents the sample custody transfer from the sampler to the laboratory, often through another person or agency (common carrier). Field chain-of-custody requirements are provided in SOP-03. Upon arrival at the laboratory, internal sample custody procedures will be followed as defined in the laboratory SOPs included in Appendix C.

Laboratory Chain of Custody – Katahdin Analytical Services

The laboratory sample custody procedures (receipt of samples, archiving, and disposal) documented in Katahdin Analytical Services SOPs will be followed. Coolers will be received and checked for proper temperature. A sample cooler receipt form will be filled out to note conditions and any discrepancies. The chain-of-custody will be checked against the sample containers for correctness. Samples will be logged into the laboratory information management system and given a unique log number which can be tracked through processing. The client will be notified of any problems.

SAP Worksheet No. 28 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	No analytes > ½ LOQ.	Investigate source of contamination. Evaluate the samples and associated QC: if the blank results are above the LOQ, then report sample results which are < the LOQ or > 10X the blank concentration. Re-prepare and analyze the method blank and all samples processed with the contaminated blank.	Analyst, Department Manager, QA Officer	Accuracy / Bias / Contamination	See Method/SOP QC Acceptance Limits.
Laboratory Control Sample (LCS)	One per preparation batch of 20 or fewer samples of similar matrix.	Katahdin statistically derived acceptance limits. Limits are provided in Appendix C.	Reextract and reanalyze all associated samples for affected analyte.	Analyst, Department Manager, QA Officer	Accuracy / Bias	See Method/SOP QC Acceptance Limits.
MS/MSD	One per preparation batch of 20 or fewer samples of similar matrix.	Katahdin statistically derived acceptance limits. Limits are provided in Appendix C.	CA 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, then re-prepare and analyze the samples and QC.	Analyst, Department Manager, QA Officer	Accuracy / Bias	See Method/SOP QC Acceptance Limits.

Matrix	Soil Samples					
Analytical Group	PAHs					
Analytical Method/ SOP Reference	SW846 8270C / Katahdin SOP CA-204					
QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal standard	Six per every sample, standard, and QC sample: 1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	Retention time \pm 30 seconds from retention time of the midpoint standard in the initial calibration: extracted ion current profile area within - 50 to + 100% of initial midpoint standard.	Inspect MS or GC for malfunctions: mandatory reanalysis of samples analyzed while system was malfunctioning. If reanalysis confirms matrix interference, then report sample and narrate.	Analyst, Department Manager, QA Officer	Accuracy / Bias	See Method/SOP QC Acceptance Limits.
Surrogates	Six added to all field and QC samples.	Katahdin statistically derived acceptance limits. %R: <u>Soil</u> 2-Fluorophenol 43-99 Phenol-d6 53-98 Nitrobenzene-d5 47-100 2-Fluorobiphenyl 49-114 2,4,6-Tribromophenol 44-111 Terphenyl-d14 58-140	(1) Check chromatogram for interference; if found, then flag data. (2) If not found, then 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 not compliant, then flag data.	Analyst, Department Manager, QA Officer	Accuracy / Bias	See Method/SOP QC Acceptance Limits.

Matrix	Aqueous Samples					
Analytical Group	SIM PAHs					
Analytical Method/ SOP Reference	SW846 8270C / 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
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	No analytes > ½ LOQ.	Investigate source of contamination. Evaluate the samples and associated QC: if the blank results are above the LOQ, then report sample results which are < the LOQ or > 10X the blank concentration. Re-prepare and analyze the method blank and all samples processed with the contaminated blank.	Analyst, Department Manager, QA Officer	Accuracy / Bias / Contamination	See Method/SOP QC Acceptance Limits.
LCS	One per preparation batch of 20 or fewer samples of similar matrix.	Katahdin statistically derived acceptance limits. Limits are provided in Appendix C.	Reextract and reanalyze all associated samples for affected analyte.	Analyst, Department Manager, QA Officer	Accuracy / Bias	See Method/SOP QC Acceptance Limits.
MS/MSD	One per preparation batch of 20 or fewer samples of similar matrix.	Katahdin statistically derived acceptance limits. Limits are provided in Appendix C.	CA 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 and analyze the samples and QC.	Analyst, Department Manager, QA Officer	Accuracy / Bias	See Method/SOP QC Acceptance Limits.
Internal standard	Every sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the initial calibration: extracted ion current profile area within -50 to + 100% of initial midpoint standard.	Inspect MS or GC for malfunctions: mandatory reanalysis of samples analyzed while system was malfunctioning. If reanalysis confirms matrix interference, report sample and narrate.	Analyst, Department Manager, QA Officer	Accuracy / Bias	See Method/SOP QC Acceptance Limits.
Surrogates	Three added to all field and QC samples.	Katahdin statistically derived acceptance limits.	(1) Check chromatogram for interference; if found, flag data.	Analyst, Department Manager, QA Officer	Accuracy / Bias	See Method/SOP QC Acceptance Limits.

Matrix	Aqueous Samples					
Analytical Group	SIM PAHs					
Analytical Method/ SOP Reference	SW846 8270C / 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
		%R: Methylnaphtahlene-d10 34-110 Fluorene-d10 46-122 Pyrene-d10 36-134	(2) If not found, check instrument performance; if problem is found, correct and reanalyze. (3) If still out, re-extract and analyze sample. (4) If reanalysis is out, flag data.			

Matrix	Water					
Analytical Group	Total Dissolved Solids					
Analytical Method/ SOP Reference	SM2540C/CA-719					
QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples).	Must be less than the LOQ.	Investigate source of contamination. Evaluate the samples and associated QC: i.e. If the blank results are above the LOQ, then report sample results which are <LOQ or > 10X the blank concentration. Otherwise, re-prepare a blank and the associated samples.	Analyst, Supervisor, QA Manager	Accuracy/Bias, Contamination	See Method/SOP QC Acceptance Limits.
Laboratory Duplicate	One sample duplicate per ten samples.	RPD must be <20.	Investigate problem and reanalyze sample in duplicate. If RPD is still >20, then report original result with notation.	Analyst, Supervisor, QA Manager	Precision	See Method/SOP QC Acceptance Limits.
LCS	One per preparation	%R must be 90%-110%.	(1) Investigate source of	Analyst, Supervisor, QA	Accuracy/Bias	See Method/SOP QC

Matrix	Water					
Analytical Group	Total Dissolved Solids					
Analytical Method/ SOP Reference	SM2540C/CA-719					
QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
	batch of 20 or fewer samples).		problem. If the LCS recovery is high but the sample results are <LOQ, then narrate. Otherwise, re-prepare the blank and the remaining samples.	Manager		Acceptance Limits.

SAP Worksheet No. 29 -- Project Documents and Records Table

(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
<u>Sample Collection Documents and Records</u> Project Personnel Sign-Off Record Field logbook (and sampling notes) Field sample forms (e.g. boring logs, sample log sheets, drilling logs, etc.) Chain-of-custody records Sample shipment airbills Equipment calibration logs Photographs Field Task Modification Forms Sampling and Analysis Plan Field Sampling SOPs	TtNUS project file (may include hard copy as well as electronic information), results will be discussed in subject document.
<u>Laboratory Documents and Records</u> Sample receipt/log-in forms Sample storage records Sample preparation logs Standard traceability logs Equipment calibration logs Sample analysis run logs Equipment maintenance, testing, and inspection logs Reported field sample results Reported results for standards, QC checks, and QC samples Data completeness checklists Sample storage and disposal records Telephone logs Extraction/clean-up records Raw data <u>Data Assessment Documents and Records</u> Field sampling audit checklist (if an audit is conducted) Analytical audit checklist (if an audit is conducted) Data validation memoranda	TtNUS project file (may include hard-copy as well as electronic information), long-term data package storage at third-party professional document storage firm; results will be discussed in subject document. Electronic data results will be maintained in a database on a password protected Structured Query Language (SQL) server.
<u>Other Documents</u> HASP All versions of SAP All versions of reports (e.g., SI, RI, FS, etc.)	All versions of the subject document and all support documents will be stored in hard-copy in the TtNUS project file and electronically in the server library.

SAP Worksheet No. 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)
Aqueous	PAHs	See Worksheet No. 18	SW-846 8270C SIM	21 calendar days	Katahdin Analytical Services, Inc. 600 Technology Way Scarborough, Maine 04074 Ms. Kate Zaleski 207.874.2400 kzaleski@katahdinlab.com	NA
Soil	PAHs	See Worksheet No. 18	SW-846 8270C (scan)			

NA = Not applicable

PAH = Polycyclic Aromatic Hydrocarbon

SIM = Selective Ion Monitoring

SAP Worksheet No. 31 -- Planned Project Assessments 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 Action (CA) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Laboratory Systems Audit	Every 3 years	External	DoD ELAP	DoD ELAP	Laboratory QAM or Laboratory Manager Katahdin Analytical Services	Laboratory QAM or Laboratory Manager Katahdin Analytical Services	DoD ELAP and TtNUS
Field Sampling Systems Audit	1 per contract year	Internal	TtNUS	TBD	TtNUS TOM and FOL	Auditor and QAM TtNUS	QAM TtNUS

¹ Katahdin has successfully completed the laboratory evaluation process required as part of the DoD QSM. The DoD ELAP accreditation letter is included in Appendix C.

SAP Worksheet No. 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)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Field sampling system audit ⁽¹⁾	Audit checklist [as per Navy Installation Restoration Chemical Data Quality Manual (IRCDQM)] and written audit report	Ken Grim, TtNUS TOM; Larry Basilio, TtNUS FOL Debbie Humbert, Program Manager, TtNUS;	Dependant on findings; if major, a stop work maybe issued immediately; if minor, within 1 week of audit	Written memorandum	Kelly Carper, QAM, TtNUS TBD, Auditor, TtNUS Debbie Humbert, Program Manager, TtNUS;	Within 48 hours of notification
Laboratory systems audit	Written audit report	Laboratory Manager or Laboratory QAM, Katahdin Analytical Services	Not specified by DoD ELAP	Letter	DoD ELAP	Specified by DoD ELAP

Notes:

¹ Audits are scheduled at the TtNUS program level and may or may not include this project.

SAP Worksheet No. 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	Completion of data validation	DVM or designee TtNUS	TOM TtNUS and TtNUS project file
Major analysis problem identification (Internal memo)	When persistent analysis problems are detected	On the same day	QAM TtNUS	TOM TtNUS, QAM TtNUS, Program Manager TtNUS and project file
Project monthly progress report	Monthly for duration of project	Monthly	TOM TtNUS	RPM Navy, project file
Field progress reports	Daily, oral, during the course of sampling	Every day field sampling occurs	FOL TtNUS	TOM TtNUS
Laboratory QA report	When significant plan deviations result from unanticipated circumstances	On the same day	PM Katahdin Analytical Services	QAM or Project Chemist TtNUS, project file

DVM – Data Validation Manager
 FOL – Field Operations Leader
 QAM – Quality Assurance Manager
 TOM – Task Order Manager
 SDG – Sample Delivery Group

SAP Worksheet No. 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	<p>A TtNUS representative will review and sign the chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory and that the sample information is accurate. The forms will be signed by the sampler and a copy will be retained for the project file, the Project Manager, and the data validators.</p> <p>The laboratory sample custodian will review the sample shipment for completeness and integrity and will sign accepting the shipment. The data validators will check that the chain-of-custody form was signed/dated by the TtNUS FOL or designee relinquishing the samples and also by the laboratory sample custodian receiving the samples for analyses.</p>	<p>Internal</p> <p>Internal/ External</p>	<p>TtNUS, field personnel</p> <p>1 - Laboratory sample custodian, Katahdin 2 - Project Chemist or Data Validators, TtNUS</p>
Sample tables	Verify that all proposed samples listed in the SAP tables have been collected.	Internal	FOL, field personnel, TtNUS
Sample log sheets	Verify that information recorded in the log sheets is accurate and complete.	Internal	FOL, field personnel, TtNUS
Sample coordinates	Verify that sample locations are correct and in accordance with the SAP proposed locations. Take into account the potential for locations to have been updated.	Internal	FOL, field personnel, TtNUS
Field QC samples	Check that field QC samples listed in Worksheet No. 20 were collected as required.	Internal	FOL, field personnel, TtNUS
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	Laboratory QAM, Katahdin

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
	The data package will be verified for completeness by TtNUS data validators. Missing information will be requested from the laboratory, and validation will be suspended until missing data are received. This occurs as part of the data validation process	External	Project Chemist or Data Validators, TtNUS
Analytical data package and Electronic data deliverables	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	Data Validators, TtNUS

Fixed base laboratory data will be subject to full data validation. Verification includes field data verification and laboratory data verification. Verification inputs as per this worksheet will be checked.

SAP Worksheet No. 35 -- Validation (Steps IIa and IIb) Process Table

(UFP-QAPP Manual Section 5.2.2) (Figure 37, page 110 UFP-QAPP Manual) (Table 9 UFP-QAPP Manual)

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Field SOPs/Field Logs/Sample Collection Logs	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and Measurement Performance Criteria (MPC) have been achieved. Particular attention will 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.	Designee, TtNUS
IIa	Analytical SOPs	Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied.	Data Validators, TtNUS
IIa	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 TtNUS for guidance prior to report preparation.	Data Validators, TtNUS
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.	Project Chemist or Data Validators, TtNUS
IIa	Holding times	Review that the samples were shipped and stored at the required temperature and sample pH values for chemically preserved samples meet the requirements listed in Worksheet No. 19. Ensure that the analyses were performed within the holding times listed in Worksheet No. 19.	Project Chemist or Data Validators, TtNUS
IIa/IIb	Laboratory Data Results for Accuracy	Ensure that the laboratory QC samples listed in Worksheet No. 28 were analyzed and that the MPC listed in Worksheet No. 12 were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical quality control criteria set up for this project were met.	Project Chemist or Data Validators, TtNUS

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa/IIb	Field and Laboratory Duplicate Analyses for Precision	Check field sampling precision by calculating the RPD for field duplicate and triplicate samples. Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses, matrix spike/matrix spike duplicates, and LCS. Ensure compliance with the methods and project MPC accuracy goals listed in Worksheet No. 12.	Project Chemist or Data Validators, TtNUS
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.	Project Chemist or Data Validators, TtNUS
IIa/IIb	PALs	Discuss the impact on matrix interferences or sample dilutions performed because of the high concentration of one or more contaminant on the other target compounds reported as not-detected. Document this usability issue and inform the TOM.	Project Chemist or Data Validators, TtNUS
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 reason for data qualifications. Determine if the data met the MPC and determine the impact of any deviations on the technical usability of the data.	Project Chemist or Data Validators, TtNUS
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 TtNUS TOM.	Designee, TtNUS

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa, IIb	Documentation of Analytical Reports for Completeness	Ensure that the chain-of-custody form generated in the field to delivery of analytical data 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 validations are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet 36. Check that all data have been transferred correctly and completely to the final Structured Query Language (SQL) database.	Project Chemist or Data Validators, TtNUS
IIa/IIb	PALs	Review and add PALs to the laboratory electronic data deliverable (EDD). Flag samples and notify TtNUS TOM of samples that exceed PALs as listed on Worksheet 15.	Designee, TtNUS
IIb	Project QLs for sensitivity	Ensure that the project QLs listed in Worksheet No. 15 were achieved.	Project Chemist or Data Validators, TtNUS
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.	Project Chemist or Data Validators, TtNUS

Note: Fixed base laboratory analytical data will be subject to full data validation.

SAP Worksheet No. 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	Aqueous and Soil	PAHs	Method-specific criteria for SW-846 8270C (SIM and scan) listed in Worksheets # 12, # 15, # 24, and # 28, TRRP 13, and DOD QSM will be used. If not included in Worksheet #12, #15, #24 or #28, the logic outlined in USEPA CLP National Functional Guidelines for Organic Data Review USEPA-540/R-99-008, October 1999 will be used to apply qualifiers to data.	Project Chemist or Data Validator, TtNUS

SAP Worksheet No. 37 -- Usability Assessment

[\(UFP-QAPP Manual Section 5.2.3\)](#)

Data Usability Assessment

The usability of the data directly affects whether project objectives can be achieved. The following characteristics will be evaluated at a minimum. The results of these evaluations will be included in the project report. The characteristics will be evaluated for multiple concentration levels if the evaluator determines that this is necessary. To the extent required by the type of data being reviewed, the assessors will consult with other technically competent individuals to render sound technical assessments of these data characteristics:

Completeness

- For each matrix that was scheduled to be sampled, the FOL acting on behalf of the project team will prepare a table listing planned samples/analyses to collected samples/analyses. If deviations from the scheduled sample collection or analyses are identified, the TtNUS TOM and risk assessor will determine whether the deviations compromise the ability to meet project objectives. If they do, the TtNUS TOM will consult with the Navy RPM and other project team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions.

Precision

- The Project Chemist acting on behalf of the project team will determine whether precision goals for field duplicates and laboratory duplicates were met. This will be accomplished by comparing duplicate results to precision goals identified in Worksheets Nos. 12 and 28. This will also include a comparison of field and laboratory precision with the expectation that field duplicate results will be no less precise than laboratory duplicate results. If the goals are not met, or data have been flagged as estimated (J qualifier), limitations on the use of the data will be described in the project report.

Accuracy

- The Project Chemist acting on behalf of the project team will determine whether the accuracy/bias goals were met for project data. This will be accomplished by comparing percent recoveries of LCS, MS, MSD, and surrogate compounds to accuracy goals identified in Worksheet No. 28. This assessment will include an evaluation of field and laboratory contamination; instrument calibration variability; and analyte recoveries for surrogates, matrix spike, and laboratory control samples. If the goals are not met, limitations on the use of the data

will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report.

Representativeness

- A project scientist identified by the TtNUS TOM and acting on behalf of the project team will determine whether the data are adequately representative of intended populations, both spatially and temporally. This will be accomplished by verifying that samples were collected and processed for analysis in accordance with the SAP, by reviewing spatial and temporal data variations, and by comparing these characteristics to expectations. The usability report will describe the representativeness of the data for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the project scientist indicates that a quantitative analysis is required.

Comparability

- The Project Chemist acting on behalf of the project team will determine whether the data generated under this project are sufficiently comparable to historical site data generated by different methods and for samples collected using different procedures and under different site conditions. This will be accomplished by comparing overall precision and bias among data sets for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the Project Chemist indicates that such quantitative analysis is required.

Sensitivity

- The Project Chemist acting on behalf of the project team will determine whether project sensitivity goals listed in Worksheet No. 15 are achieved. The overall sensitivity and LOQs from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be described. The Project Chemist will enlist the help of the project risk assessor to evaluate deviations from planned sensitivity goals.

Project Assumptions and Data Outliers

- The TtNUS TOM and designated team members will evaluate whether project assumptions were valid. This will typically be a qualitative evaluation but may be supported by quantitative evaluations. The type of evaluation depends on the assumption being tested. Quantitative assumptions include assumptions related to data distributions (e.g., Normal versus log-normal) and estimates of data variability. Potential outliers will be removed if a review of the associated documentation indicates that the results have an assignable cause that renders them inconsistent

with the rest of the data. During this evaluation, the team will consider whether outliers could be indications of unanticipated site conditions.

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

After completion of the data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. In addition to the evaluations described above, a series of inspections and statistical analyses will be performed to estimate these characteristics. The statistical evaluations will include simple summary statistics for target analytes, such as maximum concentration, minimum concentration, number of samples exhibiting non-detected results, number of samples exhibiting positive results, and the proportion of samples with detected and non-detected results. The project team members identified by the TOM will assess whether the data collectively support the attainment of project objectives. They will consider whether any missing or rejected data have compromised the ability to make decisions or to make the decisions with the desired level of confidence. The data will be evaluated to determine whether missing or rejected data can be compensated by other data. Although rejected data will generally not be used, there may be reason to use them in a weight of evidence argument, especially when they supplement data that have not been rejected. If rejected data are used, their use will be supported by technically defensible rationales.

For statistical comparisons and mathematical manipulations, non-detected values will be represented by a concentration equal to one-half the sample-specific reporting limit. Duplicate results (original and duplicate) will not be averaged for the purpose of representing the range of concentrations. However, the maximum concentration of the original and duplicate samples will be used to represent the concentration at a particular sampled location.

Identify the personnel responsible for performing the usability assessment:

The TtNUS TOM, Project Chemist, and FOL will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed with the Navy RPM, the EPA Project Manager, and the TCEQ Project Manager. The review will take place either in a face to face meeting or a teleconference depending on the extent of identified deficiencies. If no significant deficiencies are identified, the data usability assessment will simply be documented in the project report and reviewed during the normal document review cycle.

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 data will be presented in tabular format, including data qualifications such as estimation (J, UJ) or rejection (R). Written documentation will support the non-compliance estimated or rejected data results. The project report will identify and describe the data usability limitations and suggest re-sampling or other corrective actions, if necessary.

APPENDIX A

ECOLOGICAL RISK SCREENING METHODOLOGY

ECOLOGICAL RISK SCREENING METHODOLOGY

1.0 INTRODUCTION

The goal of the Ecological Risk Assessment (ERA) will be to determine whether adverse ecological impacts are present as a result of exposure to chemicals released to the environment through historical activities at the NALF Cabaniss former Skeet Range. The ERA will contain information that enables risk managers to conclude that either ecological risks at the site are negligible or that further information is necessary to evaluate potential ecological risks at the site.

The ERA methodology is in accordance with guidance presented in the following documents:

- Final Guidelines for Ecological Risk Assessment (USEPA, 1998).
- Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Ecological Risk Assessments (USEPA, 1997).
- Guidance for Conducting Ecological Risk Assessments at Remediation Sites in Texas (TNRCC, 2001)
- Update to Guidance for Conducting Ecological Risk Assessments at Remediation Sites in Texas RG-263 (Revised) (TCEQ, 2006)

This ERA will consist of Steps 1, 2, and 3a of the eight step USEPA ERA process, and Tier 1 and 2 of the TCEQ ERA guidance. The first two screening steps of the USEPA guidance, and Elements 1 through 6 of the TCEQ guidance comprise the screening-level ecological risk assessment (SERA), where conservative exposure estimates are compared to screening-level and threshold toxicity values. Step 3a of the USEPA guidance is the first step of a baseline ecological risk assessment (BERA) and consists of refining the conservative assumptions to further focus the ERA on the chemicals and receptors of greatest concern at a site. This step is similar to Element 7 in the TCEQ guidance, which consists of a less conservative analysis. The remaining steps of the ERA process would require revisions to the Sampling and Analysis Plan prior to initiation and are not included in this ERA methodology. These remaining steps generally occur after Steps 1, 2, and 3a are completed and are applicable only if proceeding further in the BERA process is necessary to better evaluate ecological risks.

2.0 PROBLEM FORMULATION

Problem formulation is the first phase of an ERA and discusses the goals and focus of the assessment. It includes general descriptions of the site with emphasis on the habitats and ecological receptors present. This phase also involves characterization of site-related chemicals, chemical sources, migration routes, and an evaluation of routes of chemical exposure. The assessment and measures of effects to be evaluated are also selected. Finally, a conceptual site model (CSM) is developed that describes how chemicals associated with the site in question may come into contact with ecological receptors.

2.1 Environmental Setting

The objectives of this step are to 1) initially identify and characterize the habitats and ecological resources throughout the site, and, 2) describe the likely chemical sources, release mechanisms, migration pathways, and the fate of chemicals resulting from site-related activities, as well as ecological receptors that could be adversely affected by chemicals.

The former Skeet Range is located in the southeastern corner of NALF Cabaniss, just north of Oso Creek. The former Skeet Range is covered with vegetation. Target analytes are polycyclic aromatic hydrocarbons (PAHs). Section 10.2 (Worksheet 10) of this SAP presents a more detailed site description and list of potential contaminants.

2.2 Potential Exposure Pathways

Terrestrial receptors at the site can be exposed to chemicals in soil. Surface soil for the purpose of this ERA will be soil from the ground surface to a depth of 1 feet. Several groups of terrestrial ecological receptors can be exposed to chemicals in surface soil. Invertebrates such as earthworms are exposed to chemicals while moving through soil, and invertebrates ingest soil particles while searching for food. Plants are exposed to chemicals via direct contact as chemicals are absorbed through the roots and may then translocate to different parts of the plants (i.e., leaves, seeds).

Small mammals may be exposed to chemicals in soil via several exposure routes. They may be exposed by direct contact as they search for food or burrow into the soil. Exposure of terrestrial wildlife to chemicals in the soil via dermal contact is unlikely to represent a major exposure pathway because fur, feathers, and chitinous exoskeletons are expected to minimize transfer of chemicals across dermal tissue. Small mammals can be exposed to chemicals in the soil via incidental ingestion of soil, and through ingestion of plants and/or invertebrates that have accumulated chemicals from the soil.

Terrestrial vertebrates may be exposed to chemicals found in the air via inhalation. Although this pathway is possible, it is not a significant pathway and will not be evaluated in this ERA.

Larger predatory species, such as the red fox and red-tailed hawk, can be exposed (indirectly) to chemicals in soil by ingesting prey items such as small mammals that have accumulated chemicals from the soil and food items.

2.3 Endpoints

2.3.1 Assessment Endpoints

An assessment endpoint is an explicit expression of the environmental value that is to be protected (USEPA, 1997). The selection of these endpoints is based on the habitats present, the migration pathways of chemicals, and the routes that chemicals may take to enter receptors.

For this ERA, the assessment endpoints will include the protection of the following groups of receptors from a reduction in growth, survival, and/or reproduction caused by site-related chemicals:

- Soil invertebrates
- Terrestrial vegetation
- Terrestrial invertivorous birds and mammals
- Terrestrial herbivorous birds and mammals

The following paragraphs discuss why the above assessment endpoints were selected for this ERA.

Soil Invertebrates: Soil invertebrates present at the site aid in the formation of soil, as well as in the redistribution and decomposition of organic matter in the soil, and serve as a food source for higher trophic-level organisms. They can also accumulate some contaminants, which can then be transferred to the higher trophic-level organisms that consume invertebrates.

Terrestrial Vegetation: Terrestrial vegetation at the site consists of grasses, shrubs, and trees. These plant types serve as a food source, provide shade and cover for many organisms, and help prevent soil erosion, among other important functions. They can also accumulate some contaminants, which can then be transferred to the higher trophic-level organisms that consume plants.

Terrestrial Herbivorous Birds and Mammals: Herbivorous birds and mammals (i.e., animals that consume only plant tissue) are present at the site. Their role in the community is essential because without them, higher trophic levels could not exist (Smith, 1966). They may be exposed to and accumulate contaminants that are present in the plants they consume, and soil they incidentally ingest.

Terrestrial Invertivorous Birds and Mammals: Birds and mammals that consume primarily invertebrates are considered first-level carnivores. They serve as a food source for higher trophic level carnivores and may be exposed to and accumulate chemicals present in the food items they consume, and soil they incidentally ingest.

As indicated in USEPA (1997), "it is not practical or possible to directly evaluate risks to all of the individual components of the ecosystem at a site. Instead, assessment endpoints focus the risk assessment on particular components of the ecosystem that could be adversely affected by contaminants from the site." Therefore, the ERA will focus on the endpoints that tend to yield the highest risks, which will account for endpoints that have lower risks.

Carnivorous birds and mammals generally have large home ranges. The site covers approximately 12 acres of land. When the size of the site is compared to the home range of top carnivores, such as the red-tailed hawk (with an average of 1,692 acres) and the red fox (with an average of 1,793 acres), carnivores would receive only a very small portion of their diet from site and, therefore, will not be included as receptors in the ERA. Threshold oral toxicity values for reptiles are not available for most chemicals, so risks to reptiles were not quantitatively evaluated. With the above factors in mind, reptiles, and carnivores were not selected as assessment endpoints.

2.3.2 Measures of Effects

Measures of effects are estimates of biological impacts (i.e., survival, growth and/or reproduction) that are used to evaluate the assessment endpoints. The following measures of effects will be used to evaluate the assessment endpoints in this ERA.

- Decreases in survival, growth, and/or reproduction of plants and terrestrial invertebrates will be evaluated by comparing measured concentrations of chemicals in surface soil to screening values designed to be protective of ecological receptors.
- Decreases in survival, reproduction, and/or developmental effects of birds and mammals will be evaluated by comparing the estimated ingested dose of contaminants in surface soil to no-

observed-adverse-effects levels (NOAELs) and lowest-observed-adverse-effects levels (LOAELs) for surrogate wildlife species.

Many receptors in the soil environments at the site are adequately described in general categories such as soil invertebrates. This is due to the nature of the threshold values, effects values, or criteria typically used to characterize risk for such organisms. For vertebrate receptors, selection of a particular surrogate species is required so that intake through eating and drinking can be estimated. The availability of exposure parameters such as body mass, feeding rate, and drinking rate, and the potential for the species or a similar species to be present at the site are primary factors in selecting surrogate species. The following surrogate receptor species will be used for the food-chain modeling conducted as part of the ERA:

- White-footed mouse: terrestrial herbivorous mammal
- Mourning dove: terrestrial herbivorous bird
- Short-tailed shrew: terrestrial invertivorous mammal
- American robin: terrestrial invertivorous bird

2.4 Conceptual Site Model

A CSM in ERA problem formulation is a written description of predicted relationships between ecological entities and the stressors to which they may be exposed (USEPA, 1998). The CSM consists of two primary components: predicted relationships among stressor, exposure, and assessment endpoint response, and a diagram that illustrates the relationships (USEPA, 1998). Worksheet 10 in the SAP presents the CSM for the site, and Figure 10-4 in the SAP is a graphic illustration of the CSM.

In summary, contamination was released to the soil via several activities. Plants, soil invertebrates, and vertebrates are exposed to chemicals in the surface soil by direct contact and/or ingestion of soil and food items.

3.0 ECOLOGICAL EFFECTS EVALUATION

The ecological effects assessment is an investigation of the relationship between the exposure to a chemical and the potential for adverse effects resulting from exposure. In this step, screening levels for toxicity of the chemicals to ecological receptors are compiled.

3.1 Terrestrial plants and invertebrates

Potential risks to terrestrial plants and invertebrates resulting from exposure to chemicals in surface soil will be evaluated by comparing chemical concentrations to ecological screening levels. These toxicity values are expressed in units of concentration because terrestrial plants and invertebrates are in direct contact with the soil. The screening levels consist of the USEPA Ecological Soil Screening Levels (Eco SSLs) (USEPA, June 2007) and TCEQ (2006) screening levels. Table 1 presents the screening levels, along with the sources of each screening level.

3.2 Mammals and birds

Risk to wildlife from exposure to chemicals in surface soil will be determined by estimating the Chronic Daily Intake (CDI) using food chain models and comparing the CDI to toxicity reference values (TRVs) representing acceptable daily doses in mg/kg-day. The TRVs will be developed from NOAELs and LOAELs obtained from wildlife studies.

The NOAELs and LOAELs were obtained from the USEPA Eco SSL document for PAHs (USEPA, June 2007). This Eco SSL document provides both NOAELs and LOAELs for various studies, and overall NOAELs for specific chemicals, but the Eco SSL documents do not provide overall LOAELs. Therefore, the geometric mean of the chemical-specific growth and reproduction LOAELs from the chemical-specific Eco SSL documents was used as the LOAEL.

Table 2 presents the NOAELs and LOAELs that will be used to develop the TRVs for the test species used in the study. The available literature-based toxicological data are based on animals other than the selected indicator species, so in accordance with TNRCC (2001), the allometric scaling model based on Sample and Arenal (1999) will be used to derive NOAELs and LOAELs for the wildlife species evaluated in the ERA from the NOAELs and LOAELs for the test species. The following equation will be used to derive these values:

$$\text{NOAEL}_w = \text{NOAEL}_t (\text{BW}_t / \text{BW}_w)^{(1-b)}$$

where:

NOAEL_w = Toxicity value (mg/kg body weight-day) for selected avian or mammalian wildlife species.

NOAEL_t = Toxicity value for avian or mammalian species “t,” test species to extrapolate from (e.g., rat) mg/kg body weight-day

BW_t = Body weight of avian or mammalian test species (kg)

BW_w = Body weight of avian or mammalian wildlife species (kg)

b = Allometric scaling factor that is specific to either birds or mammals (unitless)

When a COPC-specific allometric scaling factor is available from Sample and Arenal (1999), it will be used to extrapolate toxicity endpoints from known test species' endpoints to the receptor species. In the absence of COPC-specific allometric scaling factors, default allometric scaling factors of 1.2 for birds and 0.94 for mammals will be used, as recommended by Sample and Arenal (1999) and the TCEQ (TNRCC, 2001). Table 3 presents the body weights of the receptor species that will be used in the food chain model. Table 2 presents the body weights for all the test species, when available. The body weights in Table 2 were obtained from the studies themselves. Because the LOAELs are based on the geometric mean of LOAELs from several studies, species body weights associated with those values are not available. Therefore, allometric scaling will not be used for those values.

4.0 CHARACTERIZATION OF EXPOSURE

This portion of the ERA includes identification of contaminant concentration data used as the exposure point concentrations (EPCs) to represent ecological exposure in various media. As discussed in Section 11.3 (Worksheet 11), the site is being divided into 0.5 acre (or smaller) exposure units based on human receptors. Composite soil samples will be collected in each exposure unit to represent an average concentration within that 0.5 acre area.

Terrestrial plants and invertebrates are exposed to chemicals in surface soil through ingestion and/or direct contact. Maximum chemical concentrations across all of the exposure using will be used as the EPCs for the initial screening step.

As discussed above, the total exposure dose of terrestrial wildlife to chemicals in soil and associated food items such as plants and invertebrates will be estimated using food chain models. Selection of a particular species is required so that intake through ingestion can be estimated. The availability of exposure parameters (i.e., body mass, and ingestion rates) are factors in selecting surrogate receptor species. The surrogate receptor species are provided in Section 2.3.2.

All detected chemicals will be included in the food chain model, even if they are not considered bioaccumulative chemicals and even if they are detected at concentrations below screening levels.

The following equation will be used to calculate the CDI for wildlife receptors:

$$CDI = \frac{[(Cf * If) + (Cs * Is)] * H}{BW}$$

Where:

CDI	=	Chronic daily intake [milligrams per kilogram (mg/kg)-day]
Cf	=	Chemical concentration in food – (see discussion below)
Cs	=	Chemical concentration in surface soil (mg/kg)
If	=	Food ingestion rate [kilograms per day (kg/day)]
Is	=	Incidental surface soil ingestion rate (kg/day)
H	=	Portion of food intake from the contaminated area (unitless)
BW	=	Body weight (kg)

Table 3 presents the body weights and ingestion rates for each of the receptor species. Chemical concentrations in food items of terrestrial invertivorous and herbivorous receptors will be calculated using soil-to-invertebrate bioaccumulation factors (BAFs), soil-to-plant BAFs, and regression equations from the USEPA Eco SSL Guidance Document (USEPA, 2007) or other published sources. The sources of the BAFs are documented in Table 4. The following equation will be used to calculate the chemical concentration in plants or invertebrates when BAFs are used:

$$C_f = C_s * BAF$$

Where:

Cf	=	Contaminant concentration in food (mg/kg)
Cs	=	Contaminant concentration in surface soil (mg/kg)
BAF	=	Biota-soil bioaccumulation factor (unitless)

Table 3 summarizes the exposure factors that will be used for the food chain model. The food ingestion rates are on a dry weight basis and were obtained or calculated from Nagy (2001). The following input parameters will be used in the dose equations under the conservative screening scenario:

- Maximum surface soil concentrations across all of the exposure units
- Conservative BAFs
- Conservative receptor body weights and ingestion rate

For refining the conservative exposure assumptions in Step 3a, the following input parameters will be used:

- Average surface soil concentrations across contiguous exposure units within the home range of the receptor species.

- Average BAFs
- Average receptor body weights and ingestion rates

5.0 RISK CHARACTERIZATION

The risk characterization is the final phase of an ERA, and compares exposure to ecological effects. It is at this phase that the likelihood of adverse effects occurring as a result of exposure to a stressor is evaluated. An ecological effects quotient (EEQ) approach will be used to characterize the potential risk to ecological receptors by comparing exposure concentrations and doses to effects data. When EEQ values exceed 1.0, it is an indication that ecological receptors are potentially at risk; additional evaluation or data may be necessary to confirm with greater certainty whether ecological receptors are actually at risk, especially since most benchmarks are developed using conservative exposure assumptions and/or studies. The EEQ value should not be construed as being probabilistic; rather, it is a numerical indicator of the extent to which an EPC exceeds or is less than a benchmark.

The EEQs for surface soil receptors will be calculated as follows:

$$EEQ = \frac{C_{ss}}{SSSL}$$

where:

EEQ	=	Ecological Effects Quotient (unitless)
C _{ss}	=	Chemical concentration in surface soil [micrograms per kilogram (µg/kg) or mg/kg]
SSSL	=	Surface soil screening level (µg/kg or mg/kg)

The EEQs for terrestrial wildlife will be calculated as follows:

$$EEQ = \frac{CDI}{TRV}$$

where:

EEQ	=	Ecological effects quotient (unitless)
CDI	=	Chronic daily intake dose (mg/kg-day)
TRV	=	Toxicity reference value (NOAEL or LOAEL) (mg/kg-day)

The final part of the screening evaluation is selection of chemicals of potential concern (COPCs). Chemicals that are not selected as COPCs are assumed to present negligible risk to ecological receptors and are not further evaluated in the ERA for those receptors. Chemicals that are initially selected as COPCs will be evaluated further in Step 3a. Ecological COPCs will be selected using the following procedures:

- Chemicals with EEQs greater than 1.0 (using screening values) will be initially selected as COPCs for plants and invertebrates because they have a potential to cause risk to those receptors.
- Chemicals with EEQs greater than 1.0 based on the conservative food chain model using NOAELs will be initially selected as COPCs for mammals and birds because they have a potential to cause risk to those receptors.
- Chemicals without screening values will be initially selected as COPCs to be conservative

6.0 STEP 3A REFINEMENT

Step 3a consists of a refinement of the conservative exposure assumptions and concentrations to evaluate the potential risks to ecological receptors (i.e., plants, invertebrates, and wildlife receptors). The objective of the Step 3a evaluation is to further refine the number of chemicals that are retained as COPCs in order to focus additional efforts (if necessary) on chemicals that are of significant ecological concern. The following describes the process that will be used to further evaluate chemicals initially selected as COPCs in soil.

For chemicals that are evaluated further in Step 3a, the following factors will be evaluated, as appropriate, to determine if the risks are great enough to warrant additional evaluations. Note that all of these factors might not be applicable for each chemical and/or receptor group.

- Magnitude of criterion exceedance: Although the magnitude of the risks may not relate directly to the magnitude of a criterion exceedance, the magnitude of the criterion exceedance may be one item used in a lines-of-evidence approach to determine the need for further site evaluation. The greater the criterion exceedance, the greater the probability and concern that an unacceptable risk exists.
- Frequency of chemical detection and spatial distribution: A chemical detected at a low frequency typically is of less concern than a chemical detected at higher frequency if toxicity and concentrations and spatial areas represented by the data are similar. All else being

- Contaminant bioavailability: Many contaminants (especially inorganics) are present in the environment in forms that are typically not bioavailable, and the limited bioavailability will be considered when evaluating the exposures of receptors to site contaminants. Contaminants with generally less bioavailability will be considered to be less toxic than the more bioavailable contaminants, all other factors being equal.
- More Appropriate Benchmarks: More appropriate benchmarks will be used to further evaluate risks to specific groups of ecological receptors (e.g., plants and invertebrates) because while screening levels are useful for initial screening, they might not be appropriate for evaluating all of the assessment endpoints.

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TABLE 1

ECOLOGICAL SCREENING LEVELS
NALF CABANISS FORMER SKEET RANGE

Chemical	Plant Screening Level ⁽¹⁾		Soil Invertebrate Screening Level ⁽¹⁾	
	Value	Source	Value	Source
PAHs (mg/kg)				
LMW PAHs	NA ⁽²⁾		29	Eco SSL (USEPA, 2007)
HMW PAHs	NA		18	Eco SSL (USEPA, 2007)

1 - The USEPA Eco SSLs for PAHs for invertebrates are provided for LMW PAHs and HMW PAHs, but the levels are for individual PAHs within each class; the screening levels are not applied to "total" PAH values.

2 - There is an ecological plant benchmark for acenaphthene of 20 mg/kg in TCEQ (2006).

NA Not available

Eco SSL - Ecological soil screening level

PAHs - polycyclic aromatic hydrocarbons

LMW - Low Molecular Weight (acenaphthylene, anthracene, fluoranthene, fluorene, phenanthrene, 1-methylnaphthalene, 2-methylnaphthalene, naphthalene)

HMW - High Molecular Weight (benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(g,h,i)perylene, chrysene, dibenzo(a,h)anthracene, indeno(1,2,3-c,d)pyrene, pyrene)

TABLE 2

NOAELS AND LOELS FOR TERRESTRIAL WILDLIFE
NALF CABANISS FORMER SKEET RANGE

Parameters	Concentration (mg/kg-day)	Endpoint	Effect	Chronic/ Subchronic	Species	Body Weight (grams)	Primary Reference	Source of Reference
PAHs								
Low Molecular Weight PAHs	356	LOAEL	reproduction & growth	chronic	mammals		USEPA, 2007	
Low Molecular Weight PAHs	65.6	NOAEL	reproduction & growth	chronic	rat	247	Verschuuren et al., 1976	USEPA, 2007
High Molecular Weight PAHs	38.4	LOAEL	reproduction & growth	chronic	mammals		USEPA, 2007	
High Molecular Weight PAHs	0.615	NOAEL	reproduction & growth	chronic	mouse	37	Culp et al., 1998	USEPA, 2007

Notes:

NOAEL = No Observed Adverse Effects Level

LOAEL = Lowest Observed Adverse Effects Level

TABLE 3

**CALCULATION OF INGESTION RATES
NALF CABANISS FORMER SKEET RANGE**

Species	Feeding Group	Body Weight (grams) ⁽¹⁾	Feeding Rate Calculation ⁽²⁾			Soil/Sediment Ingestion Rate ⁽⁴⁾		Food Ingestion Rate ⁽⁵⁾	
			a	b	Dry Matter Intake (g/day)	Conservative (g/day)	Average (g/day)	Conservative (g/day)	Average (g/day)
White-footed mouse	Herbivore	10	0.859	0.628	3.65	0.117	0.044	3.531	3.604
Mourning dove	Omnivore	150	0.67	0.627	15.51	2.155	0.946	13.350	14.560
Short-tailed shrew	Insectivore	15	0.373	0.622	2.01	0.060	0.018	1.950	1.992
American Robin	Insectivore	18.7	---	---	3.96 ⁽³⁾	0.649	0.253	3.311	3.707

1 - Body weights from USEPA (1999)

2 - Intake equation and parameters from Nagy (2001)

$$\text{Dry matter intake} = a * (\text{grams body weight})^b$$

3 - Feeding rate was estimated from field metabolic rate in Nagy (2001)

4 - Soil/sediment ingestion rate is calculated by multiplying the dry matter intake by the incidental soil/sediment ingestion rates listed below

5 - The food ingestion rates are calculated by subtracting the soil/sediment ingestion rate by the feeding rate.

Incidental soil/sediment ingestion rates			
Species	Conservative	Average	Source
White-footed mouse	3.20%	1.20%	1,2
Mourning dove	13.90%	6.10%	1
Short-tailed Shrew	3%	0.90%	1
American Robin	16.40%	6.40%	1,3

Conservative value is 90th percentile
Average value is 50th percentile

1 - USEPA (April, 2007)
2 - Based on the meadow vole.
3 - Based on the American woodcock

TABLE 4

**PLANT AND EARTHWORM BIOACCUMULATION FACTORS
NALF CABANISS FORMER SKEET RANGE**

Chemical	Plant Bioaccumulation Factors			Earthworm Bioaccumulation Factors		
	Conservative	Average	Source	Conservative	Average	Source
PAHs						
1-Methylnaphthalene	Regression equation from Eco SSL		⁽¹⁾	2.29E+00	2.29E+00	⁽¹⁾
2-Methylnaphthalene	Regression equation from Eco SSL		⁽²⁾	2.29E+00	2.29E+00	⁽⁴⁾
Acenaphthene	Regression equation from Eco SSL		⁽³⁾	1.47E+00	1.47E+00	⁽³⁾
Acenaphthylene	Regression equation from Eco SSL		⁽³⁾	2.29E+01	2.29E+01	⁽³⁾
Anthracene	Regression equation from Eco SSL		⁽³⁾	2.42E+00	2.42E+00	⁽³⁾
Benzo(a)anthracene	Regression equation from Eco SSL		⁽³⁾	1.59E+00	1.59E+00	⁽³⁾
Benzo(a)pyrene	Regression equation from Eco SSL		⁽³⁾	1.33E+00	1.33E+00	⁽³⁾
Benzo(b)fluoranthene	3.10E-01	3.10E-01	⁽³⁾	2.60E+00	2.60E+00	⁽³⁾
Benzo(g,h,i)perylene	Regression equation from Eco SSL		⁽³⁾	2.94E+00	2.94E+00	⁽³⁾
Benzo(k)fluoranthene	Regression equation from Eco SSL		⁽³⁾	2.60E+00	2.60E+00	⁽³⁾
Chrysene	Regression equation from Eco SSL		⁽³⁾	2.29E+00	2.29E+00	⁽³⁾
Dibenzo(a,h)anthracene	1.30E-01	1.30E-01	⁽³⁾	2.31E+00	2.31E+00	⁽³⁾
Fluoranthene	5.00E-01	5.00E-01	⁽³⁾	3.04E+00	3.04E+00	⁽³⁾
Flourene	Regression equation from Eco SSL		⁽³⁾	9.57E+00	9.57E+00	⁽³⁾
Indeno(1,2,3-cd)pyrene	1.10E-01	1.10E-01	⁽³⁾	2.86E+00	2.86E+00	⁽³⁾
Naphthalene	1.22E+01	1.22E+01	⁽³⁾	4.40E+00	4.40E+00	⁽³⁾
Phenanthrene	Regression equation from Eco SSL		⁽³⁾	1.72E+00	1.72E+00	⁽³⁾
Pyrene	7.20E-01	7.20E-01	⁽³⁾	1.75E+00	1.75E+00	⁽³⁾
Metals						
Lead	Regression equation from Eco SSL		⁽³⁾	Regression equation from Eco SSL		⁽³⁾

1- Value for 2-Methylnaphthalene used as a surrogate.

2- Value for low molecular weight PAHs used as a surrogate.

3 - USEPA (2007). Where "Regression equation from Eco-SSL" is given, tissue concentration will be calculated using regression equations from USEPA (2007), Attachment 4-1, Table 4A (for inorganics), Table 4B (for organics).

4 - Value calculated as described in Table 5 for the calculation of soil to earthworm bioaccumulation factors.

TABLE 5

**CALCULATION OF SOIL TO EARTHWORM BIOACCUMULATION FACTORS
NALF CABANISS FORMER SKEET RANGE**

Parameter	log K_{ow}⁽¹⁾	log K_{ww}⁽²⁾	K_{ww(wet wt)}⁽²⁾	K_{ww(dry wt)}⁽²⁾	K_{oc}	f_{oc}	K_d	BAF⁽²⁾
2-METHYLNAPHTHALENE	3.9	1.4	22.7	142	6190	0.01	61.9	2.29

1 - Source of this value is USEPA, 2003 (PAH Mixtures)

2 - These values were calculated as described in Table 5 in USEPA (2007).

APPENDIX B

FIELD STANDARD OPERATING PROCEDURES

APPENDIX B

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STANDARD OPERATING PROCEDURE

SOP-01

SAMPLE LABELING

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures to be used for labeling sample containers. Sample labels are used to document the sample ID, date, time, analysis to be performed, preservative, matrix, sampler, and the analytical laboratory. A sample label will be attached to each sample container.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Writing utensil (preferably black pen with indelible ink)

Disposable medical-grade gloves (e.g. latex, nitrile)

Sample log sheets

Required sample containers: All sample containers for analysis by fix-based laboratories will be supplied and deemed certified clean by the laboratory.

Preprinted sample labels

Chain-of-custody records

Sealable polyethylene bags

Heavy-duty cooler

Ice

3.0 PROCEDURES

3.1 The following information will be electronically printed on each sample label prior to mobilizing for field activities. Additional "generic" labels will also be printed prior to mobilization to be used for field QC and backups.

- Project number (CTO 0135)
- Sample location ID
- Contract Task Order number

- Sample ID
- Matrix
- Preservative
- Analysis to be performed
- Laboratory name

3.2 Select the container(s) that are appropriate for a given sample. Select the sample-specific ID label(s), complete date, time, and sampler name, and affix to the sample container(s).

3.3 Fill the appropriate containers with sample material. Securely close the container lids without overtightening.

3.4 Place the sample container in a sealable polyethylene bag and place in a cooler containing ice.

Example of a sample label is attached at the end of this SOP.

4.0 ATTACHMENTS

1. Sample Label

ATTACHMENT 1 SAMPLE LABEL

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

STANDARD OPERATING PROCEDURE

SOP-02

SAMPLE IDENTIFICATION NOMENCLATURE

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to establish a consistent sample nomenclature system that will facilitate subsequent data management at the Naval Auxiliary Landing Field Cabaniss. The sample nomenclature system has been devised such that the following objectives can be attained.

- Sorting of data by site, location, or matrix
- Maintenance of consistency (field, laboratory, and database sample numbers)
- Accommodation of all project-specific requirements
- Accommodation of laboratory sample number length constraints
- Ease of sample identification

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Writing utensil (preferably black pen with indelible ink)

Sample container labels

3.0 SAMPLE IDENTIFICATION NOMENCLATURE

3.1 Samples

All samples will be properly labeled with a sample label affixed to the sample container. Each sample will be assigned a unique sample tracking number.

3.1.1 Sample Numbering Scheme

The sample tracking number will consist of a four- or five-segment alpha-numeric code that identifies the sample's associated with the NALF Cabaniss site, sample type, location, and for aqueous samples, where applicable, whether a sample is filtered, and/or the sample round number. For soil samples, the

final four tracking numbers will identify the depth in units of feet below ground surface (bgs) at which the sample was collected.

The alphanumeric coding to be used is explained in the following diagram and subsequent definitions:

AA	AA	NN	NNNN (Soils only)	AA
Site Acronym	Matrix	Sample Location Number	Sequential depth interval from freshly exposed surface	Blank Type

Character Type:

A = Alpha
 N = Numeric

Site Name (NN):

SR = Skeet Range

Matrix Code (AA):

SS = Soil Sample
 GW = Groundwater

Location Number (NNA):

Sequential number beginning with "01" for each matrix.

Depth Interval:

This code section will be used for soil samples only.

The depth code is used to note the depth bgs at which a soil sample is collected. The first two numbers of the four-number code specify the top interval and the third and fourth specify the bottom interval in feet bgs of the sample. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet, boring log, logbook, etc.

Blank Type (AA):

This code section will be used for field or equipment blanks only.

RB = Rinsate Blank
 FB = Field Blank

3.1.2 Examples of Sample Nomenclature

A soil sample collected from the Skeet Range, sampling location 003, to a depth of 0 to 1 foot bgs would be labeled as "SRSS030001". A soil sample collected from the Skeet Range, sampling location 010, to a depth of 5 to 6 feet bgs would be labeled as "SRSS0100506". A rinsate blank associated with a soil sample collected from the Skeet Range, sampling location 004, to a depth of 0 to 1 foot bgs would be labeled as "SRSS040001RB"

3.2 Field Quality Assurance/Quality Control (QA/QC) Sample Nomenclature

Field QA/QC samples are described in this UFP SAP. They will be designated using a different coding system than the one used for regular field samples.

3.2.1 QC Sample Numbering

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
QC Type	Date	Sequence Number (per day)

Character Type:

A = Alpha
 N = Numeric

QC Types:

FD = Field Duplicate
 TB = Trip Blank

The sampling time recorded on the Chain-of-Custody Form, labels, and tags for field 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 sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory).

3.2.2 Examples of Field QA/QC Sample Nomenclature

The first duplicate of the day at Skeet Range site for a surface soil sample collected on March 24, 2010 would be designated as FD03241001.

The third duplicate of the day taken at Skeet Range site of a surface soil sample collected on April 12, 2010 would be designated as FD04121003.

The first trip blank associated with samples collected on March 18, 2010 would be designated as TB03181001.

STANDARD OPERATING PROCEDURE

SOP-03

SAMPLE CUSTODY AND DOCUMENTATION OF FIELD ACTIVITIES

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures for sample custody and documentation of field sampling and field analyses activities.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following logbooks, forms, labels, and equipment are required.

Writing utensil (preferably black pen with indelible ink)

Site logbook

Field logbook

Sample label

Chain-of-Custody Form

Custody seals

Equipment calibration log

Soil and Sediment Sample Log Sheet

Groundwater Sample Log Sheet

3.0 PROCEDURES

This section describes custody and documentation procedures. All entries made into the logbooks, custody documents, logs, and log sheets described in this SOP must be made in indelible ink (black is preferred). No erasures are permitted. If an incorrect entry is made, the entry will be crossed out with a single strike mark, initialed, and dated.

3.1 Site Logbook

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

- All field personnel present
- Arrival/departure of site visitors
- Arrival/departure of equipment
- Start or completion of sampling activities
- Daily on-site activities performed each day
- Sample pickup information
- Health and safety issues
- Weather conditions

The site logbook is initiated at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day that on-site activities take place.

The following information must be recorded on the cover of each site logbook:

- Project name
- Project number
- Book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). At the completion of each day's entries, the site logbook must be signed and dated by the field operations leader (FOL).

3.2 Field Logbooks

The field logbook is a separate dedicated notebook used by field personnel to document his or her activities in the field. This notebook is hardbound and paginated.

3.3 Sample Labels

Adhesive sample container labels must be completed and applied to every sample container. Information on the label includes the project name, location, sample number, date, time, preservative, analysis, matrix, sampler's initials, and the name of the laboratory performing the analysis.

3.4 Chain-of-Custody Form

The Chain-of-Custody Form (COC) is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as it is transferred from person to person. Each COC is numbered. This form must accompany any samples collected for laboratory chemical analysis. A copy of a blank COC form is attached at the end of this SOP.

The FOL must include the name of the laboratory in the "Remarks" section to ensure that the samples are forwarded to the correct location. If more than one COC is necessary for any cooler, the FOL will indicate "Page ___ of ___" on each COC. The original (top) signed copy of the COC will be placed inside a sealable polyethylene bag and taped inside the lid of the shipping cooler. Once the samples are received at the laboratory, the sample custodian checks the contents of the cooler(s) against the enclosed COC(s). Any problems are noted on the enclosed COC Form (bottle breakage, discrepancies between the sample labels, COC form, etc.) and will be resolved through communication between the laboratory point-of-contact and the Task Order Manager (TOM). The COC form is signed and retained by the laboratory and becomes part of the sample's corresponding analytical data package.

3.5 Custody Seal

The custody seal is an adhesive-backed label, and it is part of the 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 transit to the laboratory. The custody seals are signed and dated by the samplers and affixed across the opening edges of each cooler (two seals per cooler) containing environmental samples. The laboratory sample custodian will examine the custody seal for evidence of tampering and will notify the Tetra Tech TOM if evidence of tampering is observed.

3.6 Equipment Calibration Log

The Equipment Calibration Log is used to document calibration of measuring equipment used in the field. The Equipment Calibration Log documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. An Equipment Calibration Log must be maintained for each electronic measuring device requiring calibration. Entries must be made for each day the equipment is used.

3.7 Sample Log Sheets

The Soil and Sediment Sample Log Sheets are used to document the sampling of soils and sediments (see SOPs-05 and -08).

4.0 ATTACHMENTS

1. Chain-of-Custody Record
2. Equipment Calibration Log
3. Soil and Sediment Sample Log
4. Groundwater Sample Log Sheet

STANDARD OPERATING PROCEDURE

SOP-04

DECONTAMINATION OF FIELD SAMPLING EQUIPMENT

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures to be followed when decontaminating non-dedicated field sampling equipment during the field investigations.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Writing utensil (preferably black pen with indelible ink)

Non-latex rubber or plastic gloves

Cotton gloves

Field logbook

Potable water

Deionized water

LiquiNox detergent

Brushes, spray bottles, paper towels, etc.

Container to collect and transport decontamination fluids

3.0 DECONTAMINATION PROCEDURES

- 3.1 Don non-latex and/or cotton gloves and decontaminate sampling equipment (in accordance with the following steps) prior to field sampling and between samples.
- 3.2 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.
- 3.3 Wash the equipment with a solution of LiquiNox detergent. Prepare the LiquiNox wash solution in accordance with the instructions on the LiquiNox container. Collect the LiquiNox wash solution into a container. Use brushes or sprays as appropriate for the equipment. If oily residue has accumulated on the sampling equipment, remove the residue with an isopropanol wash and repeat the Liquinox wash.

- 3.4 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.
- 3.5 Rinse the equipment with deionized water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the deionized water rinsate into a container.
- 3.6 Remove excess water by air drying, shaking, or by wiping with paper towels as necessary.
- 3.7 Document decontamination by recording it in the field logbook.
- 3.8 Containerized decontamination solutions will be managed in accordance with the procedures described in SOP-09 and this UFP SAP.

STANDARD OPERATING PROCEDURE

SOP-05

SOIL CORING AND SAMPLING USING HAND AUGER TECHNIQUES

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures for collecting surface and subsurface soil cores from unconsolidated overburden materials using hand augering techniques.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Disposable medical-grade gloves (e.g., latex, nitrile)

Writing utensil (preferably black pen with indelible ink)

Indelible marker

Stainless Steel Auger Buckets

Stainless Steel Extension Rods

Cross Handle

Required decontamination materials

Bentonite pellets

Sealable polyethylene bags

Sample labels

Shipping containers (containing ice)

Disposable plastic trowels or stainless steel trowels

Stainless steel mixing bowls

Sample containers: Sample containers are certified clean by the laboratory supplying the containers.

Soil Sample Log Forms

Daily Activity Logs

Chain-of-Custody Form

Soil Boring Log

3.0 BOREHOLE ADVANCEMENT AND SOIL SAMPLING USING A HAND AUGER

Hand Augers may be employed to collect the soil cores. A hand augering system generally consists of a variety of all stainless steel bucket bits (i.e. cylinders 6-1/2" long and 2-3/4", 3-1/4", and 4" in diameter), a series of extension rods (available in 2', 3', 4' and 5' lengths), a cross handle.

3.1 The hand auger can be used in a wide variety of soil conditions. It can be used to sample soil, both from the surface, or to depths in excess of 12 feet. However, the presence of rock layers and the collapse of the borehole normally contribute to its limiting factors.

Attach a properly decontaminated bucket bit into a clean extension rod and further attach the cross handle to the extension rod.

3.2 Clear the area to be sampled of any surface debris (vegetation, twigs, rocks, litter, etc.).

3.3 Turn the hand auger sampler into the ground to a depth of 1 foot. The 0- to 1-foot depth soil interval is considered to be the surface soil. Subsurface soil samples will be collected at depths greater than 1 foot below ground surface.

3.4 After reaching the desired depth, slowly and carefully withdraw the apparatus from the borehole.

3.5 Utilizing a properly decontaminated stainless steel trowel or disposable trowel, remove the sample material from the bucket bit and place into a sealable polyethylene bag. Note in a field notebook or on a standardized data sheet any changes in the color, texture or odor of the soil.

3.6 Thoroughly homogenize the sample material and write sample ID, date, and time on the bag with an indelible marker.

3.7 Complete required information on the Soil Sample Log Sheet (copy attached at the end of this SOP). Update the Chain-of-Custody (COC) Form.

3.8 Excess soil core materials will be returned to the hole and tamped. If insufficient soil is available to fill the hole to the ground surface, then bentonite pellets mixed with the soil will be used to backfill the hole.

- 3.9 Decontaminate all soil sampling equipment in accordance with SOP-04 before collecting the next sample.
- 3.10 Soil samples will be transported to the field office where a portion of the sample will undergo XRF analysis for lead (See SOP-14).
- 3.11 For soil samples selected for fixed-base laboratory analysis, a portion of the sample will be used to fill the required sample containers as supplied by the laboratory. The sample labels will be completed and affixed to the sample container. The samples will then be packaged and shipped to the fixed-base laboratory in accordance with SOP-11.

4.0 ATTACHMENTS

1. Soil and Sediment Sample Log Sheet

STANDARD OPERATING PROCEDURE

SOP-09

MANAGEMENT OF INVESTIGATION-DERIVED WASTE

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes how investigation-derived waste (IDW) will be collected, segregated, classified, and managed during the field investigations at the NALF Cabaniss facility. The following types of IDW will be generated during this investigation:

- Decontamination solutions
- Soil and drill cuttings
- Purge and development water
- Personal protective equipment and clothing (PPE)
- Miscellaneous trash and incidental items

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Health and safety equipment (with PPE)

Decontamination equipment

Field logbook

Writing utensil (preferably black pen with indelible ink)

Plastic sheeting and/or tarps

55-gallon drums with sealable lids

IDW labels for drums

Wastewater container tanks

Plastic garbage bags

3.0 PROCEDURES

Management of IDW includes the collection, segregation, temporary storage, classification, final disposal, and documentation of the waste-handling activities if necessary.

3.1 Liquid Wastes

Liquid wastes that will be generated during the site activities include purge and development water from monitoring wells and decontamination solutions from sampling equipment. These wastes will be collected and transported to a central location at NALF Cabaniss. This waste will be containerized and handed over to the NAS Corpus Christi Environmental Department Manager at the conclusion of field activities.

3.2 Solid Wastes

Solid wastes that may be generated during the site activities include collection of soil from surface soil sampling and drill cuttings from subsurface soil sampling. These wastes will be collected and transported to a central location at NALF Cabaniss. This waste will be containerized and handed over to the NAS Corpus Christi Environmental Department Manager at the conclusion of field activities.

3.3 PPE and Incidental Trash

All PPE wastes and incidental trash materials (e.g., wrapping or packing materials from supply cartons, waste paper) will be decontaminated (if contaminated), double bagged, securely tied shut, and placed in a designated waste receptacle at NALF Cabaniss.

STANDARD OPERATING PROCEDURE SOP-10

BOREHOLE AND SOIL SAMPLE LOGGING

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the standard procedures and technical guidance on the logging of soil cores.

2.0 FIELD FORMS AND EQUIPMENT

Knife

Ruler (marked in tenths and hundredths of feet)

Boring Log: An example of this form is attached.

Photoionization detector (PID)

Writing utensil (preferably black pen with indelible ink)

3.0 RESPONSIBILITIES

A field geologist or engineer is responsible for supervising all boring activities and assuring that each borehole is properly and completely logged.

4.0 PROCEDURES FOR BOREHOLE AND SAMPLE LOGGING

To maintain a consistent classification of soil, it is imperative that the field geologist understands and accurately uses the field classification system described in this SOP. This identification is based on visual examination and manual tests.

4.1 USCS Classification

Soils are to be classified according to the Unified Soil Classification System (USCS). This method of classification is detailed in Figure 1 (attached to this SOP).

This method of classification identifies soil types on the basis of grain size and cohesiveness.

Fine-grained soils, or fines, are smaller than the No. 200 sieve and are of two types: silt (M) and clay (C). Some classification systems define size ranges for these soil particles, but for field classification purposes, they are identified by their respective behaviors. Organic material (O) is a common component of soil but has no distinguishable size range; it is recognized by its composition. The careful study of the USCS will aid in developing the competence and consistency necessary for the classification of soils.

Coarse-grained soils will be divided into categories: rock fragments, sand, or gravel. The terms "sand" and "gravel" not only refer to the size of the soil particles but also to their depositional history. To insure accuracy in description, the term "rock fragments" will be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges that are typically observed indicate little or no transport from their source area; and therefore, the term provides additional information in reconstructing the depositional environment of the soils encountered. When the term "rock fragments" is used, it will be followed by a size designation such as "(1/4 inch Φ -1/2 inch Φ)" or "coarse-sand size" either immediately after the entry or in the remarks column. The USCS classification would not be affected by this variation in terms.

4.2 Color

Soil colors will be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light gray" or "blue-gray." Because color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent from one boring to another.

Colors must be described while the sample is still moist. Soil samples will be broken or split vertically to describe colors. Samplers tend to smear the sample surface, creating color variations between the sample interior and exterior.

The term "mottled" will be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

4.3 Relative Density and Consistency

To classify the relative density and/or consistency of a soil, the geologist is to first identify the soil type. Granular soils contain predominantly sands and gravels. They are non-cohesive (particles do not adhere well when compressed). Finer-grained soils (silts and clays) are cohesive (particles will adhere together when compressed).

Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

The consistency of cohesive soils is determined by performing field tests and identifying the consistency as shown in the following table.

CONSISTENCY FOR COHESIVE SOILS

Consistency	Standard Penetration Resistance (Blows per Foot)	Unconfined Compressive Strength (Tons/Sq. Foot by pocket penetration)	Field Identification
Very soft	0 to 2	Less than 0.25	Easily penetrated several inches by fist.
Soft	2 to 4	0.25 to 0.50	Easily penetrated several inches by thumb.
Medium stiff	4 to 8	0.50 to 1.0	Can be penetrated several inches by thumb with moderate effort.
Stiff	8 to 15	1.0 to 2.0	Readily indented by thumb but penetrated only with great effort.
Very stiff	15 to 30	2.0 to 4.0	Readily indented by thumbnail.
Hard	Over 30	More than 4.0	Indented with difficulty by thumbnail.

Cohesive soils are given the USCS classifications ML, MH, CL, CH, OL, or OH (see Figure 1).

The consistency of cohesive soils is determined by hand by determining the resistance to penetration by the thumb. The thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample. The sample will be broken in half and the thumb pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. One of the other methods will be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in the above-listed table.

4.4 Weight Percentages

In nature, soils are consist of particles of varying size and shape and are combinations of the various grain types. The following terms are useful in the description of soil:

Terms of Identifying Proportion of the Component	Defining Range of Percentages by Weight
Trace	0 - 10 percent
Some	11 - 30 percent
Adjective form of the soil type (e.g., sandy)	31 - 50 percent

Examples:

- Silty fine sand: 50 to 69 percent fine sand, 31 to 50 percent silt.
- Medium to coarse sand, some silt: 70 to 80 percent medium to coarse sand, 11 to 30 percent silt.
- Fine sandy silt, trace clay: 50 to 68 percent silt, 31 to 49 percent fine sand, 1 to 10 percent clay.
- Clayey silt, some coarse sand: 70 to 89 percent clayey silt, 11 to 30 percent coarse sand.

4.5 Moisture

Moisture content is estimated in the field according to four categories: dry, moist, wet, and saturated. In dry soil, there appears to be little or no water. Saturated samples obviously have all the water they can hold. Moist and wet classifications are somewhat subjective and often are determined by the individual's judgment. A suggested parameter for this would be calling a soil wet if rolling it in the gloved hand or on a porous surface liberates water (i.e., dirties or muddies the surface). Whatever method is adopted for describing moisture, it is important that the method used by an individual remains consistent throughout an entire field activity.

4.6 Classification of Soil Grain Size for Chemical Analysis

To determine the gross grain size classification (e.g., clay, silt, and sand) from the USCS classification described above, the following table will be used.

Gross Soil Grain Size Classification	USCS Abbreviation	Description
Clay	CL	inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays.
	CH	inorganic clays of high plasticity, fat clays.
	OH	organic clays of medium to high plasticity, organic silts.
Silt	ML	inorganic silts and very fine sands, rock four, silty or clayey fine sands with slight plasticity.
	OL	organic silts and organic silty clays of low plasticity
	MH	inorganic silts, micaceous or diatomaceous fine sand or silty soils.

Gross Soil Grain Size Classification	USCS Abbreviation	Description
Sand	SW	well graded sands, gravelly sands, little or no fines.
	SP	poorly graded sands, gravelly sands, little or no fines.
	SM	silty sands, sand-silt mixtures.
	SC	clayey sands, sand-clay mixtures.

4.7 Summary of Soil Classification

In summary, soils will be classified in a similar manner by each geologist/engineer at a project site. The hierarchy of classification is as follows:

- Density and/or consistency
- Color
- Plasticity (optional)
- Soil types
- Moisture content
- Other distinguishing features
- Grain size
- Depositional environment

5.0 ATTACHMENTS

1. Figure 1 - Unified Soil Classification System
2. Boring Log

ATTACHMENT 1

FIGURE 1 - UNIFIED SOIL CLASSIFICATION SYSTEM

Unified Soil Classification System				
Coarse Grained Soils (more than half of soil > No. 200 sieve)	Gravels (More than half of coarse fraction > no. 4 sieve size)		GW	Well graded gravels or gravel-sand mixtures, little or no fines
			GP	Poorly graded gravels or gravel-sand mixtures, little or no fines
			GM	Sandy gravels, gravel-sand-silt mixtures
			GC	Clayey gravels, gravel-sand-silt mixtures
	Sands (More than half of coarse fraction < no. 4 sieve size)		SW	Well graded sands or gravelly sands, little or no fines
			SP	Poorly graded sands or gravelly sands, little or no fines
		SM	Silty sands, sand-silt mixtures	
		SC	Inorganic silts and very fine sands, rock flour, silty or clayey fine sands or clayey silts with slight plasticity	
Fine Grained Soils (more than half of soil < No. 200 sieve)	Silts and Clays LL = < 50		ML	Inorganic silts and very fine sands, rock flour, silty fine sands or clayey silts with slight plasticity
			CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, lean clays
			OL	Organic silts and organic silty clays of low plasticity
	Silts and Clays LL = > 50		MH	Inorganic silts, micaceous or diatomaceous fine sand or silty soils, elastic silts
			CH	Inorganic silts of high plasticity, fat clays
	OH	Organic clays of high plasticity, organic silty clays, organic silts		
Highly Organic Soils		Pt	Peat and other highly organic soils	

Grain Size Chart

Classification	Range of Grain Sizes	
	U.S. Standard Sieve Size	Grain Size In Millimeters
Boulders	Above 12"	Above 305
Cobbles	12" to 3"	305 to 76.2
Gravel	3" to No. 4	76.2 to 7.76
	3" to 3/4"	76.2 to 4.76
Sand	3/4" to No. 4	19.1 to 4.76
	No. 4 to No. 200	4.76 to 0.074
Silt and Clay	No. 4 to No. 10	4.76 to 2.00
	No. 10 to No. 40	2.00 to 0.420
	No. 40 to No. 200	0.420 to 0.074

Relative Density (SPT)

SANDS AND GRAVELS	BLOWS/FOOT
VERY LOOSE	0 - 4
LOOSE	4 - 10
MEDIUM DENSE	10 - 30
DENSE	32 - 50
VERY DENSE	OVER 50

Consistency (SPT)

SILTS AND CLAYS	BLOWS/FOOT
VERY SOFT	0 - 2
SOFT	2 - 4
MEDIUM STIFF	4 - 8
STIFF	8 - 16
VERY STIFF	16 - 22
HARD	OVER 22

STANDARD OPERATING PROCEDURE

SOP-11

SAMPLE PRESERVATION, PACKAGING, AND SHIPPING

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures for sample preservation, packaging, and shipping to be used in handling soil, sediment, and aqueous samples.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Shipping labels

Custody seals

Chain-of-custody (COC) form(s)

Sample containers with preservatives: All sample containers for analysis by fixed-base laboratories will be supplied, with preservatives added (if required) and deemed certified clean by the laboratory.

Sample shipping containers (coolers): All sample shipping containers are supplied by the laboratory.

Packaging material: Bubble wrap, sealable polyethylene bags, strapping tape, etc.

3.0 PROCEDURES FOR SAMPLE PRESERVATION, PACKAGING, AND SHIPPING

3.1 The laboratory provides sample containers with preservative already included (as required) for the analytical parameter for which the sample is to be analyzed. All samples will be held, stored, and shipped at 4°C. This will be accomplished through refrigeration (used to hold samples prior to shipment) and/or ice.

3.2 The sampler shall maintain custody of the samples until the samples are relinquished to another custodian or to the common carrier.

3.3 Check that each sample container is properly labeled, the container lid is securely fastened, and the container is sealed in a polyethylene bag.

3.4 If the container is glass, place the sample container into a bubble-out shipping bag and seal the bag using the self-sealing, pressure sensitive tape supplied with the bag.

- 3.5 Inspect the insulated shipping cooler. Check for any cracks, holes, broken handles, etc. If the cooler has a drain plug, make certain it is sealed shut, both inside and outside of the cooler. If the cooler is questionable for shipping, the cooler must be discarded.
- 3.6 Put ice into sealable polyethylene bags and place a layer of the sealed bags on the bottom of the cooler. Place the sample containers into the shipping cooler on top of the ice in an upright position (containers will be upright, with the exception of any 40-ml vials). Place sealable polyethylene bags of ice flat against the sides of the cooler. Continue filling the cooler with samples until the cooler is nearly full and the movement of the sample containers is limited.
- 3.7 Add a final layer of ice sealed in polyethylene bags to the top of the samples just before the cooler is closed and sealed.
- 3.8 Place the original (top) signed copy of the COC form inside a sealable polyethylene bag. Tape the bag to the inside of the lid of the shipping cooler.
- 3.9 Close the cooler and seal the cooler with approximately four wraps of strapping tape at each end of the cooler. Prior to wrapping the last wrap of strapping tape, apply a signed and dated custody seal to each side of the cooler (one per side). Cover the custody seal with the last wrap of tape. This will provide a tamper evident custody seal system for the sample shipment.
- 3.10 Affix shipping labels to each of the coolers, ensuring all of the shipping information is filled in properly. Overnight (e.g., FedEx Priority Overnight) courier services will be used for all sample shipments.
- 3.11 All samples will be shipped to the laboratory no more than 72 hours after collection. Under no circumstances should sample hold times be exceeded.

STANDARD OPERATING PROCEDURE SOP-15

MONITORING WELL DEVELOPMENT

1.0 PURPOSE

This procedure provides general guidance and information pertaining to proper development of new and existing monitoring wells. The methods described herein are specific for monitoring wells located at NALF Cabaniss.

2.0 RESPONSIBILITIES

The drilling contractor or TtNUS personnel shall provide adequate and operable equipment, sufficient quantities of materials, and an experienced and efficient labor force capable of developing monitoring wells. The field personnel must have all the health and safety training required to perform the work, as specified in the health and safety plan (HASP).

3.0 REQUIRED EQUIPMENT/ITEMS

The following list includes equipment and items required for monitoring well development:

Health and safety equipment as required by the HASP and the site safety officer.

Well development equipment with associated materials (supplied by the driller or TtNUS).

Hydrogeologic equipment (water-level indicator, electronic calculator, clipboard, paint and ink marker for marking existing monitoring wells, well development forms, and a field notebook).

4.0 WELL DEVELOPMENT METHODS

The development of new monitoring wells will not occur until at least 24 hours after the well has been installed and grouted. This time is required so that the grout in the annulus can set and harden. The purpose of well development is to stabilize and increase the permeability of the sand pack and the well screen and to restore the permeability of the formation that may have been reduced by drilling operations.

Wells are typically developed until all fine material and drilling water, if any, is removed from the well. Wells will be developed by bailing and surging, and/or by pumping and surging, as determined by the TtNUS field geologist. The subcontractor may provide the surge block and pump used during development. The wells will be developed until the discharge water is visibly clear or as determined by the TtNUS field geologist. The TtNUS field geologist will obtain field parameters, such as pH, temperature, conductivity, and turbidity during development. All development water will be containerized in 55-gallon drums in accordance with the master plans.

A surge block or a stainless steel bailer that is approximately the same diameter as the well casing may be used to agitate the water, causing it to move in and out of the screens. This movement of water pulls fine materials into the well, where they may be removed by any of several methods, and prevents bridging of sand particles in the gravel pack.

Development should proceed until the following criteria are met:

- The well water is clear to the unaided eye.
- When field parameters become stable +/- 10%.

or

- A minimum removal of five times the standing water volume in the well (to include the well screen and casing plus saturated borehole annulus, assuming 30% annular porosity).

If for any reason the above criteria cannot be met, the site geologist should document the event in writing and consult with the Task Order Manager regarding an alternate plan of action.

Well development must be completed at least 24 hours before well sampling. The intent of this hiatus is to provide time for the groundwater surrounding the newly developed well to sufficiently equilibrate to static conditions.

5.0 ATTACHMENTS

1. Monitoring Well Development Record

STANDARD OPERATING PROCEDURE SOP-16

TEMPORARY MONITORING WELL INSTALLATION

1.0 PURPOSE

This procedure provides general guidance and information pertaining to proper design and installation of temporary groundwater monitoring wells. The methods described herein are specific for temporary monitoring well construction at NALF Cabaniss.

2.0 RESPONSIBILITIES

Driller - The driller provides adequate and operable equipment, sufficient quantities of materials, and an experienced and efficient labor force capable of performing all phases of proper monitoring well installation and construction. The drilling contractor personnel must have all the health and safety training required to perform the work, as specified in the health and safety plan. All well drilling activities shall be performed under the direct supervision of a driller licensed in the State of Texas. The driller is also responsible for obtaining, in advance, any required permits for drilling and monitoring well installation and construction for the State of Texas.

Field Geologist - The field geologist supervises and documents well installation and construction performed by the driller and ensures that the screen interval for each monitoring well is properly placed to provide representative groundwater data from the monitored interval. Geotechnical engineers, field technicians, or other suitable trained personnel may also serve in this capacity.

Site Safety Officer – The site safety officer is responsible for clearing the drill site for underground and overhead utilities or other potentially hazardous obstructions.

3.0 REQUIRED EQUIPMENT/ITEMS

The following list includes equipment and items required for monitoring well installation:

Health and safety equipment as required by the HASP and the site safety officer.

Well drilling and installation equipment with associated materials (typically supplied by the driller). Wells can be installed using either hollow-stem auger (HSA) or Direct push techniques (DPT) drilling methods.

Hydrogeologic equipment (weighted engineer's tape, water-level indicator, retractable engineer's rule, electronic calculator, clipboard, mirror and flashlight for observing downhole activities, paint and ink marker for marking monitoring wells, sample jars, well installation forms, boring logs, soil sample log forms, chain-of-custody records, sample coolers with ice, and a field notebook).

4.0 WELL DESIGN, CONSTRUCTION, AND ABANDONMENT

Wells can be installed using either hollow-stem auger (HSA) or direct push techniques (DPT) drilling methods. The following SOP provides procedures for both methods.

Temporary wells shall be constructed using nominal 2-inch ID, PVC riser and nominal 2-inch ID, PVC factory slotted screen (.010 slot). Clean silica sand of U.S. Standard Sieve Size No. 20 to 40 will be used for the sand pack, 100 percent certified pure sodium bentonite will be used for the seal above the sand pack and hydrated. The depths of backfill materials will be constantly monitored, if possible, during well installation using a weighted stainless-steel or fiberglass tape measure. The well boring will be abandoned using cement bentonite grout.

DIRECT PUSH TECHNIQUES

The following procedures will be used for DPT installation. The temporary well will be installed by driving a nominal 2-inch ID drill casing (with an expendable tip) to the desired depth. After the casing has been advanced to approximately 8 to 9 feet below the water table or to the first water bearing zone, a 10-foot-long screen attached to the riser pipe will be lowered to the bottom through the casing. The casing will then be withdrawn from the ground, exposing the PVC screen to the formation material. The saturated formation material may collapse around the screen, and the remaining annular space around the screen will be filled with silica sand to at least 1 to 2 feet above the screen. A bentonite seal will then be installed to the ground surface completing the temporary well construction.

HOLLOW-STEM AUGER DRILLING

The following procedures shall be used for hollow-stem auger drilling for well installation. Nominal 3½ or 4½-inch ID hollow-stem augers will be used to install the well borings. All hollow-stem auger drilling will

include continuous split-spoon sampling to the bottom of the boring as per SOP-10. Once the boring reaches the desired depth, the screen and the riser pipe are in place, the annulus of the boring will be backfilled with clean silica sand filter pack from the bottom of the boring to 2 to 3 feet above the top of the well screen. As the filter pack is being installed, the level of sand will be several inches up inside of the augers to ensure that an adequate filter pack is installed around the well screen. A bentonite seal will be installed above the filter pack to the ground surface or approximately 2 linear feet whichever is the smaller length. The depths of the backfill materials will be constantly monitored during the monitoring well installation with a weighted stainless steel or plastic tape. The exact depth and thickness of backfill materials will be determined in the field by the TtNUS representative.

The Subcontractor shall be responsible for measuring backfill placement in the wells to the satisfaction of the TtNUS representative. The annular space at the ground surface will be covered with plastic sheeting around the riser if needed to prevent infiltration of surface runoff or rainwater into the annulus. The riser pipe will be capped to prevent rain water from entering into the well and will remain in place until the point is abandoned.

Once the well has been sampled by TtNUS personnel, the Subcontractor shall abandon the well in accordance with State of Texas regulations. This requires that the PVC screen and riser be removed, if possible, from the boring and the boring backfilled with cement/bentonite grout from the bottom up using a tremie pipe.

5.0 DOCUMENTATION OF FIELD ACTIVITIES

A critical part of monitoring well installation is recording of significant details and events in the site logbook, on field forms, and in a field logbook. Details of borehole logging are contained in SOP-10.

6.0 ATTACHMENTS

1. Overburden Monitoring Well Sheet

ATTACHMENT 1
OVERBURDEN MONITORING WELL SHEET



Tetra Tech NUS, Inc.

TEMPORARY OVERBURDEN MONITORING WELL SHEET

BORING NO.: _____

PROJECT: _____	DRILLING Co.: _____	BORING No.: _____
PROJECT No.: _____	DRILLER: _____	DATE COMPLETED: _____
SITE: _____	DRILLING METHOD: _____	NORTHING: _____
GEOLOGIST: _____	DEV. METHOD: _____	EASTING: _____

	ELEVATION OF TOP OF SURFACE CASING: _____
	STICK -UP TOP OF SURFACE CASING: _____
	ELEVATION OF TOP OF RISER PIPE: _____
	RISER STICK-UP ABOVE GROUND SURFACE: _____
	I.D. OF SURFACE CASING: _____
	TYPE OF SURFACE CASING: _____
	GROUND ELEVATION: _____
	TYPE OF SURFACE SEAL: _____
	RISER PIPE I.D.: _____
	TYPE OF RISER PIPE: _____
	BOREHOLE DIAMETER: _____
	TYPE OF SEAL: _____
	ELEVATION / DEPTH OF SEAL: _____ /
	TYPE OF SEAL: _____
	ELEVATION / DEPTH TOP OF FILTER PACK: _____ /
ELEVATION / DEPTH TOP OF SCREEN: _____ /	
TYPE OF SCREEN: _____	
SLOT SIZE X LENGTH: _____	
I.D. OF SCREEN: _____	
TYPE OF FILTER PACK: _____	
ELEVATION / DEPTH BOTTOM OF SCREEN: _____ /	
ELEVATION / DEPTH BOTTOM OF FILTER PACK: _____ /	
TYPE OF BACKFILL BELOW WELL: _____	
ELEVATION / DEPTH OF BOREHOLE: _____ /	

STANDARD OPERATING PROCEDURE SOP-17

LOW-FLOW WELL PURGING AND STABILIZATION

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for well purging and stabilization utilizing low-flow techniques.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following field forms and equipment are required for low-flow purging.

Low-Flow Purge Data Sheet: A copy of this form is attached at the end of this SOP.

Ground Water Sample Log Sheet: A copy of this form and instructions for its completion are included in SOP-18.

Bound field logbook

Writing utensil

Well key

Electronic water-level indicator: The water-level indicator must have a cable of sufficient length to reach the water surface and be capable of measurements of 0.01 foot.

Submersible Bladder Pump: QED Sample Pro or equivalent using twin bonded ¼-inch PE tubing.

Electronic Programmable Controller, MP-10: This controller regulates air flow in a bladder pump.

Cylinder of compressed nitrogen with regulator: Compressed gas serves as the power source for the bladder pump.

Peristaltic Pump: Using siliclastic tubing and ¼-inch PE tubing

Multiple parameter water-quality meter: This unit measures and displays field parameters measured in the field including pH, dissolved oxygen, oxidation-reduction potential (ORP), temperature, and specific conductance (see SOP-12).

Flow-through cell adapter for water-quality meter

LaMotte Turbidity Meter: Used to measure turbidity.

Purge water containers

Graduated cylinder and stopwatch: Used to calculate flow rate.

Decontamination supplies: SOP-04 describes required decontamination supplies.

Disposable medical-grade gloves (e.g., latex, nitrile)

3.0 PROCEDURES FOR WELL PURGING

- 3.1 Prior to mobilizing to the site, clean, check for proper operation, and calibrate above equipment in accordance with manufacturer requirements as necessary.
- 3.2 Obtain a static water-level measurement of the well to be purged. Record the information on the Groundwater Sample Log Sheet (see SOP-18) and the Low-Flow Purge Data Sheet. Leave the water-level meter suspended in the well casing.
- 3.3 Calculate saturated screen length well casing volume as follows:
 1. Obtain the total depth of the well by measurement.
 2. Using the static water level determined in Step 3.2 of this SOP, the total depth of the well and the screen length, calculate the saturated screen length well casing volume using the following formula:

$$V = (0.163)(T)(r^2)$$

where:

- | | | |
|-------|---|---|
| V | = | Static casing volume of well (in gallons). |
| T | = | Vertical height of water column (linear feet of water) across the screen interval. |
| 0.163 | = | A constant conversion factor that compensates for the conversion of the casing radius from inches to feet, the conversion of cubic feet to gallons, and pi. |
| r | = | Inside radius of the well casing (in inches). |

Note: For wells of 1-inch radius (2-inch diameter), $V = 0.163$ gallons per foot of water column. The minimum purge volume should be two saturated screen lengths.

- 3.4 Wells shall be purged using either a submersible bladder pump or surface peristaltic pump. For wells with a nominal ID of 1-inch the peristaltic pump will be used to purge and sample the well.

If the depth to water level exceeds the capacity (about 25 ft to water) of the peristaltic pump then a submersible bladder pump will be used to purge and sample the well. Follow steps 3.5 through 3.9 for bladder pump procedures skip to 3.10 for peristaltic pump procedures.

- 3.5 Connect the pump controller to the well pump air supply (at the well cap) by following the instructions in the pump control manual. The pump controller must be turned off when it is being connected.
- 3.6 Connect the nitrogen cylinder to the pump controller. The nitrogen cylinder valve must be closed and the regulator line pressure set at zero pounds per square inch (psi) when it is being connected.
- 3.7 Following the instructions found in the water-quality meter manual, connect the flow-through cell to the pump discharge line (at the well cap).
- 3.8 Place the discharge tubing from the flow-through cell to direct the purge water discharge into the graduated cylinder or purge water container.
- 3.9 Following the instructions in the pump controller manual, start pumping water from the well.
- 3.10 Peristaltic pump be used to purge and sample groundwater monitoring wells. Attach monitoring well tubing to the input side of the pump via the siliclastic tubing and the out from the pump to the input side of the flow through cell.
- 3.11 Start with the initial pump rate set at approximately 0.1 liters per minute. Use the graduated cylinder and stopwatch to measure the pumping rate. Adjust pumping rates as necessary to prevent drawdown from exceeding 0.3 foot during purging. If no drawdown is noted, the pump rate may be increased (to a max of 0.5 liters per minute) to expedite the purging and sampling event. The pump rate will be reduced if turbidity is greater than 10 NTUs after all other field parameters have stabilized. Slow recovering wells will be identified and purged at the beginning of the workday. If possible, samples will be collected from these wells within the same 8-hour workday and no later than 24 hours after the start of purging.

The time to sample any given well will vary greatly due to the many variables associated with low flow purging and sampling:

- Stabilization of parameters
- Possible drawdown
- Variable pump rates

Normally, the time from the start of purging to the end of sampling will be between 1 and 4 hours.

- 3.12 Measure the well water level using the water-level meter every 5 minutes. Record the well water level on the Low-Flow Purge Data Form (attached at the end of this SOP).
- 3.13 Every 5 minutes, record on the Low-Flow Purge Data Form the water-quality parameters (pH, specific conductance, temperature, turbidity, oxidation-reduction potential, and dissolved oxygen) measured by the water-quality meter and turbidity meter. If the cell needs to be cleaned during purging operations, continue pumping (allow the pump to discharge into a container) and disconnect the cell. Rinse the cell with distilled water. After cleaning is completed, reconnect the flow-through cell and continue purging. Document the cell cleaning on the Low-Flow Purge Data Form.
- 3.14 Measure the flow rate using a graduated cylinder. Remeasure the flow rate any time the pump rate is adjusted.
- 3.15 During purging, check for the presence of bubbles in the flow-through cell. The presence of bubbles is an indication that connections are not tight. If bubbles are observed, check for loose connections.
- 3.16 Stabilization is achieved and sampling can begin when a minimum of two saturated screen lengths volume has been removed and three consecutive readings, taken at 5 minute intervals, are within the following limits:
- pH \pm 0.1 standard units
 - Specific conduct \pm 5%
 - Temperature \pm 10%
 - Turbidity less than 10 NTUs
 - Dissolved oxygen \pm 10%

If the above conditions have still not been met after the well has been purged for 4 hours, purging will be considered complete and sampling can begin. For the temporary wells, if the above condition(s) have not been met after three well point volumes have been removed, this will be recorded on the field sample form and the groundwater sample collection can commence.

Record the final well stabilization parameters from the Low-Flow Purge Data Form onto the Groundwater Sample Log Form.

If there is a need to leave a well during purging, there are two options:

- One, if the sampler must move for 30 minutes or less but still has a clear line of sight to the well, the sampler may leave the pump running and watch the well from a distance until he or she is able to return to the well.
- Two, if for whatever reason, the sampler must stop purging for an extended period of time or a clear line of sight cannot be maintained, the pump and cell will be shut down. All equipment and supplies will be loaded into the sample vehicle, and the well will be secured before the sampler departs.

In both cases, the time purging was stopped and restarted will be noted on the Low-Flow Purge Data Form.

- 3.17 Rinse the flow-through cell, the water-quality meter probes, and the turbidity cell with analyte-free water and pack the cell and meters for transport.

4.0 ATTACHMENTS

1. Low-Flow Purge Data Sheet

STANDARD OPERATING PROCEDURE SOP-18

GROUNDWATER SAMPLING

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for collecting groundwater samples from permanent and temporary monitoring wells. Low-flow sampling techniques will be used for groundwater sampling at NALF Cabaniss.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following field forms and equipment are required for low-flow sampling of monitoring wells:

Writing utensil (preferably black ink)

Stainless steel Geoprobe Screen Point Groundwater Sampler (or equivalent)

Groundwater Sample Log Form: A copy of this form is attached at the end of this SOP

Low Flow Purge Data Sheet: A copy of this form is attached at the end of SOP-17

Bound field log book

Chain-of-Custody Form

Bladder pump: With accessories: twin bonded ¼-inch tubing, MP-10 control box, nitrogen gas cylinder, and nitrogen regulator.

Peristaltic pump: Silicon and ¼-inch tubing.

Required sample containers with appropriate preservative: All sample containers for analysis by fixed-base laboratories will be supplied and deemed certified clean by the laboratory.

Surgical gloves

Water-level indicator

0.45-micron filter cartridge: If the metal analysis requires field filtering.

Bucket: to collect development/purge water

Calculator, wristwatch, and timer

Stainless steel clamps

Plastic storage bags

Shipping containers with ice

3.0 SAMPLING PROCEDURES FOR MONITORING WELLS

- 3.1 Groundwater sampling may be initiated when the monitoring well has been purged and stabilized in accordance with SOP-17.
- 3.2 Record the sample start time (using military time) on the Groundwater Sample Log Sheet. Record the field measurements for pH, oxidation-reduction potential (ORP), specific conductance, temperature, dissolved oxygen, and turbidity.
- 3.3 With the pump continuing to run, disconnect the flow-through cell from the pump discharge tube and immediately start filling sample bottles directly from the pump discharge. All sample containers will be supplied by the laboratory, and the laboratory will pre-preserve all sample containers, where appropriate.
- 3.4 Allow the pump discharge to flow gently down the inside of the container with minimal turbulence when filling sample containers. Avoid immersing the discharge tube into the sample as the sample container is being filled. Sample containers for volatile constituents (VOCs) must be completely filled so that no headspace exists in the container. The VOC vials will be filled to the top so that a convex meniscus is formed. Gently secure the cap, turn the vial upside down, and check to see if any air has been trapped inside the vial. If so, open the cap, reform the meniscus,

and attempt again to secure the lid without trapping air in the sample. All other sample containers can have air space included when the container lid is secured.

- 3.5 Cap each container immediately after filling.
- 3.6 Record the sample time on the Groundwater Sample Log Form, the sample label, and the sample label.
- 3.7 Place the tagged sample container into a plastic storage bag and then into a cooler containing ice.
- 3.8 Enter the proper information on the Chain-of-Custody Form for each sample container (see SOP-03).
- 3.9 Repeat steps 3.3 through 3.9 for each sample container collected.
- 3.10 The pump rate should not be adjusted after sampling has commenced. If it becomes necessary to adjust the pump rate, document the change on the Groundwater Sample Log Form.
- 3.11 All samples will be collected into pre-preserved bottles (if required) supplied by an approved laboratory. All samples will be collected in the following sequence (where applicable):
 - Volatile organic compounds (VOCs)
 - Other organics
 - Metals
 - Other Inorganics
 - Filtered Metals
- 3.12 Filtered aliquots of groundwater may be collected and analyzed for dissolved metals. Without turning off the pump, attach a disposable, inline, 0.45-um filter cartridge at the end of the discharge tube. Fill sample containers marked for dissolved metals so that the laboratory knows that these aliquots are distinct sample fractions and that the results should be reported as dissolved analytes.
- 3.13 Repeat steps 3.5 through 3.9 for the filtered sample containers.

- 3.14 After completion of sample collection, remove the bladder pump (if bladder pump is used for sampling) from the well and decontaminate the pump following the procedures in SOP-04. Leave dedicated tubing inside the well for possible future sampling events.
- 3.15 Replace the outer protective well cap and lock the well.
- 3.16 All equipment should be cleaned and packed into the sample vehicle, along with the sample cooler for transport. Disposable gloves and other equipment should be placed in a plastic trash bag and handled as investigation-derived waste (SOP-09).

4.0 SAMPLING PROCEDURES FOR TEMPORARY MONITORING WELLS

- 4.1 Groundwater samples shall be collected from temporary monitoring wells. Temporary monitoring wells shall be constructed as per SOP-16.
- 4.2 The temporary monitoring wells shall be developed as per SOP-15 prior to purging.
- 4.3 The temporary monitoring wells shall be purged as per SOP-17.
- 4.4 Samples will be collected using the peristaltic pump following procedures in Section 3.0 of this SOP.
- 4.5 Proceed to abandon the temporary well as per SOP-16.

5.0 ATTACHMENTS

- 1. Groundwater Sample Log Sheet
- 2. Low Flow Purge Data Sheet

APPENDIX C

LABORATORY STANDARD OPERATING PROCEDURES

AND

DOD ELAP ACCREDITATION

(provided on attached CD)



State of Florida
 Department of Health, Bureau of Laboratories
 This is to certify that

E87604

KATAHDIN ANALYTICAL SERVICES, INC.
 600 TECHNOLOGY WAY
 SCARBOROUGH, ME 04074

has complied with Florida Administrative Code 64E-1,
 for the examination of Environmental samples in the following categories

DRINKING WATER - GROUP II UNREGULATED CONTAMINANTS, DRINKING WATER - OTHER REGULATED CONTAMINANTS, DRINKING WATER - MICROBIOLOGY, DRINKING WATER - PRIMARY INORGANIC CONTAMINANTS, DRINKING WATER - SECONDARY INORGANIC CONTAMINANTS, DRINKING WATER - RADIOCHEMISTRY, DRINKING WATER - SYNTHETIC ORGANIC CONTAMINANTS, NON-POTABLE WATER - EXTRACTABLE ORGANICS, NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER - METALS, NON-POTABLE WATER - MICROBIOLOGY, NON-POTABLE WATER - PESTICIDES-HERBICIDES-PCB'S, NON-POTABLE WATER - VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS - EXTRACTABLE ORGANICS, SOLID AND CHEMICAL MATERIALS - GENERAL CHEMISTRY, SOLID AND CHEMICAL MATERIALS - METALS, SOLID AND CHEMICAL MATERIALS - PESTICIDES-HERBICIDES-PCB'S, SOLID AND CHEMICAL MATERIALS - VOLATILE ORGANICS, BIOLOGICAL TISSUE - GENERAL CHEMISTRY, BIOLOGICAL TISSUE - PESTICIDES-HERBICIDES-PCB'S

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

EFFECTIVE July 01, 2010 THROUGH June 30, 2011



Max Saifinger

Max Saifinger, M.D.
 Chief, Bureau of Laboratories
 Florida Department of Health
 DH Form 1697, 7/04

NON-TRANSFERABLE E87604-15-07/01/2010
 Supersedes all previously issued certificates

Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

**Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074**

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,1,1-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,1,2,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,1,2-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,1-Dichloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,1-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,1-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,2,3-Trichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/26/2002
1,2,3-Trichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,2,4-Trichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,2,4-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,2-Dibromo-3-chloropropane (DBCP)	EPA 504.1	Synthetic Organic Contaminants	NELAP	2/4/2002
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 504.1	Synthetic Organic Contaminants	NELAP	2/4/2002
1,2-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,2-Dichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,2-Dichloropropane	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
1,3,5-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,3-Dichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,3-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
1,4-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
2,2-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
2-Butanone (Methyl ethyl ketone, MEK)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
2-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
2-Hexanone	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
4-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
4-Isopropyltoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
4-Methyl-2-pentanone (MIBK)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Acetone	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Alkalinity as CaCO ₃	SM 2320 B	Primary Inorganic Contaminants	NELAP	4/26/2002
Aluminum	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/4/2002
Aluminum	EPA 200.8	Secondary Inorganic Contaminants	NELAP	4/26/2002
Amenable cyanide	SM 4500-CN G	Primary Inorganic Contaminants	NELAP	2/4/2002
Antimony	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Arsenic	EPA 200.8	Primary Inorganic Contaminants	NELAP	4/26/2002
Barium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Barium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: **E87604** EPA Lab Code: **ME00019** (207) 874-2400

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Drinking Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
Benzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Beryllium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Beryllium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Bromobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Bromochloromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Bromodichloromethane	EPA 524.2	Group II Unregulated Contaminants, Other Regulated Contaminants	NELAP	2/4/2002
Bromoform	EPA 524.2	Group II Unregulated Contaminants, Other Regulated Contaminants	NELAP	2/4/2002
Cadmium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Cadmium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Calcium	CA-628-01(EPA 200.8)/ICP-MS	Primary Inorganic Contaminants	NELAP	11/7/2006
Calcium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Carbon disulfide	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Carbon tetrachloride	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Chloride	EPA 300.0	Secondary Inorganic Contaminants	NELAP	4/26/2002
Chloride	EPA 325.2	Secondary Inorganic Contaminants	NELAP	2/4/2002
Chlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Chloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Chloroform	EPA 524.2	Group II Unregulated Contaminants, Other Regulated Contaminants	NELAP	2/4/2002
Chromium	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Chromium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
cis-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
cis-1,3-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Color	EPA 110.2	Secondary Inorganic Contaminants	NELAP	2/4/2002
Color	SM 2120 B	Secondary Inorganic Contaminants	NELAP	4/17/2007
Copper	EPA 200.7	Secondary Inorganic Contaminants, Primary Inorganic Contaminants	NELAP	2/4/2002
Copper	EPA 200.8	Secondary Inorganic Contaminants, Primary Inorganic Contaminants	NELAP	2/4/2002
Cyanide	EPA 335.4	Primary Inorganic Contaminants	NELAP	2/4/2002
Dibromochloromethane	EPA 524.2	Group II Unregulated Contaminants, Other Regulated Contaminants	NELAP	2/4/2002
Dibromomethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604 EPA Lab Code: ME00019 (207) 874-2400

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dichlorodifluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Dichloromethane (DCM, Methylene chloride)	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Diethyl ether	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Escherichia coli	SM 9223 B	Microbiology	NELAP	3/22/2010
Ethylbenzene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Fluoride	SM 4500 F-C	Secondary Inorganic Contaminants, Primary Inorganic Contaminants	NELAP	2/4/2002
Heterotrophic plate count	SIMPLATE	Microbiology	NELAP	11/7/2006
Hexachlorobutadiene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Iron	CA-628-01(EPA 200.8)/ICP-MS	Primary Inorganic Contaminants	NELAP	11/7/2006
Iron	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/4/2002
Isopropylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Lead	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Magnesium	CA-628-01(EPA 200.8)/ICP-MS	Primary Inorganic Contaminants	NELAP	11/7/2006
Magnesium	EPA 200.7	Primary Inorganic Contaminants	NELAP	4/26/2002
Manganese	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/4/2002
Manganese	EPA 200.8	Secondary Inorganic Contaminants	NELAP	2/4/2002
Mercury	EPA 245.1	Primary Inorganic Contaminants	NELAP	2/4/2002
Methyl bromide (Bromomethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Methyl chloride (Chloromethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Methyl tert-butyl ether (MTBE)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Naphthalene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
n-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Nickel	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Nickel	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Nitrate	EPA 300.0	Primary Inorganic Contaminants	NELAP	4/26/2002
Nitrate	EPA 353.2	Primary Inorganic Contaminants	NELAP	2/4/2002
Nitrate-nitrite	EPA 300.0	Primary Inorganic Contaminants	NELAP	4/26/2002
Nitrite	EPA 300.0	Primary Inorganic Contaminants	NELAP	4/26/2002
Nitrite	EPA 353.2	Primary Inorganic Contaminants	NELAP	2/4/2002
n-Propylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Orthophosphate as P	EPA 300.0	Primary Inorganic Contaminants	NELAP	7/30/2004
Perchlorate	EPA 314.0	Secondary Inorganic Contaminants	NELAP	7/30/2004
pH	EPA 150.1	Primary Inorganic Contaminants, Secondary Inorganic Contaminants	NELAP	2/4/2002

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
pH	SM 4500-H+-B	Secondary Inorganic Contaminants	NELAP	4/17/2007
sec-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Selenium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Silica as SiO ₂	EPA 200.7	Primary Inorganic Contaminants	NELAP	2/4/2002
Silver	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/4/2002
Silver	EPA 200.8	Secondary Inorganic Contaminants	NELAP	2/4/2002
Sodium	CA-628-01(EPA 200.8)/ICP-MS	Primary Inorganic Contaminants	NELAP	11/7/2006
Sodium	EPA 200.7	Primary Inorganic Contaminants	NELAP	4/26/2002
Styrene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Sulfate	ASTM D516-02	Secondary Inorganic Contaminants	NELAP	5/8/2009
Sulfate	ASTM D516-90	Secondary Inorganic Contaminants	NELAP	5/8/2009
Sulfate	EPA 300.0	Secondary Inorganic Contaminants, Primary Inorganic Contaminants	NELAP	2/4/2002
tert-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Tetrachloroethylene (Perchloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Tetrahydrofuran (THF)	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Thallium	EPA 200.8	Primary Inorganic Contaminants	NELAP	2/4/2002
Toluene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Total coliforms	SM 9223 B	Microbiology	NELAP	3/22/2010
Total dissolved solids	EPA 160.1	Secondary Inorganic Contaminants	NELAP	2/4/2002
Total dissolved solids	SM 2540 C	Secondary Inorganic Contaminants	NELAP	2/4/2002
Total nitrate-nitrite	EPA 353.2	Primary Inorganic Contaminants	NELAP	2/4/2002
Total organic carbon	SM 5310 B	Primary Inorganic Contaminants	NELAP	5/8/2009
Total trihalomethanes	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
trans-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
trans-1,3-Dichloropropylene	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Trichloroethene (Trichloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Trichlorofluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	2/4/2002
Turbidity	EPA 180.1	Secondary Inorganic Contaminants	NELAP	2/4/2002
Uranium	EPA 200.8	Radiochemistry	NELAP	11/7/2006
Vinyl chloride	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Xylene (total)	EPA 524.2	Other Regulated Contaminants	NELAP	2/4/2002
Zinc	EPA 200.7	Secondary Inorganic Contaminants	NELAP	2/4/2002
Zinc	EPA 200.8	Secondary Inorganic Contaminants	NELAP	2/4/2002

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State Laboratory ID: E87604 EPA Lab Code: ME00019 (207) 874-2400

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,1-Trichloroethane	EPA 624	Volatile Organics	NELAP	2/4/2002
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,1-Trichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2,2-Tetrachloroethane	EPA 624	Volatile Organics	NELAP	2/4/2002
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,2,2-Tetrachloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2,2-Tetrachloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2,2-Tetrachloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloro-1,2,2-trifluoroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2-Trichloro-1,2,2-trifluoroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloro-1,2,2-trifluoroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloroethane	EPA 624	Volatile Organics	NELAP	2/4/2002
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,2-Trichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2-Trichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethane	EPA 624	Volatile Organics	NELAP	2/4/2002
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1-Dichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethylene	EPA 624	Volatile Organics	NELAP	2/4/2002
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1-Dichloroethylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003

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Expiration Date: 6/30/2011



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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,3-Trichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,3-Trichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2,4,5-Tetrachlorobenzene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
1,2,4-Trichlorobenzene	EPA 625	Extractable Organics	NELAP	2/4/2002
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2,4-Trichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2,4-Trichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,4-Trichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromo-3-chloropropane (DBCP)	EPA 504	Volatile Organics	NELAP	2/4/2002
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8011	Volatile Organics	NELAP	7/1/2003
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromo-3-chloropropane (DBCP)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dibromo-3-chloropropane (DBCP)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dibromo-3-chloropropane (DBCP) (with SIM)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 504	Volatile Organics	NELAP	2/4/2002
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8011	Volatile Organics	NELAP	7/1/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dibromoethane (EDB, Ethylene dibromide)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dibromoethane (EDB, Ethylene dibromide) (with SIM)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	2/4/2002
1,2-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	2/4/2002
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003

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State Laboratory ID: **E87604** EPA Lab Code: **ME00019** (207) 874-2400

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: **Non-Potable Water**

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2-Dichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloroethane	EPA 624	Volatile Organics	NELAP	2/4/2002
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloropropane	EPA 624	Volatile Organics	NELAP	2/4/2002
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichloropropane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dichloropropane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloropropane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	7/30/2004
1,3-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	2/4/2002
1,3-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	2/4/2002
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,3-Dichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,3-Dichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	7/30/2004
1,4-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	2/4/2002
1,4-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	2/4/2002
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,4-Dichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,4-Dichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Dichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,4-Dioxane (1,4-Diethyleneoxide)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Dioxane (1,4-Diethyleneoxide) (without SIM)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ 195)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,4',5,6-Heptachlorobiphenyl (BZ 183)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ 184)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ 187)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',4,5'-Tetrachlorobiphenyl (BZ 49)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ 189)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,3',4,4',5-Hexachlorobiphenyl (BZ 156)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,3',4,4',5'-Hexachlorobiphenyl (BZ 157)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,3',4,4'-Pentachlorobiphenyl (BZ 105)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4',5,5'-Hexachlorobiphenyl (BZ 167)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,4,4',5-Pentachlorobiphenyl (BZ 114)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code:

ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,3',4,4',5-Pentachlorobiphenyl (BZ 118)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4',5'-Pentachlorobiphenyl (BZ 123)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,3,4,6-Tetrachlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4,4'-Trichlorobiphenyl (BZ 28)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,5-Trichlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4,5-Trichlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4,6-Trichlorophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trichlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4,6-Trichlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4-Dichlorobiphenyl (BZ 8)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,4-Dichlorophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dichlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dichlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4-Dimethylphenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dimethylphenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dimethylphenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4-Dinitrophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dinitrophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4-Dinitrotoluene (2,4-DNT)	EPA 625	Extractable Organics	NELAP	2/4/2002
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2,4-Dinitrotoluene (2,4-DNT)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4-Dinitrotoluene (2,4-DNT)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 625	Extractable Organics	NELAP	2/4/2002
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2,6-Dinitrotoluene (2,6-DNT)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Butanone (Methyl ethyl ketone, MEK)	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
2-Butanone (Methyl ethyl ketone, MEK)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Butanone (Methyl ethyl ketone, MEK)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Chloroethyl vinyl ether	EPA 624	Volatile Organics	NELAP	2/4/2002
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Chloronaphthalene	EPA 625	Extractable Organics	NELAP	2/4/2002
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chloronaphthalene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Chloronaphthalene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Chlorophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Chlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Hexanone	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Hexanone	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
2-Hexanone	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Hexanone	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Methyl-4,6-dinitrophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methyl-4,6-dinitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylnaphthalene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Methylnaphthalene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylphenol (o-Cresol)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Methylphenol (o-Cresol)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Naphthylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Nitrophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Nitrophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/30/2004
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,3',4,4',5,5'-Hexachlorobiphenyl (BZ 169)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3,3',4,4',5-Pentachlorobiphenyl (BZ 126)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3,3',4,4'-Tetrachlorobiphenyl (BZ 77)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3,3'-Dichlorobenzidine	EPA 625	Extractable Organics	NELAP	2/4/2002
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,3'-Dichlorobenzidine	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
3,3'-Dichlorobenzidine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,4,4',5-Tetrachlorobiphenyl (BZ 81)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
3-Nitroaniline	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/30/2004
4,4'-DDD	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDD	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDD	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
4,4'-DDE	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDE	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
4,4'-DDE	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
4,4'-DDT	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDT	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDT	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/30/2004
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Bromophenyl phenyl ether	EPA 625	Extractable Organics	NELAP	2/4/2002
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Bromophenyl phenyl ether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Bromophenyl phenyl ether	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chloro-3-methylphenol	EPA 625	Extractable Organics	NELAP	2/4/2002
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloro-3-methylphenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Chloro-3-methylphenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Chloroaniline	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chlorophenyl phenylether	EPA 625	Extractable Organics	NELAP	2/4/2002
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chlorophenyl phenylether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Chlorophenyl phenylether	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Methyl-2-pentanone (MIBK)	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
4-Methyl-2-pentanone (MIBK)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
4-Methyl-2-pentanone (MIBK)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Methylphenol (p-Cresol)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Methylphenol (p-Cresol)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009

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Issue Date: 7/1/2010

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Laboratory Scope of Accreditation

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State Laboratory ID: E87604 EPA Lab Code: ME00019 (207) 874-2400

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Nitroaniline	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Nitrophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Nitrophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/30/2004
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthene	EPA 625	Extractable Organics	NELAP	2/4/2002
Acenaphthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Acenaphthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Acenaphthylene	EPA 625	Extractable Organics	NELAP	2/4/2002
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthylene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Acenaphthylene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Acetone	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acetone	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
Acetone	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Acetone	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Acetonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acetophenone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acetophenone	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Acetophenone	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Acidity, as CaCO ₃	EPA 305.1	General Chemistry	NELAP	2/4/2002
Acidity, as CaCO ₃	SM 2310 B (4A)	General Chemistry	NELAP	4/17/2007
Acrolein (Propenal)	EPA 624	Volatile Organics	NELAP	4/26/2002
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acrylonitrile	EPA 624	Volatile Organics	NELAP	4/26/2002

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State Laboratory ID: E87604 EPA Lab Code: ME00019 (207) 874-2400

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Aldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aldrin	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aldrin	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Alkalinity as CaCO3	EPA 310.1	General Chemistry	NELAP	2/4/2002
Alkalinity as CaCO3	EPA 310.2	General Chemistry	NELAP	7/30/2004
Alkalinity as CaCO3	SM 2320 B	General Chemistry	NELAP	2/4/2002
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
alpha-BHC (alpha-Hexachlorocyclohexane)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
alpha-Chlordane	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
alpha-Chlordane	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aluminum	EPA 200.7	Metals	NELAP	2/4/2002
Aluminum	EPA 200.8	Metals	NELAP	2/4/2002
Aluminum	EPA 6010	Metals	NELAP	7/1/2003
Aluminum	EPA 6020	Metals	NELAP	7/1/2003
Aluminum	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Aluminum	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Amenable cyanide	EPA 335.1	General Chemistry	NELAP	2/4/2002
Amenable cyanide	EPA 335.4	General Chemistry	NELAP	2/4/2002
Amenable cyanide	EPA 9012	General Chemistry	NELAP	7/1/2003
Amenable cyanide	SM 4500-CN G	General Chemistry	NELAP	2/4/2002
Ammonia as N	EPA 350.1	General Chemistry	NELAP	2/4/2002
Ammonia as N	SM 4500-NH3 H	General Chemistry	NELAP	2/4/2002
Aniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
Anthracene	EPA 625	Extractable Organics	NELAP	2/4/2002
Anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Anthracene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Anthracene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Antimony	EPA 200.7	Metals	NELAP	2/4/2002

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Antimony	EPA 200.8	Metals	NELAP	2/4/2002
Antimony	EPA 6010	Metals	NELAP	7/1/2003
Antimony	EPA 6020	Metals	NELAP	7/1/2003
Antimony	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Antimony	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Aramite	EPA 8270	Extractable Organics	NELAP	7/1/2003
Aroclor-1016 (PCB-1016)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1016 (PCB-1016)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1016 (PCB-1016)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1221 (PCB-1221)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1221 (PCB-1221)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1221 (PCB-1221)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1232 (PCB-1232)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1232 (PCB-1232)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1232 (PCB-1232)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1242 (PCB-1242)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1242 (PCB-1242)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1242 (PCB-1242)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1248 (PCB-1248)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1248 (PCB-1248)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1248 (PCB-1248)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1254 (PCB-1254)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1254 (PCB-1254)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1254 (PCB-1254)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1260 (PCB-1260)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1260 (PCB-1260)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604 EPA Lab Code: ME00019 (207) 874-2400

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Aroclor-1260 (PCB-1260)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1262 (PCB-1262)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1268 (PCB-1268)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Arsenic	EPA 200.7	Metals	NELAP	2/4/2002
Arsenic	EPA 200.8	Metals	NELAP	2/4/2002
Arsenic	EPA 6010	Metals	NELAP	7/1/2003
Arsenic	EPA 6020	Metals	NELAP	2/4/2002
Arsenic	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Arsenic	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Atrazine	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Atrazine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Barium	EPA 200.7	Metals	NELAP	2/4/2002
Barium	EPA 200.8	Metals	NELAP	2/4/2002
Barium	EPA 6010	Metals	NELAP	7/1/2003
Barium	EPA 6020	Metals	NELAP	7/1/2003
Barium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Barium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Benzaldehyde	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzaldehyde	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzene	EPA 624	Volatile Organics	NELAP	2/4/2002
Benzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Benzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Benzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Benzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Benzidine	EPA 625	Extractable Organics	NELAP	2/4/2002
Benzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)anthracene	EPA 625	Extractable Organics	NELAP	2/4/2002
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)anthracene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(a)anthracene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(a)pyrene	EPA 625	Extractable Organics	NELAP	2/4/2002

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600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)pyrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(a)pyrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(b)fluoranthene	EPA 625	Extractable Organics	NELAP	2/4/2002
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(b)fluoranthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(b)fluoranthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(g,h,i)perylene	EPA 625	Extractable Organics	NELAP	2/4/2002
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(g,h,i)perylene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(g,h,i)perylene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(k)fluoranthene	EPA 625	Extractable Organics	NELAP	2/4/2002
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(k)fluoranthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(k)fluoranthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzoic acid	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Beryllium	EPA 200.7	Metals	NELAP	2/4/2002
Beryllium	EPA 200.8	Metals	NELAP	2/4/2002
Beryllium	EPA 6010	Metals	NELAP	7/1/2003
Beryllium	EPA 6020	Metals	NELAP	7/1/2003
Beryllium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Beryllium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
beta-BHC (beta-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
beta-BHC (beta-Hexachlorocyclohexane)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
beta-BHC (beta-Hexachlorocyclohexane)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Biochemical oxygen demand	EPA 405.1	General Chemistry	NELAP	2/4/2002
Biochemical oxygen demand	SM 5210 B	General Chemistry	NELAP	2/4/2002
Biphenyl	EPA 8270	Extractable Organics	NELAP	5/8/2009
Biphenyl	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Biphenyl	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Chloroethoxy)methane	EPA 625	Extractable Organics	NELAP	2/4/2002

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Laboratory Scope of Accreditation

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroethoxy)methane	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Chloroethoxy)methane	SOM01.2 Exhibit D	Extractable Organics	NELAP	5/8/2009
bis(2-Chloroethyl) ether	Semivolatiles/GC-MS			
bis(2-Chloroethyl) ether	EPA 625	Extractable Organics	NELAP	2/4/2002
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroethyl) ether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Chloroethyl) ether	SOM01.2 Exhibit D	Extractable Organics	NELAP	5/8/2009
bis(2-Chloroethyl) ether	Semivolatiles/GC-MS			
bis(2-Chloroisopropyl) ether	EPA 625	Extractable Organics	NELAP	2/4/2002
(2,2'-Oxybis(1-chloropropane))				
bis(2-Chloroisopropyl) ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
(2,2'-Oxybis(1-chloropropane))				
bis(2-Chloroisopropyl) ether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
(2,2'-Oxybis(1-chloropropane))				
bis(2-Chloroisopropyl) ether	SOM01.2 Exhibit D	Extractable Organics	NELAP	5/8/2009
(2,2'-Oxybis(1-chloropropane))	Semivolatiles/GC-MS			
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 625	Extractable Organics	NELAP	2/4/2002
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Ethylhexyl) phthalate (DEHP)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Ethylhexyl) phthalate (DEHP)	SOM01.2 Exhibit D	Extractable Organics	NELAP	5/8/2009
bis(2-Ethylhexyl) phthalate (DEHP)	Semivolatiles/GC-MS			
Boron	CA-627-02(EPA6020)/ICP-MS	Metals	NELAP	11/7/2006
Boron	CA-628-01(EPA 200.8)/ICP-MS	Metals	NELAP	11/7/2006
Boron	EPA 200.7	Metals	NELAP	2/4/2002
Boron	EPA 6010	Metals	NELAP	7/1/2003
Bromide	EPA 300.0	General Chemistry	NELAP	2/4/2002
Bromide	EPA 9056	General Chemistry	NELAP	7/1/2003
Bromobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromochloromethane	SOM01.2 Exhibit D	Volatile Organics	NELAP	5/8/2009
Bromochloromethane	Low-Medium Volatiles/GC-MS			
Bromochloromethane	SOM01.2 Exhibit D Trace	Volatile Organics	NELAP	5/8/2009
Bromochloromethane	Volatiles/GC-MS			
Bromodichloromethane	EPA 624	Volatile Organics	NELAP	2/4/2002
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromodichloromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Bromodichloromethane	SOM01.2 Exhibit D	Volatile Organics	NELAP	5/8/2009
Bromodichloromethane	Low-Medium Volatiles/GC-MS			

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Bromodichloromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromoform	EPA 624	Volatile Organics	NELAP	2/4/2002
Bromoform	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromoform	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Bromoform	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromoform	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Butyl benzyl phthalate	EPA 625	Extractable Organics	NELAP	2/4/2002
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Butyl benzyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Butyl benzyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Cadmium	EPA 200.7	Metals	NELAP	2/4/2002
Cadmium	EPA 200.8	Metals	NELAP	2/4/2002
Cadmium	EPA 6010	Metals	NELAP	7/1/2003
Cadmium	EPA 6020	Metals	NELAP	2/4/2002
Cadmium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Cadmium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Calcium	EPA 200.7	Metals	NELAP	2/4/2002
Calcium	EPA 6010	Metals	NELAP	7/1/2003
Calcium	EPA 6020	Metals	NELAP	11/7/2006
Calcium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Caprolactam	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Caprolactam	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Carbazole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Carbazole	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Carbazole	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	7/1/2003
Carbon disulfide	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Carbon disulfide	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbon disulfide	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbon tetrachloride	EPA 624	Volatile Organics	NELAP	2/4/2002

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Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Carbon tetrachloride	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Carbon tetrachloride	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbon tetrachloride	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbonaceous BOD (CBOD)	SM 5210 B	General Chemistry	NELAP	4/26/2002
Chemical oxygen demand	EPA 410.4	General Chemistry	NELAP	2/4/2002
Chemical oxygen demand	HACH 8000	General Chemistry	NELAP	4/26/2002
Chlordane (tech.)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chloride	EPA 300.0	General Chemistry	NELAP	2/4/2002
Chloride	EPA 325.2	General Chemistry	NELAP	2/4/2002
Chloride	EPA 9056	General Chemistry	NELAP	7/1/2003
Chloride	EPA 9251	General Chemistry	NELAP	7/1/2003
Chloride	SM 4500 Cl- E	General Chemistry	NELAP	2/4/2002
Chlorobenzene	EPA 624	Volatile Organics	NELAP	2/4/2002
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Chlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chloroethane	EPA 624	Volatile Organics	NELAP	2/4/2002
Chloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Chloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chloroform	EPA 624	Volatile Organics	NELAP	2/4/2002
Chloroform	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chloroform	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Chloroprene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chromium	EPA 200.7	Metals	NELAP	2/4/2002
Chromium	EPA 200.8	Metals	NELAP	4/26/2002

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Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Chromium	EPA 6010	Metals	NELAP	7/1/2003
Chromium	EPA 6020	Metals	NELAP	4/26/2002
Chromium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Chromium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Chromium VI	EPA 7196	General Chemistry	NELAP	7/1/2003
Chromium VI	SM 3500-Cr D (18th/19th Ed.)/UV-VIS	General Chemistry	NELAP	4/26/2002
Chrysene	EPA 625	Extractable Organics	NELAP	2/4/2002
Chrysene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Chrysene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Chrysene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
cis-1,2-Dichloroethylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
cis-1,2-Dichloroethylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
cis-1,2-Dichloroethylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
cis-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	2/4/2002
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
cis-1,3-Dichloropropene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
cis-1,3-Dichloropropene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
cis-1,3-Dichloropropene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Cobalt	EPA 200.7	Metals	NELAP	2/4/2002
Cobalt	EPA 200.8	Metals	NELAP	2/4/2002
Cobalt	EPA 6010	Metals	NELAP	7/1/2003
Cobalt	EPA 6020	Metals	NELAP	7/1/2003
Cobalt	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Cobalt	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Color	EPA 110.2	General Chemistry	NELAP	4/26/2002
Color	SM 2120 B	General Chemistry	NELAP	2/4/2002
Conductivity	EPA 120.1	General Chemistry	NELAP	2/4/2002
Conductivity	SM 2510 B	General Chemistry	NELAP	2/4/2002
Copper	EPA 200.7	Metals	NELAP	2/4/2002
Copper	EPA 200.8	Metals	NELAP	2/4/2002

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604 EPA Lab Code: ME00019 (207) 874-2400

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Copper	EPA 6010	Metals	NELAP	7/1/2003
Copper	EPA 6020	Metals	NELAP	2/4/2002
Copper	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Copper	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Cyanide	EPA 335.3	General Chemistry	NELAP	2/4/2002
Cyanide	EPA 335.4	General Chemistry	NELAP	2/4/2002
Cyclohexane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Cyclohexane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Cyclohexane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Decachlorobiphenyl (BZ 209)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
delta-BHC	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
delta-BHC	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
delta-BHC	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dibenz(a,h)anthracene	EPA 625	Extractable Organics	NELAP	2/4/2002
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibenz(a,h)anthracene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Dibenz(a,h)anthracene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibenzofuran	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Dibenzofuran	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Dibromochloromethane	EPA 624	Volatile Organics	NELAP	2/4/2002
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dibromochloromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Dibromochloromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dibromochloromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dibromomethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dichlorodifluoromethane	EPA 624	Volatile Organics	NELAP	2/4/2002

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

**Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074**

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dichlorodifluoromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Dichlorodifluoromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dichlorodifluoromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dieldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dieldrin	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Dieldrin	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	7/1/2003
Diesel range organics (DRO)	MA-EPH	Extractable Organics	NELAP	7/1/2003
Diesel range organics (DRO)	MEDEP 4.1.25	Extractable Organics	NELAP	7/1/2003
Diethyl ether	EPA 8260	Volatile Organics	NELAP	7/1/2003
Diethyl phthalate	EPA 625	Extractable Organics	NELAP	2/4/2002
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Diethyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Diethyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Di-isopropylether (DIPE)	EPA 8260	Volatile Organics	NELAP	5/8/2009
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dimethyl phthalate	EPA 625	Extractable Organics	NELAP	2/4/2002
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dimethyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Di-n-butyl phthalate	EPA 625	Extractable Organics	NELAP	2/4/2002
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-butyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Di-n-butyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Di-n-octyl phthalate	EPA 625	Extractable Organics	NELAP	2/4/2002
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-octyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Di-n-octyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan I	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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Laboratory Scope of Accreditation

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Endosulfan I	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan I	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endosulfan II	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan II	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan II	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endosulfan sulfate	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan sulfate	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan sulfate	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endrin aldehyde	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin aldehyde	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin aldehyde	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin ketone	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin ketone	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Escherichia coli	SM 9223 B /QUANTI-TRAY	Microbiology	NELAP	9/4/2007
Ethanol	EPA 8015	Volatile Organics	NELAP	7/1/2003
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Ethylbenzene	EPA 624	Volatile Organics	NELAP	2/4/2002
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ethylbenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Ethylbenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Ethylbenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Ethylene glycol	EPA 8015	Volatile Organics	NELAP	5/12/2005

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Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Ethyl-t-butylether (ETBE)	EPA 8260	Volatile Organics	NELAP	5/8/2009
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Fecal coliforms	SM 9222 D	Microbiology	NELAP	7/30/2004
Fluoranthene	EPA 625	Extractable Organics	NELAP	2/4/2002
Fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluoranthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Fluoranthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Fluorene	EPA 625	Extractable Organics	NELAP	2/4/2002
Fluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluorene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Fluorene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Fluoride	EPA 340.2	General Chemistry	NELAP	2/4/2002
Fluoride	SM 4500 F-C	General Chemistry	NELAP	2/4/2002
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
gamma-Chlordane	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
gamma-Chlordane	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	7/1/2003
Gasoline range organics (GRO)	MA-VPH	Extractable Organics	NELAP	7/1/2003
Gasoline range organics (GRO)	MEDEF 4.2.17	Extractable Organics	NELAP	7/1/2003
Hardness	SM 2340 B	Metals	NELAP	2/4/2002
Hardness (calc.)	EPA 200.7	Metals	NELAP	4/26/2002
Heptachlor	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Heptachlor	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Heptachlor	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Heptachlor epoxide	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Heptachlor epoxide	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004

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EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Heptachlor epoxide	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Hexachlorobenzene	EPA 625	Extractable Organics	NELAP	2/4/2002
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorobenzene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachlorobenzene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachlorobutadiene	EPA 625	Extractable Organics	NELAP	2/4/2002
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorobutadiene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachlorobutadiene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachlorocyclopentadiene	EPA 625	Extractable Organics	NELAP	2/4/2002
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorocyclopentadiene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachlorocyclopentadiene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachloroethane	EPA 625	Extractable Organics	NELAP	2/4/2002
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachloroethane	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachloroethane	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Ignitability	EPA 1010	General Chemistry	NELAP	7/1/2003
Indeno(1,2,3-cd)pyrene	EPA 625	Extractable Organics	NELAP	2/4/2002
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Indeno(1,2,3-cd)pyrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Indeno(1,2,3-cd)pyrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Iron	EPA 200.7	Metals	NELAP	2/4/2002
Iron	EPA 6010	Metals	NELAP	7/1/2003
Iron	EPA 6020	Metals	NELAP	11/7/2006
Iron	1LM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Iron	SM 3500-Fe D (18th/19th Ed.)/UV-VIS	General Chemistry	NELAP	4/26/2002
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8015	Volatile Organics	NELAP	7/1/2003
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Isodrin	EPA 8270	Extractable Organics	NELAP	7/1/2003

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Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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EPA Lab Code: ME00019

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E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Isophorone	EPA 625	Extractable Organics	NELAP	2/4/2002
Isophorone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Isophorone	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Isophorone	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Isopropyl alcohol (2-Propanol)	EPA 8015	Volatile Organics	NELAP	7/1/2003
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Isopropylbenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Isopropylbenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Isopropylbenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Isosafrole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Kjeldahl nitrogen - total	EPA 351.2	General Chemistry	NELAP	2/4/2002
Lead	EPA 200.7	Metals	NELAP	2/4/2002
Lead	EPA 200.8	Metals	NELAP	2/4/2002
Lead	EPA 6010	Metals	NELAP	7/1/2003
Lead	EPA 6020	Metals	NELAP	2/4/2002
Lead	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Lead	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
m+p-Xylenes	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
m+p-Xylenes	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Magnesium	EPA 200.7	Metals	NELAP	2/4/2002
Magnesium	EPA 6010	Metals	NELAP	7/1/2003
Magnesium	EPA 6020	Metals	NELAP	11/7/2006
Magnesium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Magnesium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Manganese	EPA 200.7	Metals	NELAP	2/4/2002
Manganese	EPA 200.8	Metals	NELAP	2/4/2002
Manganese	EPA 6010	Metals	NELAP	7/1/2003
Manganese	EPA 6020	Metals	NELAP	7/1/2003
Manganese	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Mercury	EPA 1631	Metals	NELAP	4/26/2002
Mercury	EPA 245.1	Metals	NELAP	2/4/2002
Mercury	EPA 7470	Metals	NELAP	7/1/2003
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methanol	EPA 8015	Volatile Organics	NELAP	7/1/2003
Methapyrilene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methoxychlor	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Methoxychlor	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Methyl acetate	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methyl acetate	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl acetate	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl bromide (Bromomethane)	EPA 624	Volatile Organics	NELAP	2/4/2002
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl bromide (Bromomethane)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl bromide (Bromomethane)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl chloride (Chloromethane)	EPA 624	Volatile Organics	NELAP	2/4/2002
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl chloride (Chloromethane)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methyl chloride (Chloromethane)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl chloride (Chloromethane)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl tert-butyl ether (MTBE)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methylcyclohexane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methylcyclohexane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methylcyclohexane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009

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State Laboratory ID: E87604 EPA Lab Code: ME00019 (207) 874-2400

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Methylene chloride	EPA 624	Volatile Organics	NELAP	2/4/2002
Methylene chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methylene chloride	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methylene chloride	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methylene chloride	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Molybdenum	EPA 200.7	Metals	NELAP	2/4/2002
Molybdenum	EPA 200.8	Metals	NELAP	2/4/2002
Molybdenum	EPA 6010	Metals	NELAP	7/1/2003
Molybdenum	EPA 6020	Metals	NELAP	4/26/2002
Naphthalene	EPA 625	Extractable Organics	NELAP	2/4/2002
Naphthalene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Naphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Naphthalene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Naphthalene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Nickel	EPA 200.7	Metals	NELAP	2/4/2002
Nickel	EPA 200.8	Metals	NELAP	2/4/2002
Nickel	EPA 6010	Metals	NELAP	7/1/2003
Nickel	EPA 6020	Metals	NELAP	2/4/2002
Nickel	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Nickel	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Nitrate	EPA 9056	General Chemistry	NELAP	7/1/2003
Nitrate as N	EPA 300.0	General Chemistry	NELAP	2/4/2002
Nitrate as N	EPA 353.2	General Chemistry	NELAP	2/4/2002
Nitrate as N	SM 4500-NO3 F	General Chemistry	NELAP	2/4/2002
Nitrate-nitrite	EPA 300.0	General Chemistry	NELAP	2/4/2002
Nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	2/4/2002
Nitrate-nitrite	SM 4500-NO3 F	General Chemistry	NELAP	2/4/2002
Nitrite	EPA 9056	General Chemistry	NELAP	7/1/2003
Nitrite as N	EPA 300.0	General Chemistry	NELAP	2/4/2002
Nitrite as N	EPA 353.2	General Chemistry	NELAP	2/4/2002
Nitrite as N	SM 4500-NO3 F	General Chemistry	NELAP	2/4/2002
Nitrobenzene	EPA 625	Extractable Organics	NELAP	2/4/2002
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003

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Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	7/30/2004
Nitrobenzene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Nitrobenzene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Nitroglycerin	EPA 8332	Extractable Organics	NELAP	5/12/2005
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodimethylamine	EPA 625	Extractable Organics	NELAP	2/4/2002
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodi-n-propylamine	EPA 625	Extractable Organics	NELAP	2/4/2002
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodi-n-propylamine	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
n-Nitrosodi-n-propylamine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
n-Nitrosodiphenylamine	EPA 625	Extractable Organics	NELAP	2/4/2002
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiphenylamine	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
n-Nitrosodiphenylamine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Propanol	EPA 8015	Volatile Organics	NELAP	7/1/2003
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
o,o,o-Triethyl phosphorothioate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	7/30/2004
Oil & Grease	EPA 1664A	General Chemistry	NELAP	2/4/2002
Oil & Grease	EPA 9070	General Chemistry	NELAP	7/1/2003
Organic nitrogen	TKN minus AMMONIA	General Chemistry	NELAP	2/4/2002
Orthophosphate as P	EPA 300.0	General Chemistry	NELAP	2/4/2002
Orthophosphate as P	EPA 365.1	General Chemistry	NELAP	11/7/2006
Orthophosphate as P	EPA 365.2	General Chemistry	NELAP	2/4/2002
Orthophosphate as P	EPA 9056	General Chemistry	NELAP	7/1/2003
Orthophosphate as P	SM 4500-P E	General Chemistry	NELAP	2/4/2002
o-Toluidine	EPA 8270	Extractable Organics	NELAP	7/1/2003

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E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
o-Xylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
o-Xylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
p-Dioxane	CA-204.07/GC-MS	Extractable Organics	NELAP	11/7/2006
Pentachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pentachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Pentachloronitrobenzene (Quintozene)	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pentachlorophenol	EPA 625	Extractable Organics	NELAP	2/4/2002
Pentachlorophenol	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pentachlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Pentachlorophenol (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Perchlorate	EPA 314.0	General Chemistry	NELAP	7/30/2004
pH	EPA 150.1	General Chemistry	NELAP	2/4/2002
pH	EPA 9040	General Chemistry	NELAP	7/1/2003
pH	SM 4500-H ⁺ -B	General Chemistry	NELAP	4/26/2002
Phenacetin	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenanthrene	EPA 625	Extractable Organics	NELAP	2/4/2002
Phenanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenanthrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Phenanthrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Phenol	EPA 625	Extractable Organics	NELAP	2/4/2002
Phenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Phenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Phosphorus, total	EPA 365.4	General Chemistry	NELAP	2/4/2002
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Potassium	EPA 200.7	Metals	NELAP	2/4/2002
Potassium	EPA 6010	Metals	NELAP	7/1/2003
Potassium	EPA 6020	Metals	NELAP	11/7/2006
Potassium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Pronamide (Kerb)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	7/1/2003

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E87604

Katahdin Analytical Services, Inc.

600 Technology Way

Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Pyrene	EPA 625	Extractable Organics	NELAP	2/4/2002
Pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pyrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Pyrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Pyridine	EPA 8270	Extractable Organics	NELAP	7/1/2003
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	7/30/2004
Residue-filterable (TDS)	EPA 160.1	General Chemistry	NELAP	2/4/2002
Residue-filterable (TDS)	SM 2540 C	General Chemistry	NELAP	2/4/2002
Residue-nonfilterable (TSS)	EPA 160.2	General Chemistry	NELAP	2/4/2002
Residue-nonfilterable (TSS)	SM 2540 D	General Chemistry	NELAP	2/4/2002
Residue-settleable	EPA 160.5	General Chemistry	NELAP	2/4/2002
Residue-settleable	SM 2540 F	General Chemistry	NELAP	2/4/2002
Residue-total	EPA 160.3	General Chemistry	NELAP	2/4/2002
Residue-total	SM 2540 B	General Chemistry	NELAP	2/4/2002
Residue-volatile	EPA 160.4	General Chemistry	NELAP	2/4/2002
Saffrole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Salinity	SM 2520 B	General Chemistry	NELAP	2/4/2002
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Selenium	EPA 200.7	Metals	NELAP	2/4/2002
Selenium	EPA 200.8	Metals	NELAP	2/4/2002
Selenium	EPA 6010	Metals	NELAP	7/1/2003
Selenium	EPA 6020	Metals	NELAP	2/4/2002
Selenium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Selenium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Silicon	EPA 200.7	Metals	NELAP	2/4/2002
Silver	EPA 200.7	Metals	NELAP	2/4/2002
Silver	EPA 200.8	Metals	NELAP	2/4/2002
Silver	EPA 6010	Metals	NELAP	7/1/2003
Silver	EPA 6020	Metals	NELAP	7/1/2003
Silver	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Silver	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Sodium	EPA 200.7	Metals	NELAP	2/4/2002
Sodium	EPA 6010	Metals	NELAP	7/1/2003
Sodium	EPA 6020	Metals	NELAP	11/7/2006

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Sodium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Strontium	CA-627-02(EPA6020)/ICP-MS	Metals	NELAP	11/7/2006
Strontium	EPA 6010	Metals	NELAP	7/1/2003
Styrene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Styrene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Styrene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Styrene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Sulfate	ASTM D516-02	General Chemistry	NELAP	4/17/2007
Sulfate	ASTM D516-90	General Chemistry	NELAP	4/17/2007
Sulfate	EPA 300.0	General Chemistry	NELAP	2/4/2002
Sulfate	EPA 375.4	General Chemistry	NELAP	2/4/2002
Sulfate	EPA 9038	General Chemistry	NELAP	7/1/2003
Sulfate	EPA 9056	General Chemistry	NELAP	7/1/2003
Sulfide	EPA 376.1	General Chemistry	NELAP	2/4/2002
Sulfide	SM 4500-S E (18th Ed.)/ITTR	General Chemistry	NELAP	4/17/2007
Sulfite-SO3	EPA 377.1	General Chemistry	NELAP	2/4/2002
Sulfite-SO3	SM 4500-SO3 B	General Chemistry	NELAP	4/17/2007
Sulfotep	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Surfactants - MBAS	SM 5540 C	General Chemistry	NELAP	5/12/2005
T-amylmethylether (TAME)	EPA 8260	Volatile Organics	NELAP	5/8/2009
tert-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	5/8/2009
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Tetrachloroethylene (Perchloroethylene)	EPA 624	Volatile Organics	NELAP	2/4/2002
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Tetrachloroethylene (Perchloroethylene)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Tetrachloroethylene (Perchloroethylene)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Tetrachloroethylene (Perchloroethylene)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Tetrahydrofuran (THF)	CA-202.08/GC-MS	Volatile Organics	NELAP	11/7/2006
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	7/30/2004
Thallium	EPA 200.7	Metals	NELAP	2/4/2002
Thallium	EPA 200.8	Metals	NELAP	2/4/2002
Thallium	EPA 6010	Metals	NELAP	7/1/2003

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600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Thallium	EPA 6020	Metals	NELAP	7/1/2003
Thallium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Thallium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Thionazin (Zinophos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Thorium	EPA 200.8	Metals	NELAP	2/4/2002
Tin	CA-628-01(EPA 200.8)/ICP-MS	Metals	NELAP	11/7/2006
Tin	EPA 200.7	Metals	NELAP	2/4/2002
Tin	EPA 6010	Metals	NELAP	7/30/2004
Titanium	EPA 200.7	Metals	NELAP	2/4/2002
Titanium	EPA 6010	Metals	NELAP	7/30/2004
Toluene	EPA 624	Volatile Organics	NELAP	2/4/2002
Toluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Toluene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Toluene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Toluene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Total coliforms	SM 9222 B	Microbiology	NELAP	7/30/2004
Total cyanide	EPA 9012	General Chemistry	NELAP	7/1/2003
Total hardness as CaCO3	CA-628-01(EPA 200.8)/ICP-MS	Metals	NELAP	11/7/2006
Total hardness as CaCO3	EPA 130.2	General Chemistry	NELAP	9/4/2007
Total hardness as CaCO3	SM 2340 C	General Chemistry	NELAP	9/4/2007
Total nitrate-nitrite	EPA 9056	General Chemistry	NELAP	7/1/2003
Total organic carbon	EPA 415.1	General Chemistry	NELAP	2/4/2002
Total organic carbon	EPA 9060	General Chemistry	NELAP	7/1/2003
Total organic carbon	SM 5310 B	General Chemistry	NELAP	4/17/2007
Total Petroleum Hydrocarbons (TPH)	EPA 1664A	General Chemistry	NELAP	2/4/2002
Total Petroleum Hydrocarbons (TPH)	FL-PRO	Extractable Organics	NELAP	7/1/2003
Total Petroleum Hydrocarbons (TPH)	TX1005	Extractable Organics	NELAP	9/4/2007
Total phenolics	EPA 420.1	General Chemistry	NELAP	2/4/2002
Total phenolics	EPA 9065	General Chemistry	NELAP	7/1/2003
Total residual chlorine	SM 4500-Cl G	General Chemistry	NELAP	9/4/2007
Total, fixed, and volatile residue	SM 2540 G	General Chemistry	NELAP	4/26/2002
Toxaphene (Chlorinated camphene)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

**Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074**

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Toxaphene (Chlorinated camphene)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Toxaphene (Chlorinated camphene)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
trans-1,2-Dichloroethylene	EPA 624	Volatile Organics	NELAP	2/4/2002
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,2-Dichloroethylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
trans-1,2-Dichloroethylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,2-Dichloroethylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,3-Dichloropropylene	EPA 624	Volatile Organics	NELAP	2/4/2002
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,3-Dichloropropylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
trans-1,3-Dichloropropylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,3-Dichloropropylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichloroethene (Trichloroethylene)	EPA 624	Volatile Organics	NELAP	2/4/2002
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichloroethene (Trichloroethylene)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Trichloroethene (Trichloroethylene)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Trichloroethene (Trichloroethylene)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	2/4/2002
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichlorofluoromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Trichlorofluoromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Trichlorofluoromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Turbidity	EPA 180.1	General Chemistry	NELAP	2/4/2002
Turbidity	SM 2130 B	General Chemistry	NELAP	2/4/2002
Uranium	EPA 200.8	Metals	NELAP	2/4/2002
Vanadium	EPA 200.7	Metals	NELAP	2/4/2002
Vanadium	EPA 200.8	Metals	NELAP	2/4/2002
Vanadium	EPA 6010	Metals	NELAP	7/1/2003

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code:

ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Vanadium	EPA 6020	Metals	NELAP	2/4/2002
Vanadium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Vanadium	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Vinyl chloride	EPA 624	Volatile Organics	NELAP	2/4/2002
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Vinyl chloride	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Vinyl chloride	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Vinyl chloride	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Xylene (total)	EPA 624	Volatile Organics	NELAP	4/26/2002
Xylene (total)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Xylene (total)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Zinc	EPA 200.7	Metals	NELAP	2/4/2002
Zinc	EPA 200.8	Metals	NELAP	2/4/2002
Zinc	EPA 6010	Metals	NELAP	7/1/2003
Zinc	EPA 6020	Metals	NELAP	2/4/2002
Zinc	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Zinc	ILM05.3-Exhibit D/ICP-MS	Metals	NELAP	11/7/2006

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Laboratory Scope of Accreditation

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State Laboratory ID: E87604

EPA Lab Code:

ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,1,1-Trichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,1-Trichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,1-Trichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,1,2,2-Tetrachloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2,2-Tetrachloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2,2-Tetrachloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloro-1,2,2-trifluoroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2-Trichloro-1,2,2-trifluoroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloro-1,2,2-trifluoroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,1,2-Trichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1,2-Trichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1,2-Trichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,1-Dichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1-Dichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,1-Dichloroethylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,1-Dichloroethylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloroethylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002

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Issue Date: 7/1/2010

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Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604 EPA Lab Code: ME00019 (207) 874-2400

E87604
Katabdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2,3-Trichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,3-Trichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,2,4-Trichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2,4-Trichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,4-Trichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2-Dibromo-3-chloropropane (DBCP)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dibromo-3-chloropropane (DBCP)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dibromo-3-chloropropane (DBCP) (with SIM)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2-Dibromoethane (EDB, Ethylene dibromide)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dibromoethane (EDB, Ethylene dibromide)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dibromoethane (EDB, Ethylene dibromide) (with SIM)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,2-Dichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2-Dichloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dichloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Laboratory Scope of Accreditation

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

**Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074**

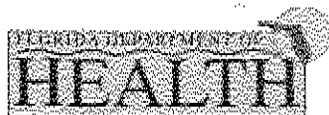
Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,2-Dichloropropane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,2-Dichloropropane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Dichloropropane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	7/30/2004
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,3-Dichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,3-Dichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,3-Dichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	4/26/2002
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	7/30/2004
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,4-Dichlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
1,4-Dichlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Dichlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	4/26/2002
1,4-Dioxane (1,4-Diethyleneoxide)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Dioxane (1,4-Diethyleneoxide) (without SIM)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	2/4/2002
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ 195)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Laboratory Scope of Accreditation

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

**Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074**

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,4',5',6'-Heptachlorobiphenyl (BZ 183)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ 184)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',3,4',5,5',6'-Heptachlorobiphenyl (BZ 187)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',4,5'-Tetrachlorobiphenyl (BZ 49)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	2/4/2002
2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ 189)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,3',4,4',5-Hexachlorobiphenyl (BZ 156)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,3',4,4',5'-Hexachlorobiphenyl (BZ 157)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,3',4,4'-Pentachlorobiphenyl (BZ 105)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4',5,5'-Hexachlorobiphenyl (BZ 167)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3,4,4',5-Pentachlorobiphenyl (BZ 114)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4',5-Pentachlorobiphenyl (BZ 118)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4',5'-Pentachlorobiphenyl (BZ 123)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,3,4,6-Tetrachlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4,4'-Trichlorobiphenyl (BZ 28)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,4,5-Trichlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4,5-Trichlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,4,6-Trichlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4,6-Trichlorophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Laboratory Scope of Accreditation

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State Laboratory ID: E87604 EPA Lab Code: ME00019 (207) 874-2400

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
2,4'-Dichlorobiphenyl (BZ 8)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,4-Dichlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dichlorophenol	SOM01.2 Exhibit D	Extractable Organics	NELAP	5/8/2009
2,4-Dichlorophenol	Semivolatiles/GC-MS			
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,4-Dimethylphenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dimethylphenol	SOM01.2 Exhibit D	Extractable Organics	NELAP	5/8/2009
2,4-Dimethylphenol	Semivolatiles/GC-MS			
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,4-Dinitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dinitrophenol	SOM01.2 Exhibit D	Extractable Organics	NELAP	5/8/2009
2,4-Dinitrophenol	Semivolatiles/GC-MS			
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2,4-Dinitrotoluene (2,4-DNT)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2,4-Dinitrotoluene (2,4-DNT)	SOM01.2 Exhibit D	Extractable Organics	NELAP	5/8/2009
2,4-Dinitrotoluene (2,4-DNT)	Semivolatiles/GC-MS			
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	2/4/2002
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2,6-Dinitrotoluene (2,6-DNT)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/30/2004
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	2/4/2002
2-Butanone (Methyl ethyl ketone, MEK)	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
2-Butanone (Methyl ethyl ketone, MEK)	SOM01.2 Exhibit D	Volatile Organics	NELAP	5/8/2009
2-Butanone (Methyl ethyl ketone, MEK)	Low-Medium			
2-Butanone (Methyl ethyl ketone, MEK)	Volatiles/GC-MS			
2-Butanone (Methyl ethyl ketone, MEK)	SOM01.2 Exhibit D Trace	Volatile Organics	NELAP	5/8/2009
2-Butanone (Methyl ethyl ketone, MEK)	Volatiles/GC-MS			
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	2/4/2002
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Chloronaphthalene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Chloronaphthalene	SOM01.2 Exhibit D	Extractable Organics	NELAP	5/8/2009
2-Chloronaphthalene	Semivolatiles/GC-MS			
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Chlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Chlorophenol	SOM01.2 Exhibit D	Extractable Organics	NELAP	5/8/2009
2-Chlorophenol	Semivolatiles/GC-MS			

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	2/4/2002
2-Hexanone	EPA 8260	Volatile Organics	NELAP	2/4/2002
2-Hexanone	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
2-Hexanone	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Hexanone	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Methyl-4,6-dinitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Methylnaphthalene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Methylnaphthalene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Methylphenol (o-Cresol)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Methylphenol (o-Cresol)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Naphthylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Nitroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
2-Nitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
2-Nitrophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/30/2004
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	2/4/2002
3,3',4,4',5,5'-Hexachlorobiphenyl (BZ 169)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3,3',4,4',5-Pentachlorobiphenyl (BZ 126)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3,3',4,4'-Tetrachlorobiphenyl (BZ 77)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
3,3'-Dichlorobenzidine	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
3,3'-Dichlorobenzidine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
3,4,4',5-Tetrachlorobiphenyl (BZ 81)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	4/26/2002
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	2/4/2002
3-Nitroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004

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Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604 EPA Lab Code: ME00019 (207) 874-2400

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
3-Nitroaniline	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/30/2004
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
4,4'-DDD	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDD	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
4,4'-DDE	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDE	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
4,4'-DDT	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDT	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/30/2004
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Bromophenyl phenyl ether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Bromophenyl phenyl ether	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Chloro-3-methylphenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Chloro-3-methylphenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Chloroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Chloroaniline	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Chlorophenyl phenylether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Chlorophenyl phenylether	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	2/4/2002
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	2/4/2002
4-Methyl-2-pentanone (MIBK)	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
4-Methyl-2-pentanone (MIBK)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
4-Methyl-2-pentanone (MIBK)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009

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Expiration Date: 6/30/2011



Laboratory Scope of Accreditation

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

**Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074**

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Methylphenol (p-Cresol)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Methylphenol (p-Cresol)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Nitroaniline	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Nitroaniline	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
4-Nitrophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
4-Nitrophenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/30/2004
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	2/4/2002
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
Acenaphthene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Acenaphthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Acenaphthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Acenaphthylene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Acenaphthylene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Acetone	EPA 8260	Volatile Organics	NELAP	2/4/2002
Acetone	OLM04.3-Exhibit D	Volatile Organics	NELAP	5/12/2005
Acetone	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Acetone	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Acetonitrile	EPA 8260	Volatile Organics	NELAP	2/4/2002
Acetophenone	EPA 8270	Extractable Organics	NELAP	2/4/2002
Acetophenone	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	2/4/2002
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aldrin	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aldrin	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	4/26/2002

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Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
alpha-BHC (alpha-Hexachlorocyclohexane)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
alpha-BHC (alpha-Hexachlorocyclohexane)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/26/2002
alpha-Chlordane	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
alpha-Chlordane	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aluminum	EPA 6010	Metals	NELAP	2/4/2002
Aluminum	EPA 6020	Metals	NELAP	2/4/2002
Aluminum	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Amenable cyanide	EPA 9012	General Chemistry	NELAP	4/26/2002
Aniline	EPA 8270	Extractable Organics	NELAP	2/4/2002
Anthracene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Anthracene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Anthracene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Antimony	EPA 6010	Metals	NELAP	2/4/2002
Antimony	EPA 6020	Metals	NELAP	2/4/2002
Antimony	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Aramite	EPA 8270	Extractable Organics	NELAP	2/4/2002
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1016 (PCB-1016)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1016 (PCB-1016)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1221 (PCB-1221)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1221 (PCB-1221)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1232 (PCB-1232)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1232 (PCB-1232)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1242 (PCB-1242)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1242 (PCB-1242)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1248 (PCB-1248)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004

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Laboratory Scope of Accreditation

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State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604

**Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074**

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Aroclor-1248 (PCB-1248)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1254 (PCB-1254)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1254 (PCB-1254)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Aroclor-1260 (PCB-1260)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1260 (PCB-1260)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1262 (PCB-1262)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Aroclor-1268 (PCB-1268)	SOM01.2 Exhibit D Aroclors/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Arsenic	EPA 6010	Metals	NELAP	2/4/2002
Arsenic	EPA 6020	Metals	NELAP	2/4/2002
Arsenic	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Atrazine	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Atrazine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Barium	EPA 6010	Metals	NELAP	2/4/2002
Barium	EPA 6020	Metals	NELAP	2/4/2002
Barium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Benzaldehyde	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzaldehyde	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Benzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Benzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Benzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Benzidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzo(a)anthracene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(a)anthracene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzo(a)pyrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(a)pyrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009

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Expiration Date: 6/30/2011

Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code:

ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzo(b)fluoranthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(b)fluoranthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzo(g,h,i)perylene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(g,h,i)perylene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzo(k)fluoranthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Benzo(k)fluoranthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Benzoic acid	EPA 8270	Extractable Organics	NELAP	2/4/2002
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	2/4/2002
Beryllium	EPA 6010	Metals	NELAP	2/4/2002
Beryllium	EPA 6020	Metals	NELAP	2/4/2002
Beryllium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
beta-BHC (beta-Hexachlorocyclohexane)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
beta-BHC (beta-Hexachlorocyclohexane)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Biphenyl	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Biphenyl	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	2/4/2002
bis(2-Chloroethoxy)methane	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Chloroethoxy)methane	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	2/4/2002
bis(2-Chloroethyl) ether	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Chloroethyl) ether	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	2/4/2002
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	2/4/2002
bis(2-Ethylhexyl) phthalate (DEHP)	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
bis(2-Ethylhexyl) phthalate (DEHP)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009

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Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code: ME00019

(207) 874-2400

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Boron	CA-627-02(EPA6020)/ICP-MS	Metals	NELAP	11/7/2006
Boron	EPA 6010	Metals	NELAP	4/26/2002
Bromide	EPA 9056	General Chemistry	NELAP	2/4/2002
Bromobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Bromochloromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromochloromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Bromodichloromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Bromodichloromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromodichloromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromoform	EPA 8260	Volatile Organics	NELAP	2/4/2002
Bromoform	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Bromoform	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Bromoform	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Butyl benzyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Butyl benzyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Cadmium	EPA 6010	Metals	NELAP	2/4/2002
Cadmium	EPA 6020	Metals	NELAP	2/4/2002
Cadmium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Calcium	EPA 6010	Metals	NELAP	2/4/2002
Calcium	EPA 6020	General Chemistry	NELAP	11/7/2006
Calcium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Caprolactam	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Caprolactam	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Carbazole	EPA 8270	Extractable Organics	NELAP	2/4/2002
Carbazole	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Carbazole	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009

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State Laboratory ID: E87604 EPA Lab Code: ME00019 (207) 874-2400

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	2/4/2002
Carbon disulfide	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Carbon disulfide	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbon disulfide	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	2/4/2002
Carbon tetrachloride	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Carbon tetrachloride	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Carbon tetrachloride	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Chloride	EPA 9056	General Chemistry	NELAP	2/4/2002
Chloride	EPA 9251	General Chemistry	NELAP	2/4/2002
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Chlorobenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Chlorobenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chlorobenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Chloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Chloroethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Chloroethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chloroethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chloroform	EPA 8260	Volatile Organics	NELAP	2/4/2002
Chloroform	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Chloroform	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chloroform	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Chloroprene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Chromium	EPA 6010	Metals	NELAP	2/4/2002
Chromium	EPA 6020	Metals	NELAP	2/4/2002
Chromium	HM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006

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Laboratory Scope of Accreditation

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State Laboratory ID: E87604 EPA Lab Code: ME00019 (207) 874-2400

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Chromium VI	EPA 7196	General Chemistry	NELAP	4/26/2002
Chrysene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Chrysene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Chrysene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/4/2002
cis-1,2-Dichloroethylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
cis-1,2-Dichloroethylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
cis-1,2-Dichloroethylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	2/4/2002
cis-1,3-Dichloropropene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
cis-1,3-Dichloropropene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
cis-1,3-Dichloropropene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Cobalt	EPA 6010	Metals	NELAP	2/4/2002
Cobalt	EPA 6020	Metals	NELAP	2/4/2002
Cobalt	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Copper	EPA 6010	Metals	NELAP	2/4/2002
Copper	EPA 6020	Metals	NELAP	2/4/2002
Copper	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Cyclohexane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Cyclohexane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Cyclohexane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
Decachlorobiphenyl (BZ, 209)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
delta-BHC	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
delta-BHC	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Dibenz(a,h)anthracene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Dibenz(a,h)anthracene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004

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Katabdin Analytical Services, Inc.
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Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Dibenz(a,h)anthracene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	2/4/2002
Dibenzofuran	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Dibromochloromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Dibromochloromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dibromochloromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dibromomethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Dichlorodifluoromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Dichlorodifluoromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dichlorodifluoromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Dieldrin	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Dieldrin	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	2/4/2002
Diesel range organics (DRO)	MA-EPH	Extractable Organics	NELAP	2/4/2002
Diesel range organics (DRO)	MEDEP 4.1.25	Extractable Organics	NELAP	2/4/2002
Diethyl ether	EPA 8260	Volatile Organics	NELAP	2/4/2002
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Diethyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Diethyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Di-isopropylether (DIPE)	EPA 8260	Extractable Organics	NELAP	5/8/2009
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	4/26/2002
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Dimethyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Di-n-butyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Di-n-butyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009

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(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Di-n-octyl phthalate	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Di-n-octyl phthalate	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endosulfan I	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan I	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endosulfan II	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan II	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endosulfan sulfate	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan sulfate	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endrin	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endrin aldehyde	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin aldehyde	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Endrin ketone	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin ketone	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	2/4/2002
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Ethylbenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Ethylbenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Ethylbenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Ethyl-t-butylether (ETBE)	EPA 8260	Extractable Organics	NELAP	5/8/2009
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	4/26/2002
Fluoranthene	EPA 8270	Extractable Organics	NELAP	2/4/2002

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Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Fluoranthene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Fluoranthene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Fluorene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Fluorene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Fluorene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/26/2002
gamma-Chlordane	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
gamma-Chlordane	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	2/4/2002
Gasoline range organics (GRO)	MA-VPH	Extractable Organics	NELAP	2/4/2002
Gasoline range organics (GRO)	MEDEP 4.2.17	Extractable Organics	NELAP	2/4/2002
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Heptachlor	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Heptachlor	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Heptachlor epoxide	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Heptachlor epoxide	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Hexachlorobenzene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachlorobenzene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Hexachlorobutadiene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachlorobutadiene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Hexachlorocyclopentadiene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachlorocyclopentadiene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	2/4/2002

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Laboratory Scope of Accreditation

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State Laboratory ID: E87604 EPA Lab Code: ME00019 (207) 874-2400

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Hexachloroethane	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Hexachloroethane	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	4/26/2002
Ignitability	EPA 1010	General Chemistry	NELAP	2/4/2002
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Indeno(1,2,3-cd)pyrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Indeno(1,2,3-cd)pyrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Iron	EPA 6010	Metals	NELAP	2/4/2002
Iron	EPA 6020	General Chemistry	NELAP	11/7/2006
Iron	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Isodrin	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Isophorone	EPA 8270	Extractable Organics	NELAP	2/4/2002
Isophorone	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Isophorone	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Isopropylbenzene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Isopropylbenzene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Isopropylbenzene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Isosafrole	EPA 8270	Extractable Organics	NELAP	2/4/2002
Lead	EPA 6010	Metals	NELAP	2/4/2002
Lead	EPA 6020	Metals	NELAP	2/4/2002
Lead	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
m+p-Xylenes	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
m+p-Xylenes	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Magnesium	EPA 6010	Metals	NELAP	2/4/2002
Magnesium	EPA 6020	General Chemistry	NELAP	11/7/2006
Magnesium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Manganese	EPA 6010	Metals	NELAP	2/4/2002

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Manganese	EPA 6020	Metals	NELAP	2/4/2002
Manganese	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
Mercury	EPA 7471	Metals	NELAP	2/4/2002
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	2/4/2002
Methapyrilene	EPA 8270	Extractable Organics	NELAP	4/26/2002
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Methoxychlor	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Methoxychlor	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Methyl acetate	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methyl acetate	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl acetate	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Methyl bromide (Bromomethane)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methyl bromide (Bromomethane)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl bromide (Bromomethane)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Methyl chloride (Chloromethane)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methyl chloride (Chloromethane)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl chloride (Chloromethane)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	2/4/2002
Methyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	2/4/2002
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	4/26/2002
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Methyl tert-butyl ether (MTBE)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methyl tert-butyl ether (MTBE)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methyl tert-butyl ether (MTBE)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methylcyclohexane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Methylcyclohexane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methylcyclohexane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methylene chloride	EPA 8260	Volatile Organics	NELAP	2/4/2002
Methylene chloride	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Methylene chloride	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Methylene chloride	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Molybdenum	EPA 6010	Metals	NELAP	4/26/2002
Molybdenum	EPA 6020	General Chemistry	NELAP	11/7/2006
Naphthalene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Naphthalene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Naphthalene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Naphthalene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Nickel	EPA 6010	Metals	NELAP	2/4/2002
Nickel	EPA 6020	Metals	NELAP	2/4/2002
Nickel	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Nitrate	EPA 9056	General Chemistry	NELAP	2/4/2002
Nitrite	EPA 9056	General Chemistry	NELAP	2/4/2002
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	7/30/2004
Nitrobenzene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Nitrobenzene	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Nitroglycerin	EPA 8332	Extractable Organics	NELAP	5/12/2005
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	4/26/2002
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitrosodi-n-propylamine	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
n-Nitrosodi-n-propylamine	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002

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Analyte	Method/Tech	Category	Certification Type	Effective Date
n-Nitrosodiphenylamine	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
o,o,o-Triethyl phosphorothioate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	7/30/2004
Oil & Grease	EPA 9071	General Chemistry	NELAP	2/4/2002
Orthophosphate as P	EPA 9056	General Chemistry	NELAP	2/4/2002
o-Toluidine	EPA 8270	Extractable Organics	NELAP	2/4/2002
o-Xylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
o-Xylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Paint Filter Liquids Test	EPA 9095	General Chemistry	NELAP	2/4/2002
p-Dioxane	CA-204.07/GC-MS	Extractable Organics	NELAP	11/7/2006
p-Dioxane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Pentachlorobenzene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Pentachloroethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Pentachloronitrobenzene (Quintozene)	EPA 8270	Extractable Organics	NELAP	2/4/2002
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
Pentachlorophenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Pentachlorophenol (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Perchlorate	EPA 314.0	General Chemistry	NELAP	7/30/2004
pH	EPA 9040	General Chemistry	NELAP	2/4/2002
pH	EPA 9045	General Chemistry	NELAP	2/4/2002
Phenacetin	EPA 8270	Extractable Organics	NELAP	2/4/2002
Phenanthrene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Phenanthrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Phenanthrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Phenol	EPA 8270	Extractable Organics	NELAP	2/4/2002
Phenol	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Phenol	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	4/26/2002

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Analyte	Method/Tech	Category	Certification Type	Effective Date
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Potassium	EPA 6010	Metals	NELAP	2/4/2002
Potassium	EPA 6020	General Chemistry	NELAP	11/7/2006
Potassium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Pronamide (Kerb)	EPA 8270	Extractable Organics	NELAP	2/4/2002
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Pyrene	EPA 8270	Extractable Organics	NELAP	2/4/2002
Pyrene	OLM04.3-Exhibit D	Extractable Organics	NELAP	7/30/2004
Pyrene (without SIM)	SOM01.2 Exhibit D Semivolatiles/GC-MS	Extractable Organics	NELAP	5/8/2009
Pyridine	EPA 8270	Extractable Organics	NELAP	2/4/2002
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	7/30/2004
Safrole	EPA 8270	Extractable Organics	NELAP	2/4/2002
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Selenium	EPA 6010	Metals	NELAP	2/4/2002
Selenium	EPA 6020	Metals	NELAP	2/4/2002
Selenium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Silver	EPA 6010	Metals	NELAP	2/4/2002
Silver	EPA 6020	Metals	NELAP	2/4/2002
Silver	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	5/12/2005
Sodium	EPA 6010	Metals	NELAP	2/4/2002
Sodium	EPA 6020	General Chemistry	NELAP	11/7/2006
Sodium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Strontium	CA-627-02(EPA6020)/ICP-MS	Metals	NELAP	11/7/2006
Strontium	EPA 6010	Metals	NELAP	2/4/2002
Styrene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Styrene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Styrene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Styrene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Sulfate	EPA 9038	General Chemistry	NELAP	2/4/2002
Sulfate	EPA 9056	General Chemistry	NELAP	2/4/2002
Sulfotep	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	4/26/2002

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Synthetic Precipitation Leaching Procedure	EPA 1312	General Chemistry	NELAP	2/4/2002
T-amylmethylether (TAME)	EPA 8260	Extractable Organics	NELAP	5/8/2009
tert-Butyl alcohol	EPA 8260	Extractable Organics	NELAP	5/8/2009
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Tetrachloroethylene (Perchloroethylene)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Tetrachloroethylene (Perchloroethylene)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Tetrachloroethylene (Perchloroethylene)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Tetrahydrofuran (THF)	CA-202.08/GC-MS	Volatile Organics	NELAP	11/7/2006
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	7/30/2004
Thallium	EPA 6010	Metals	NELAP	2/4/2002
Thallium	EPA 6020	Metals	NELAP	2/4/2002
Thallium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Thionazin (Zinophos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Tin	EPA 6010	Metals	NELAP	7/30/2004
Titanium	EPA 6010	Metals	NELAP	7/30/2004
Toluene	EPA 8260	Volatile Organics	NELAP	2/4/2002
Toluene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Toluene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Toluene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Total cyanide	EPA 9012	General Chemistry	NELAP	4/26/2002
Total nitrate-nitrite	EPA 9056	General Chemistry	NELAP	2/4/2002
Total organic carbon	EPA 9060	General Chemistry	NELAP	2/4/2002
Total Petroleum Hydrocarbons (TPH)	FL-PRO	Extractable Organics	NELAP	2/4/2002
Total Petroleum Hydrocarbons (TPH)	TX1005	Extractable Organics	NELAP	9/4/2007
Total phenolics	EPA 9065	General Chemistry	NELAP	2/4/2002
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/4/2002
Toxaphene (Chlorinated camphene)	OLM04.3-Exhibit D	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Toxaphene (Chlorinated camphene)	SOM01.2 Exhibit D Pesticides/GC-ECD	Pesticides-Herbicides-PCB's	NELAP	5/8/2009
Toxicity Characteristic Leaching Procedure	EPA 1311	General Chemistry	NELAP	2/4/2002
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	2/4/2002
trans-1,2-Dichloroethylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004

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trans-1,2-Dichloroethylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,2-Dichloroethylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	2/4/2002
trans-1,3-Dichloropropylene	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
trans-1,3-Dichloropropylene	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,3-Dichloropropylene	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	4/26/2002
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Trichloroethene (Trichloroethylene)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Trichloroethene (Trichloroethylene)	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Trichloroethene (Trichloroethylene)	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	2/4/2002
Trichlorofluoromethane	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Trichlorofluoromethane	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Trichlorofluoromethane	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Vanadium	EPA 6010	Metals	NELAP	2/4/2002
Vanadium	EPA 6020	Metals	NELAP	2/4/2002
Vanadium	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	2/4/2002
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	2/4/2002
Vinyl chloride	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Vinyl chloride	SOM01.2 Exhibit D Low-Medium Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Vinyl chloride	SOM01.2 Exhibit D Trace Volatiles/GC-MS	Volatile Organics	NELAP	5/8/2009
Xylene (total)	EPA 8260	Volatile Organics	NELAP	2/4/2002
Xylene (total)	OLM04.3-Exhibit D	Volatile Organics	NELAP	7/30/2004
Zinc	EPA 6010	Metals	NELAP	2/4/2002
Zinc	EPA 6020	Metals	NELAP	2/4/2002

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 7/1/2010

Expiration Date: 6/30/2011

Charlie Crist
Governor



Ana M. Viamonte Ros, M.D., M.P.H.
State Surgeon General

Laboratory Scope of Accreditation

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Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604 EPA Lab Code: ME00019 (207) 874-2400

E87604
Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Zinc	ILM05.3-Exhibit D/ICP-AES	Metals	NELAP	11/7/2006

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program.

Issue Date: 7/1/2010

Expiration Date: 6/30/2011



Laboratory Scope of Accreditation

Attachment to Certificate #: E87604-15, expiration date June 30, 2011. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87604

EPA Lab Code:

ME00019

(207) 874-2400

E87604

Katahdin Analytical Services, Inc.
600 Technology Way
Scarborough, ME 04074

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Mercury	EPA 7470	General Chemistry	NELAP	7/30/2004
Mercury	EPA 7471	General Chemistry	NELAP	7/30/2004
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/30/2004



**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).

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

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-butene
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
Calculation	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 / 4500NH ₃ H	Ammonia as N
UV/VIS	3500Fe D	Ferrous Iron
UV/VIS	351.2	Kjeldahl nitrogen - total
UV/VIS	353.2 / 4500NO ₃ 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-propylbenzene
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-butene
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	Pyridine
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)	Hexahydro-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



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.

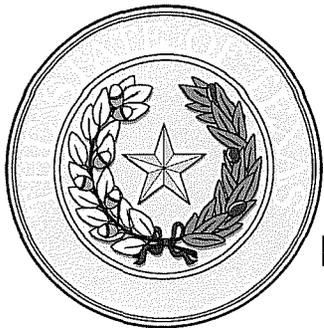


Approved By: _____

R. Douglas Leonard
Chief Technical Officer

Date: November 4, 2009

Issued: 11/04/09



Texas Commission on Environmental Quality

NELAP-Recognized Laboratory Accreditation is hereby awarded to



Katahdin Analytical Services, Inc

600 Technology Way
Scarborough, ME 04074-7647

in accordance with Texas Water Code Chapter 5, Subchapter R, Title 30 Texas Administrative Code Chapter 25, and the National Environmental Laboratory Accreditation Program.

The laboratory's scope of accreditation includes the fields of accreditation that accompany this certificate. Continued accreditation depends upon successful ongoing participation in the program. The Texas Commission on Environmental Quality urges customers to verify the laboratory's current accreditation status for particular methods and analyses.

Certificate Number: T104704243-09-1

Effective Date: 11/1/2009

Expiration Date: 10/31/2010

A handwritten signature in black ink, appearing to read "Mark Wiley".

**Executive Director Texas Commission on
Environmental Quality**



Texas Commission on Environmental Quality



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 Expiration Date: 10/31/2010
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These fields of accreditation supercede all previous fields. The Texas Commission on Environmental Quality urges customers to verify the laboratory's current accreditation status for particular methods and analyses.

Matrix: Non Potable Water

Method ASTM D516			
Analyte Sulfate	AB FL	Analyte ID 2000	Method ID 30002201
Method EPA 1010			
Analyte Ignitability	AB FL	Analyte ID 1780	Method ID 10116606
Method EPA 110.2			
Analyte Color	AB FL	Analyte ID 1605	Method ID 10005604
Method EPA 120.1			
Analyte Conductivity	AB FL	Analyte ID 1610	Method ID 10006403
Method EPA 130.2			
Analyte Total hardness as CaCO ₃	AB FL	Analyte ID 1755	Method ID 10007202
Method EPA 150.1			
Analyte pH	AB FL	Analyte ID 1900	Method ID 10008409
Method EPA 160.1			
Analyte Residue-filterable (TDS)	AB FL	Analyte ID 1955	Method ID 10009208
Method EPA 160.2			
Analyte Residue-nonfilterable (TSS)	AB FL	Analyte ID 1960	Method ID 10009606
Method EPA 160.3			
Analyte Residue-total	AB FL	Analyte ID 1950	Method ID 10010001
Method EPA 160.4			
Analyte Residue-volatile	AB FL	Analyte ID 1970	Method ID 10010409
Method EPA 160.5			
Analyte	AB	Analyte ID	Method ID



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Matrix: Non Potable Water

Residue-settleable	FL	1965	10010807
Method EPA 1631E			
Analyte	AB	Analyte ID	Method ID
Mercury	FL	1095	10237204
Method EPA 1664			
Analyte	AB	Analyte ID	Method ID
n-Hexane Extractable Material (O&G)	FL	1803	10127409
Method EPA 180.1			
Analyte	AB	Analyte ID	Method ID
Turbidity	FL	2055	10011606
Method EPA 200.7			
Analyte	AB	Analyte ID	Method ID
Aluminum	FL	1000	10013806
Antimony	FL	1005	10013806
Arsenic	FL	1010	10013806
Barium	FL	1015	10013806
Beryllium	FL	1020	10013806
Boron	FL	1025	10013806
Cadmium	FL	1030	10013806
Calcium	FL	1035	10013806
Chromium	FL	1040	10013806
Cobalt	FL	1050	10013806
Copper	FL	1055	10013806
Iron	FL	1070	10013806
Lead	FL	1075	10013806
Magnesium	FL	1085	10013806
Manganese	FL	1090	10013806
Molybdenum	FL	1100	10013806
Nickel	FL	1105	10013806
Potassium	FL	1125	10013806
Selenium	FL	1140	10013806



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Matrix: Non Potable Water

Silver	FL	1150	10013806
Sodium	FL	1155	10013806
Thallium	FL	1165	10013806
Tin	FL	1175	10013806
Titanium	FL	1180	10013806
Vanadium	FL	1185	10013806
Zinc	FL	1190	10013806
Method EPA 200.8			
Analyte	AB	Analyte ID	Method ID
Aluminum	FL	1000	10014605
Antimony	FL	1005	10014605
Arsenic	FL	1010	10014605
Barium	FL	1015	10014605
Beryllium	FL	1020	10014605
Boron	FL	1025	10014605
Cadmium	FL	1030	10014605
Calcium	FL	1035	10014605
Chromium	FL	1040	10014605
Cobalt	FL	1050	10014605
Copper	FL	1055	10014605
Iron	FL	1070	10014605
Lead	FL	1075	10014605
Magnesium	FL	1085	10014605
Manganese	FL	1090	10014605
Molybdenum	FL	1100	10014605
Nickel	FL	1105	10014605
Potassium	FL	1125	10014605
Selenium	FL	1140	10014605
Silver	FL	1150	10014605
Sodium	FL	1155	10014605
Thallium	FL	1165	10014605



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Matrix: *Non Potable Water*

Thorium	FL	1170	10014605
Tin	FL	1175	10014605
Uranium	FL	3035	10014605
Vanadium	FL	1185	10014605
Zinc	FL	1190	10014605
Method EPA 245.1			
Analyte	AB	Analyte ID	Method ID
Mercury	FL	1095	10036609
Method EPA 300.0			
Analyte	AB	Analyte ID	Method ID
Bromide	FL	1540	10053006
Chloride	FL	1575	10053006
Nitrate as N	FL	1810	10053006
Nitrate-nitrite	FL	1820	10053006
Nitrite as N	FL	1840	10053006
Orthophosphate as P	FL	1870	10053006
Sulfate	FL	2000	10053006
Method EPA 305.1			
Analyte	AB	Analyte ID	Method ID
Acidity, as CaCO ₃	FL	1500	10054203
Method EPA 310.1			
Analyte	AB	Analyte ID	Method ID
Alkalinity as CaCO ₃	FL	1505	10054805
Method EPA 310.2			
Analyte	AB	Analyte ID	Method ID
Alkalinity as CaCO ₃	FL	1505	10055206
Method EPA 314.0			
Analyte	AB	Analyte ID	Method ID
Perchlorate	FL	1895	10055400
Method EPA 325.2			
Analyte	AB	Analyte ID	Method ID



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Matrix: *Non Potable Water*

Chloride	FL	1575	10057202
Method EPA 335.1			
Analyte	AB	Analyte ID	Method ID
Amenable cyanide	FL	1510	10060001
Method EPA 335.3			
Analyte	AB	Analyte ID	Method ID
Total cyanide	FL	1645	10061004
Method EPA 335.4			
Analyte	AB	Analyte ID	Method ID
Total cyanide	FL	1645	10061402
Method EPA 340.2			
Analyte	AB	Analyte ID	Method ID
Fluoride	FL	1730	10062201
Method EPA 350.1			
Analyte	AB	Analyte ID	Method ID
Ammonia as N	FL	1515	10063408
Method EPA 351.2			
Analyte	AB	Analyte ID	Method ID
Kjeldahl nitrogen - total	FL	1795	10065200
Method EPA 353.2			
Analyte	AB	Analyte ID	Method ID
Nitrate as N	FL	1810	10067400
Nitrate-nitrite	FL	1820	10067400
Nitrite as N	FL	1840	10067400
Method EPA 365.2			
Analyte	AB	Analyte ID	Method ID
Orthophosphate as P	FL	1870	10070403
Method EPA 365.4			
Analyte	AB	Analyte ID	Method ID
Phosphorus, total	FL	1910	10071202
Method EPA 375.4			
Analyte	AB	Analyte ID	Method ID



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Matrix: Non Potable Water

Sulfate	FL	2000	10073800
Method EPA 376.1			
Analyte	AB	Analyte ID	Method ID
Sulfide	FL	2005	10074201
Method EPA 377.1			
Analyte	AB	Analyte ID	Method ID
Sulfite-SO3	FL	2015	10075000
Method EPA 405.1			
Analyte	AB	Analyte ID	Method ID
Biochemical oxygen demand	FL	1530	10075602
Method EPA 410.4			
Analyte	AB	Analyte ID	Method ID
Chemical oxygen demand	FL	1565	10077200
Method EPA 415.1			
Analyte	AB	Analyte ID	Method ID
Total organic carbon	FL	2040	10078407
Method EPA 420.1			
Analyte	AB	Analyte ID	Method ID
Total phenolics	FL	1905	10079400
Method EPA 6010			
Analyte	AB	Analyte ID	Method ID
Aluminum	FL	1000	10155201
Antimony	FL	1005	10155201
Arsenic	FL	1010	10155201
Barium	FL	1015	10155201
Beryllium	FL	1020	10155201
Boron	FL	1025	10155201
Cadmium	FL	1030	10155201
Calcium	FL	1035	10155201
Chromium	FL	1040	10155201
Cobalt	FL	1050	10155201



Texas Commission on Environmental Quality



NELAP - Recognized Laboratory Fields of Accreditation

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Matrix: Non Potable Water

Copper	FL	1055	10155201
Iron	FL	1070	10155201
Lead	FL	1075	10155201
Magnesium	FL	1085	10155201
Manganese	FL	1090	10155201
Molybdenum	FL	1100	10155201
Nickel	FL	1105	10155201
Potassium	FL	1125	10155201
Selenium	FL	1140	10155201
Silver	FL	1150	10155201
Sodium	FL	1155	10155201
Strontium	FL	1160	10155201
Thallium	FL	1165	10155201
Tin	FL	1175	10155201
Titanium	FL	1180	10155201
Vanadium	FL	1185	10155201
Zinc	FL	1190	10155201

Method EPA 6020

Analyte	AB	Analyte ID	Method ID
Aluminum	FL	1000	10156204
Antimony	FL	1005	10156204
Arsenic	FL	1010	10156204
Barium	FL	1015	10156204
Beryllium	FL	1020	10156204
Boron	FL	1025	10156204
Cadmium	FL	1030	10156204
Calcium	FL	1035	10156204
Chromium	FL	1040	10156204
Cobalt	FL	1050	10156204
Copper	FL	1055	10156204
Iron	FL	1070	10156204



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Lead	FL	1075	10156204
Magnesium	FL	1085	10156204
Manganese	FL	1090	10156204
Molybdenum	FL	1100	10156204
Nickel	FL	1105	10156204
Potassium	FL	1125	10156204
Selenium	FL	1140	10156204
Silver	FL	1150	10156204
Sodium	FL	1155	10156204
Strontium	FL	1160	10156204
Thallium	FL	1165	10156204
Vanadium	FL	1185	10156204
Zinc	FL	1190	10156204

Method EPA 608

Analyte	AB	Analyte ID	Method ID
4,4'-DDD	FL	7355	10103603
4,4'-DDE	FL	7360	10103603
4,4'-DDT	FL	7365	10103603
Aldrin	FL	7025	10103603
alpha-BHC (alpha-Hexachlorocyclohexane)	FL	7110	10103603
Aroclor-1016 (PCB-1016)	FL	8880	10103603
Aroclor-1221 (PCB-1221)	FL	8885	10103603
Aroclor-1232 (PCB-1232)	FL	8890	10103603
Aroclor-1242 (PCB-1242)	FL	8895	10103603
Aroclor-1248 (PCB-1248)	FL	8900	10103603
Aroclor-1254 (PCB-1254)	FL	8905	10103603
Aroclor-1260 (PCB-1260)	FL	8910	10103603
beta-BHC (beta-Hexachlorocyclohexane)	FL	7115	10103603
Chlordane (tech.)	FL	7250	10103603
delta-BHC (delta-Hexachlorocyclohexane)	FL	7105	10103603
Dieldrin	FL	7470	10103603



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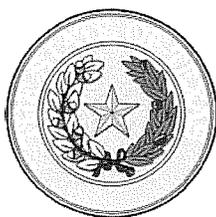
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Matrix: Non Potable Water

Endosulfan I	FL	7510	10103603
Endosulfan II	FL	7515	10103603
Endosulfan sulfate	FL	7520	10103603
Endrin	FL	7540	10103603
Endrin aldehyde	FL	7530	10103603
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	FL	7120	10103603
Heptachlor	FL	7685	10103603
Heptachlor epoxide	FL	7690	10103603
Toxaphene (Chlorinated camphene)	FL	8250	10103603

Method EPA 624

Analyte	AB	Analyte ID	Method ID
1,1,1-Trichloroethane	FL	5160	10107207
1,1,2,2-Tetrachloroethane	FL	5110	10107207
1,1,2-Trichloroethane	FL	5165	10107207
1,1-Dichloroethane	FL	4630	10107207
1,1-Dichloroethylene (1,1-Dichloroethene)	FL	4640	10107207
1,2-Dichlorobenzene	FL	4610	10107207
1,2-Dichloroethane	FL	4635	10107207
1,2-Dichloropropane	FL	4655	10107207
1,3-Dichlorobenzene	FL	4615	10107207
1,4-Dichlorobenzene	FL	4620	10107207
2-Chloroethyl vinyl ether	FL	4500	10107207
Acrolein (Propenal)	FL	4325	10107207
Acrylonitrile	FL	4340	10107207
Benzene	FL	4375	10107207
Bromodichloromethane	FL	4395	10107207
Bromoform	FL	4400	10107207
Bromomethane (Methyl bromide)	FL	4950	10107207
Carbon tetrachloride	FL	4455	10107207
Chlorobenzene	FL	4475	10107207
Chloroethane	FL	4485	10107207



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Matrix: *Non Potable Water*

Chloroform	FL	4505	10107207
Chloromethane (Methyl chloride)	FL	4960	10107207
cis-1,3-Dichloropropylene	FL	4680	10107207
Dibromochloromethane	FL	4575	10107207
Ethylbenzene	FL	4765	10107207
Methylene chloride	FL	4975	10107207
Tetrachloroethylene (Perchloroethylene)	FL	5115	10107207
Toluene	FL	5140	10107207
trans-1,2-Dichloroethylene	FL	4700	10107207
trans-1,3-Dichloropropylene	FL	4685	10107207
Trichloroethene (Trichloroethylene)	FL	5170	10107207
Trichlorofluoromethane	FL	5175	10107207
Vinyl chloride	FL	5235	10107207
Xylene (total)	FL	5260	10107207

Method EPA 625

Analyte	AB	Analyte ID	Method ID
1,2,4-Trichlorobenzene	FL	5155	10107401
1,2-Dichlorobenzene	FL	4610	10107401
1,3-Dichlorobenzene	FL	4615	10107401
1,4-Dichlorobenzene	FL	4620	10107401
2,4,6-Trichlorophenol	FL	6840	10107401
2,4-Dichlorophenol	FL	6000	10107401
2,4-Dimethylphenol	FL	6130	10107401
2,4-Dinitrophenol	FL	6175	10107401
2,4-Dinitrotoluene (2,4-DNT)	FL	6185	10107401
2,6-Dinitrotoluene (2,6-DNT)	FL	6190	10107401
2-Chloronaphthalene	FL	5795	10107401
2-Chlorophenol	FL	5800	10107401
2-Methyl-4,6-dinitrophenol	FL	6360	10107401
2-Nitrophenol	FL	6490	10107401
3,3'-Dichlorobenzidine	FL	5945	10107401



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Matrix: *Non Potable Water*

4-Bromophenyl phenyl ether	FL	5660	10107401
4-Chloro-3-methylphenol	FL	5700	10107401
4-Chlorophenyl phenylether	FL	5825	10107401
4-Nitrophenol	FL	6500	10107401
Acenaphthene	FL	5500	10107401
Acenaphthylene	FL	5505	10107401
Anthracene	FL	5555	10107401
Benzidine	FL	5595	10107401
Benzo(a)anthracene	FL	5575	10107401
Benzo(a)pyrene	FL	5580	10107401
Benzo(b)fluoranthene	FL	5585	10107401
Benzo(g,h,i)perylene	FL	5590	10107401
Benzo(k)fluoranthene	FL	5600	10107401
bis(2-Chloroethoxy)methane	FL	5760	10107401
bis(2-Chloroethyl) ether	FL	5765	10107401
bis(2-Chloroisopropyl) ether	FL	5780	10107401
bis(2-Ethylhexyl) phthalate (DEHP)	FL	6255	10107401
Butyl benzyl phthalate	FL	5670	10107401
Chrysene	FL	5855	10107401
Dibenz(a,h) anthracene	FL	5895	10107401
Diethyl phthalate	FL	6070	10107401
Dimethyl phthalate	FL	6135	10107401
Di-n-butyl phthalate	FL	5925	10107401
Di-n-octyl phthalate	FL	6200	10107401
Fluoranthene	FL	6265	10107401
Fluorene	FL	6270	10107401
Hexachlorobenzene	FL	6275	10107401
Hexachlorobutadiene	FL	4835	10107401
Hexachlorocyclopentadiene	FL	6285	10107401
Hexachloroethane	FL	4840	10107401



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Matrix: Non Potable Water

Indeno(1,2,3-cd) pyrene	FL	6315	10107401
Isophorone	FL	6320	10107401
Naphthalene	FL	5005	10107401
Nitrobenzene	FL	5015	10107401
n-Nitrosodimethylamine	FL	6530	10107401
n-Nitrosodi-n-propylamine	FL	6545	10107401
n-Nitrosodiphenylamine	FL	6535	10107401
Pentachlorophenol	FL	6605	10107401
Phenanthrene	FL	6615	10107401
Phenol	FL	6625	10107401
Pyrene	FL	6665	10107401
Method EPA 7196			
Analyte	AB	Analyte ID	Method ID
Chromium VI	FL	1045	10162206
Method EPA 7470			
Analyte	AB	Analyte ID	Method ID
Mercury	FL	1095	10165603
Method EPA 8011			
Analyte	AB	Analyte ID	Method ID
1,2-Dibromo-3-chloropropane (DBCP)	FL	4570	10173009
1,2-Dibromoethane (EDB, Ethylene dibromide)	FL	4585	10173009
Method EPA 8015			
Analyte	AB	Analyte ID	Method ID
2-Propanol (Isopropyl alcohol)	FL	4895	10173203
Diesel range organics (DRO)	FL	9369	10173203
Ethanol	FL	4750	10173203
Gasoline range organics (GRO)	FL	9408	10173203
Isobutyl alcohol (2-Methyl-1-propanol)	FL	4875	10173203
Methanol	FL	4930	10173203
n-Propanol	FL	5055	10173203



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Matrix: *Non Potable Water*

Method EPA 8081

Analyte	AB	Analyte ID	Method ID
4,4'-DDD	FL	7355	10178402
4,4'-DDE	FL	7360	10178402
4,4'-DDT	FL	7365	10178402
Aldrin	FL	7025	10178402
alpha-BHC (alpha-Hexachlorocyclohexane)	FL	7110	10178402
alpha-Chlordane	FL	7240	10178402
beta-BHC (beta-Hexachlorocyclohexane)	FL	7115	10178402
Chlordane (tech.)	FL	7250	10178402
delta-BHC (delta-Hexachlorocyclohexane)	FL	7105	10178402
Dieldrin	FL	7470	10178402
Endosulfan I	FL	7510	10178402
Endosulfan II	FL	7515	10178402
Endosulfan sulfate	FL	7520	10178402
Endrin	FL	7540	10178402
Endrin aldehyde	FL	7530	10178402
Endrin ketone	FL	7535	10178402
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	FL	7120	10178402
gamma-Chlordane	FL	7245	10178402
Heptachlor	FL	7685	10178402
Heptachlor epoxide	FL	7690	10178402
Methoxychlor	FL	7810	10178402
Toxaphene (Chlorinated camphene)	FL	8250	10178402

Method EPA 8082

Analyte	AB	Analyte ID	Method ID
2,2', 3,3', 4,4', 5-Heptachlorobiphenyl	FL	9065	10179007
2,2', 3,3',4,4'-Hexachlorobiphenyl	FL	9020	10179007
2,2', 3,4', 5,5', 6-Heptachlorobiphenyl	FL	9080	10179007
2,2', 3,4,4', 5,5'-Heptachlorobiphenyl	FL	9070	10179007
2,2', 3,4,4', 5'-Hexachlorobiphenyl	FL	9025	10179007



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Matrix: Non Potable Water

2,2', 3,5'-Tetrachlorobiphenyl	FL	8945	10179007
2,2', 4,5,5'-Pentachlorobiphenyl	FL	8980	10179007
2,2', 4,5'-Tetrachlorobiphenyl	FL	8950	10179007
2,2', 5,5'-Tetrachlorobiphenyl	FL	8955	10179007
2,2', 5-Trichlorobiphenyl	FL	8930	10179007
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	FL	9095	10179007
2,2',3,3',4,4',5,6-Octachlorobiphenyl	FL	9103	10179007
2,2',3,4,4',5,6'-Heptachlorobiphenyl	FL	9136	10179007
2,2',3',4,5-Pentachlorobiphenyl	FL	9154	10179007
2,3', 4,4', 5-Pentachlorobiphenyl	FL	9010	10179007
2,3', 4,4'-Tetrachlorobiphenyl	FL	8960	10179007
2,3,3',4,4',5,5'-Heptachlorobiphenyl	FL	9085	10179007
2,3,3',4,4',5-Hexachlorobiphenyl	FL	9050	10179007
2,3,3',4,4'-Pentachlorobiphenyl	FL	8985	10179007
2,3',4,4',5,5'-Hexachlorobiphenyl	FL	9055	10179007
2,3,4,4',5-Pentachlorobiphenyl	FL	9005	10179007
2,4,4'-Trichlorobiphenyl	FL	9252	10179007
2,4'-Dichlorobiphenyl	FL	9256	10179007
Aroclor-1016 (PCB-1016)	FL	8880	10179007
Aroclor-1221 (PCB-1221)	FL	8885	10179007
Aroclor-1232 (PCB-1232)	FL	8890	10179007
Aroclor-1242 (PCB-1242)	FL	8895	10179007
Aroclor-1248 (PCB-1248)	FL	8900	10179007
Aroclor-1254 (PCB-1254)	FL	8905	10179007
Aroclor-1260 (PCB-1260)	FL	8910	10179007
PCBs	FL	8870	10179007

Method EPA 8151

Analyte	AB	Analyte ID	Method ID
2,4,5-T	FL	8655	10183003
2,4-D	FL	8545	10183003
2,4-DB	FL	8560	10183003



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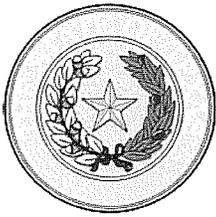
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Matrix: Non Potable Water

Dalapon	FL	8555	10183003
Dicamba	FL	8595	10183003
Dichloroprop (Dichlorprop)	FL	8605	10183003
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	FL	8620	10183003
MCPA	FL	7775	10183003
MCPP	FL	7780	10183003
Silvex (2,4,5-TP)	FL	8650	10183003

Method EPA 8260

Analyte	AB	Analyte ID	Method ID
1,1,1,2-Tetrachloroethane	FL	5105	10184404
1,1,1-Trichloroethane	FL	5160	10184404
1,1,2,2-Tetrachloroethane	FL	5110	10184404
1,1,2-Trichloroethane	FL	5165	10184404
1,1-Dichloroethane	FL	4630	10184404
1,1-Dichloroethylene (1,1-Dichloroethene)	FL	4640	10184404
1,1-Dichloropropene	FL	4670	10184404
1,2,3-Trichlorobenzene	FL	5150	10184404
1,2,3-Trichloropropane	FL	5180	10184404
1,2,4-Trichlorobenzene	FL	5155	10184404
1,2,4-Trimethylbenzene	FL	5210	10184404
1,2-Dibromo-3-chloropropane (DBCP)	FL	4570	10184404
1,2-Dibromoethane (EDB, Ethylene dibromide)	FL	4585	10184404
1,2-Dichlorobenzene	FL	4610	10184404
1,2-Dichloroethane	FL	4635	10184404
1,2-Dichloropropane	FL	4655	10184404
1,3,5-Trimethylbenzene	FL	5215	10184404
1,3-Dichlorobenzene	FL	4615	10184404
1,3-Dichloropropane	FL	4660	10184404
1,4-Dichlorobenzene	FL	4620	10184404
1,4-Dioxane (1,4-Diethyleneoxide)	FL	4735	10184404
2,2-Dichloropropane	FL	4665	10184404



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Matrix: Non Potable Water

2-Butanone (Methyl ethyl ketone, MEK)	FL	4410	10184404
2-Chloroethyl vinyl ether	FL	4500	10184404
2-Chlorotoluene	FL	4535	10184404
2-Hexanone	FL	4860	10184404
4-Chlorotoluene	FL	4540	10184404
4-Isopropyltoluene	FL	4915	10184404
4-Methyl-2-pentanone (MIBK)	FL	4995	10184404
Acetone	FL	4315	10184404
Acetonitrile	FL	4320	10184404
Acrolein (Propenal)	FL	4325	10184404
Acrylonitrile	FL	4340	10184404
Allyl chloride (3-Chloropropene)	FL	4355	10184404
Benzene	FL	4375	10184404
Bromobenzene	FL	4385	10184404
Bromochloromethane	FL	4390	10184404
Bromodichloromethane	FL	4395	10184404
Bromoform	FL	4400	10184404
Bromomethane (Methyl bromide)	FL	4950	10184404
Carbon disulfide	FL	4450	10184404
Carbon tetrachloride	FL	4455	10184404
Chlorobenzene	FL	4475	10184404
Chloroethane	FL	4485	10184404
Chloroform	FL	4505	10184404
Chloromethane (Methyl chloride)	FL	4960	10184404
Chloroprene	FL	4525	10184404
cis-1,2-Dichloroethylene	FL	4645	10184404
cis-1,3-Dichloropropylene	FL	4680	10184404
Dibromochloromethane	FL	4575	10184404
Dibromomethane	FL	4595	10184404
Dichlorodifluoromethane	FL	4625	10184404



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Matrix: Non Potable Water

Diethyl ether	FL	4725	10184404
Ethyl methacrylate	FL	4810	10184404
Ethylbenzene	FL	4765	10184404
Hexachlorobutadiene	FL	4835	10184404
Iodomethane (Methyl iodide)	FL	4870	10184404
Isobutyl alcohol (2-Methyl-1-propanol)	FL	4875	10184404
Isopropylbenzene	FL	4900	10184404
Methacrylonitrile	FL	4925	10184404
Methyl methacrylate	FL	4990	10184404
Methyl tert-butyl ether (MTBE)	FL	5000	10184404
Methylene chloride	FL	4975	10184404
Naphthalene	FL	5005	10184404
n-Butylbenzene	FL	4435	10184404
n-Propylbenzene	FL	5090	10184404
Pentachloroethane	FL	5035	10184404
Propionitrile (Ethyl cyanide)	FL	5080	10184404
sec-Butylbenzene	FL	4440	10184404
Styrene	FL	5100	10184404
tert-Butylbenzene	FL	4445	10184404
Tetrachloroethylene (Perchloroethylene)	FL	5115	10184404
Toluene	FL	5140	10184404
trans-1,2-Dichloroethylene	FL	4700	10184404
trans-1,3-Dichloropropylene	FL	4685	10184404
trans-1,4-Dichloro-2-butene	FL	4605	10184404
Trichloroethene (Trichloroethylene)	FL	5170	10184404
Trichlorofluoromethane	FL	5175	10184404
Vinyl acetate	FL	5225	10184404
Vinyl chloride	FL	5235	10184404
Xylene (total)	FL	5260	10184404

Method EPA 8270

Analyte

AB

Analyte ID

Method ID



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Matrix: *Non Potable Water*

1,2,4,5-Tetrachlorobenzene	FL	6715	10185203
1,2,4-Trichlorobenzene	FL	5155	10185203
1,2-Dichlorobenzene	FL	4610	10185203
1,2-Diphenylhydrazine	FL	6220	10185203
1,3,5-Trinitrobenzene (1,3,5-TNB)	FL	6885	10185203
1,3-Dichlorobenzene	FL	4615	10185203
1,3-Dinitrobenzene (1,3-DNB)	FL	6160	10185203
1,4-Dichlorobenzene	FL	4620	10185203
1,4-Naphthoquinone	FL	6420	10185203
1,4-Phenylenediamine	FL	6630	10185203
1-Naphthylamine	FL	6425	10185203
2,3,4,6-Tetrachlorophenol	FL	6735	10185203
2,4,5-Trichlorophenol	FL	6835	10185203
2,4,6-Trichlorophenol	FL	6840	10185203
2,4-Dichlorophenol	FL	6000	10185203
2,4-Dimethylphenol	FL	6130	10185203
2,4-Dinitrophenol	FL	6175	10185203
2,4-Dinitrotoluene (2,4-DNT)	FL	6185	10185203
2,6-Dichlorophenol	FL	6005	10185203
2,6-Dinitrotoluene (2,6-DNT)	FL	6190	10185203
2-Acetylamino fluorene	FL	5515	10185203
2-Chloronaphthalene	FL	5795	10185203
2-Chlorophenol	FL	5800	10185203
2-Methyl-4,6-dinitrophenol	FL	6360	10185203
2-Methylnaphthalene	FL	6385	10185203
2-Methylphenol (o-Cresol)	FL	6400	10185203
2-Naphthylamine	FL	6430	10185203
2-Nitroaniline	FL	6460	10185203
2-Nitrophenol	FL	6490	10185203
2-Picoline (2-Methylpyridine)	FL	5050	10185203



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NELAP - Recognized Laboratory Fields of Accreditation

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Matrix: Non Potable Water

3,3'-Dichlorobenzidine	FL	5945	10185203
3,3'-Dimethylbenzidine	FL	6120	10185203
3-Methylcholanthrene	FL	6355	10185203
3-Nitroaniline	FL	6465	10185203
4-Aminobiphenyl	FL	5540	10185203
4-Bromophenyl phenyl ether	FL	5660	10185203
4-Chloro-3-methylphenol	FL	5700	10185203
4-Chloroaniline	FL	5745	10185203
4-Chlorophenyl phenylether	FL	5825	10185203
4-Dimethyl aminoazobenzene	FL	6105	10185203
4-Methylphenol (p-Cresol)	FL	6410	10185203
4-Nitroaniline	FL	6470	10185203
4-Nitrophenol	FL	6500	10185203
5-Nitro-o-toluidine	FL	6570	10185203
7,12-Dimethylbenz(a) anthracene	FL	6115	10185203
a-a-Dimethylphenethylamine	FL	6125	10185203
Acenaphthene	FL	5500	10185203
Acenaphthylene	FL	5505	10185203
Acetophenone	FL	5510	10185203
Aniline	FL	5545	10185203
Anthracene	FL	5555	10185203
Aramite	FL	5560	10185203
Benzidine	FL	5595	10185203
Benzo(a)anthracene	FL	5575	10185203
Benzo(a)pyrene	FL	5580	10185203
Benzo(b)fluoranthene	FL	5585	10185203
Benzo(g,h,i)perylene	FL	5590	10185203
Benzo(k)fluoranthene	FL	5600	10185203
Benzoic acid	FL	5610	10185203
Benzyl alcohol	FL	5630	10185203



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Matrix: Non Potable Water

bis(2-Chloroethoxy)methane	FL	5760	10185203
bis(2-Chloroethyl) ether	FL	5765	10185203
bis(2-Chloroisopropyl) ether	FL	5780	10185203
bis(2-Ethylhexyl) phthalate (DEHP)	FL	6255	10185203
Butyl benzyl phthalate	FL	5670	10185203
Carbazole	FL	5680	10185203
Chlorobenzilate	FL	7260	10185203
Chrysene	FL	5855	10185203
Diallate	FL	7405	10185203
Dibenz(a,h) anthracene	FL	5895	10185203
Dibenzofuran	FL	5905	10185203
Diethyl phthalate	FL	6070	10185203
Dimethoate	FL	7475	10185203
Dimethyl phthalate	FL	6135	10185203
Di-n-butyl phthalate	FL	5925	10185203
Di-n-octyl phthalate	FL	6200	10185203
Ethyl methanesulfonate	FL	6260	10185203
Famphur	FL	7580	10185203
Fluoranthene	FL	6265	10185203
Fluorene	FL	6270	10185203
Hexachlorobenzene	FL	6275	10185203
Hexachlorobutadiene	FL	4835	10185203
Hexachlorocyclopentadiene	FL	6285	10185203
Hexachloroethane	FL	4840	10185203
Hexachloropropene	FL	6295	10185203
Indeno(1,2,3-cd) pyrene	FL	6315	10185203
Isodrin	FL	7725	10185203
Isophorone	FL	6320	10185203
Isosafrole	FL	6325	10185203
Methapyrilene	FL	6345	10185203



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Matrix: Non Potable Water

Methyl methanesulfonate	FL	6375	10185203
Naphthalene	FL	5005	10185203
Nitrobenzene	FL	5015	10185203
Nitroquinoline-1-oxide	FL	6515	10185203
n-Nitrosodiethylamine	FL	6525	10185203
n-Nitrosodimethylamine	FL	6530	10185203
n-Nitroso-di-n-butylamine	FL	5025	10185203
n-Nitrosodi-n-propylamine	FL	6545	10185203
n-Nitrosodiphenylamine	FL	6535	10185203
n-Nitrosomethylethylamine	FL	6550	10185203
n-Nitrosomorpholine	FL	6555	10185203
n-Nitrosopiperidine	FL	6560	10185203
n-Nitrosopyrrolidine	FL	6565	10185203
o,o,o-Triethyl phosphorothioate	FL	8290	10185203
o-Toluidine	FL	5145	10185203
Parathion, methyl (Methyl parathion)	FL	7825	10185203
Pentachlorobenzene	FL	6590	10185203
Pentachloronitrobenzene	FL	6600	10185203
Pentachlorophenol	FL	6605	10185203
Phenacetin	FL	6610	10185203
Phenanthrene	FL	6615	10185203
Phenol	FL	6625	10185203
Phorate	FL	7985	10185203
Pronamide (Kerb)	FL	6650	10185203
Pyrene	FL	6665	10185203
Pyridine	FL	5095	10185203
Safrole	FL	6685	10185203
Sulfotepp	FL	8155	10185203
Thionazin (Zinophos)	FL	8235	10185203

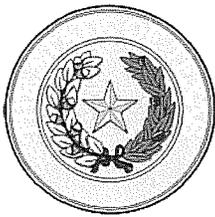
Method EPA 8330

Analyte

AB

Analyte ID

Method ID



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Matrix: Non Potable Water

1,3,5-Trinitrobenzene (1,3,5-TNB)	FL	6885	10189807
1,3-Dinitrobenzene (1,3-DNB)	FL	6160	10189807
2,4,6-Trinitrotoluene (2,4,6-TNT)	FL	9651	10189807
2,4-Dinitrotoluene (2,4-DNT)	FL	6185	10189807
2,6-Dinitrotoluene (2,6-DNT)	FL	6190	10189807
2-Amino-4,6-dinitrotoluene (2-am-dnt)	FL	9303	10189807
2-Nitrotoluene	FL	9507	10189807
3-Nitrotoluene	FL	9510	10189807
4-Amino-2,6-dinitrotoluene (4-am-dnt)	FL	9306	10189807
4-Nitrotoluene	FL	9513	10189807
Nitrobenzene	FL	5015	10189807
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	FL	9522	10189807
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	FL	9432	10189807
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	FL	9633	10189807
Method EPA 8332			
Analyte	AB	Analyte ID	Method ID
Nitroglycerin	FL	6485	10190406
Method EPA 9012			
Analyte	AB	Analyte ID	Method ID
Amenable cyanide	FL	1510	10193201
Total cyanide	FL	1645	10193405
Method EPA 9038			
Analyte	AB	Analyte ID	Method ID
Sulfate	FL	2000	10196608
Method EPA 9040			
Analyte	AB	Analyte ID	Method ID
pH	FL	1900	10196802
Method EPA 9056			
Analyte	AB	Analyte ID	Method ID
Bromide	FL	1540	10199209
Chloride	FL	1575	10199209



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Matrix: Non Potable Water

Nitrate as N	FL	1810	10199209
Nitrate-nitrite	FL	1820	10199209
Nitrite as N	FL	1840	10199209
Orthophosphate as P	FL	1870	10199209
Sulfate	FL	2000	10199209
Method EPA 9060			
Analyte	AB	Analyte ID	Method ID
Total organic carbon	FL	2040	10200201
Method EPA 9065			
Analyte	AB	Analyte ID	Method ID
Total phenolics	FL	1905	10200405
Method EPA 9070			
Analyte	AB	Analyte ID	Method ID
n-Hexane Extractable Material (O&G)	FL	1803	10201000
Silica Gel Treated n-Hexane Extractable Material	FL	10220	10201000
Method EPA 9251			
Analyte	AB	Analyte ID	Method ID
Chloride	FL	1575	10207406
Method HACH 8000			
Analyte	AB	Analyte ID	Method ID
Chemical oxygen demand	FL	1565	60003001
Method SM 2120 B			
Analyte	AB	Analyte ID	Method ID
Color	FL	1605	20001803
Method SM 2130 B			
Analyte	AB	Analyte ID	Method ID
Turbidity	FL	2055	20002408
Method SM 2310 B (4a)			
Analyte	AB	Analyte ID	Method ID
Acidity, as CaCO ₃	FL	1500	20002806
Method SM 2320 B			
Analyte	AB	Analyte ID	Method ID



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Matrix: Non Potable Water

Alkalinity as CaCO ₃	FL	1505	20003003
Method SM 2340 B			
Analyte	AB	Analyte ID	Method ID
Total hardness as CaCO ₃	FL	1755	20003401
Method SM 2510 B			
Analyte	AB	Analyte ID	Method ID
Conductivity	FL	1610	20003809
Method SM 2540 B			
Analyte	AB	Analyte ID	Method ID
Residue-total	FL	1950	20004608
Method SM 2540 C			
Analyte	AB	Analyte ID	Method ID
Residue-filterable (TDS)	FL	1955	20004404
Method SM 2540 D			
Analyte	AB	Analyte ID	Method ID
Residue-nonfilterable (TSS)	FL	1960	20004802
Method SM 2540 F			
Analyte	AB	Analyte ID	Method ID
Residue-settleable	FL	1965	20005009
Method SM 3500 Cr D			
Analyte	AB	Analyte ID	Method ID
Chromium VI	FL	1045	20009001
Method SM 4500 Cl- E			
Analyte	AB	Analyte ID	Method ID
Chloride	FL	1575	20019209
Method SM 4500 CN G			
Analyte	AB	Analyte ID	Method ID
Amenable cyanide	FL	1510	20021607
Method SM 4500 F- C			
Analyte	AB	Analyte ID	Method ID
Fluoride	FL	1730	20012800



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Matrix: *Non Potable Water*

Method	Analyte	AB	Analyte ID	Method ID
SM 4500 H+ B	pH	FL	1900	20016404
SM 4500 NH3 H	Ammonia as N	FL	1515	20023409
SM 4500 NO3 F	Nitrate-nitrite	FL	1820	20024402
	Nitrite as N	FL	1840	20024402
SM 4500 P E	Orthophosphate as P	FL	1870	20025803
SM 4500 S E	Sulfide	FL	2005	20026408
SM 4500 SO3 B	Sulfite-SO3	FL	2015	20026806
	Biochemical oxygen demand	FL	1530	20027401
SM 5210 B	Carbonaceous BOD, CBOD	FL	1555	20027401
	Total organic carbon	FL	2040	20028006
SM 5310 B	Surfactants - MBAS	FL	2025	20029009
	Total Petroleum Hydrocarbons (TPH)	FL	2050	90019208



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Matrix: *Solid & Hazardous Material*

Method EPA 1010			
Analyte	AB	Analyte ID	Method ID
Ignitability	FL	1780	10116606
Method EPA 1311			
Analyte	AB	Analyte ID	Method ID
TCLP	FL	849	10118806
Method EPA 1312			
Analyte	AB	Analyte ID	Method ID
SPLP	FL	850	10119003
Method EPA 314.0			
Analyte	AB	Analyte ID	Method ID
Perchlorate	FL	1895	10055400
Method EPA 6010			
Analyte	AB	Analyte ID	Method ID
Aluminum	FL	1000	10155201
Antimony	FL	1005	10155201
Arsenic	FL	1010	10155201
Barium	FL	1015	10155201
Beryllium	FL	1020	10155201
Boron	FL	1025	10155201
Cadmium	FL	1030	10155201
Calcium	FL	1035	10155201
Chromium	FL	1040	10155201
Cobalt	FL	1050	10155201
Copper	FL	1055	10155201
Iron	FL	1070	10155201
Lead	FL	1075	10155201
Magnesium	FL	1085	10155201
Manganese	FL	1090	10155201
Molybdenum	FL	1100	10155201
Nickel	FL	1105	10155201



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Matrix: Solid & Hazardous Material

Potassium	FL	1125	10155201
Selenium	FL	1140	10155201
Silver	FL	1150	10155201
Sodium	FL	1155	10155201
Strontium	FL	1160	10155201
Thallium	FL	1165	10155201
Tin	FL	1175	10155201
Titanium	FL	1180	10155201
Vanadium	FL	1185	10155201
Zinc	FL	1190	10155201

Method EPA 6020

Analyte	AB	Analyte ID	Method ID
Aluminum	FL	1000	10156204
Antimony	FL	1005	10156204
Arsenic	FL	1010	10156204
Barium	FL	1015	10156204
Beryllium	FL	1020	10156204
Boron	FL	1025	10156204
Cadmium	FL	1030	10156204
Calcium	FL	1035	10156204
Chromium	FL	1040	10156204
Cobalt	FL	1050	10156204
Copper	FL	1055	10156204
Iron	FL	1070	10156204
Lead	FL	1075	10156204
Magnesium	FL	1085	10156204
Manganese	FL	1090	10156204
Molybdenum	FL	1100	10156204
Nickel	FL	1105	10156204
Potassium	FL	1125	10156204
Selenium	FL	1140	10156204



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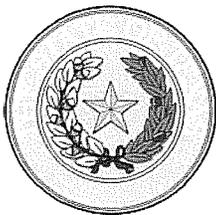
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Matrix: *Solid & Hazardous Material*

Silver	FL	1150	10156204
Sodium	FL	1155	10156204
Strontium	FL	1160	10156204
Thallium	FL	1165	10156204
Vanadium	FL	1185	10156204
Zinc	FL	1190	10156204
 Method EPA 7196			
Analyte	AB	Analyte ID	Method ID
Chromium VI	FL	1045	10162206
 Method EPA 7470			
Analyte	AB	Analyte ID	Method ID
Mercury	FL	1095	10165603
 Method EPA 7471			
Analyte	AB	Analyte ID	Method ID
Mercury	FL	1095	10166004
 Method EPA 8015			
Analyte	AB	Analyte ID	Method ID
Diesel range organics (DRO)	FL	9369	10173203
Gasoline range organics (GRO)	FL	9408	10173203
 Method EPA 8081			
Analyte	AB	Analyte ID	Method ID
4,4'-DDD	FL	7355	10178402
4,4'-DDE	FL	7360	10178402
4,4'-DDT	FL	7365	10178402
Aldrin	FL	7025	10178402
alpha-BHC (alpha-Hexachlorocyclohexane)	FL	7110	10178402
alpha-Chlordane	FL	7240	10178402
beta-BHC (beta-Hexachlorocyclohexane)	FL	7115	10178402
Chlordane (tech.)	FL	7250	10178402
delta-BHC (delta-Hexachlorocyclohexane)	FL	7105	10178402
Dieldrin	FL	7470	10178402



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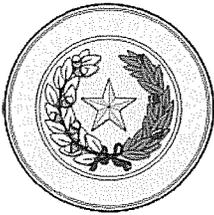
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Matrix: Solid & Hazardous Material

Endosulfan I	FL	7510	10178402
Endosulfan II	FL	7515	10178402
Endosulfan sulfate	FL	7520	10178402
Endrin	FL	7540	10178402
Endrin aldehyde	FL	7530	10178402
Endrin ketone	FL	7535	10178402
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	FL	7120	10178402
gamma-Chlordane	FL	7245	10178402
Heptachlor	FL	7685	10178402
Heptachlor epoxide	FL	7690	10178402
Methoxychlor	FL	7810	10178402
Toxaphene (Chlorinated camphene)	FL	8250	10178402

Method EPA 8082

Analyte	AB	Analyte ID	Method ID
2,2', 3,3',4,4'-Hexachlorobiphenyl	FL	9020	10179007
2,2', 3,4,4', 5,5'-Heptachlorobiphenyl	FL	9070	10179007
2,2', 3,4,4', 5'-Hexachlorobiphenyl	FL	9025	10179007
2,2', 3,5'-Tetrachlorobiphenyl	FL	8945	10179007
2,2', 4,5,5'-Pentachlorobiphenyl	FL	8980	10179007
2,2', 4,5'-Tetrachlorobiphenyl	FL	8950	10179007
2,2', 5,5'-Tetrachlorobiphenyl	FL	8955	10179007
2,2', 5-Trichlorobiphenyl	FL	8930	10179007
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	FL	9095	10179007
2,2',3,3',4,4',5,6-Octachlorobiphenyl	FL	9103	10179007
2,2',3,4,4',5,6'-Heptachlorobiphenyl	FL	9136	10179007
2,2',3,4',5,5',6-Heptachlorobiphenyl	FL	10318	10179007
2,2',3,4,5'-Pentachlorobiphenyl	FL	8975	10179007
2,2',4,4',5-Pentachlorobiphenyl	FL	9175	10179007
2,3', 4,4'-Tetrachlorobiphenyl	FL	8960	10179007
2,3,3',4,4',5,5'-Heptachlorobiphenyl	FL	9085	10179007
2,3,3',4,4',5-Hexachlorobiphenyl	FL	9050	10179007



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Matrix: *Solid & Hazardous Material*

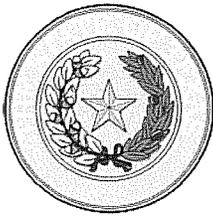
2,3,3',4,4'-Pentachlorobiphenyl	FL	8985	10179007
2,3',4,4',5,5'-Hexachlorobiphenyl	FL	9055	10179007
2,3',4,4',5-Pentachlorobiphenyl	FL	8995	10179007
2,3,4,4',5-Pentachlorobiphenyl	FL	9005	10179007
2,4,4'-Trichlorobiphenyl	FL	9252	10179007
2,4'-Dichlorobiphenyl	FL	9256	10179201
Aroclor-1016 (PCB-1016)	FL	8880	10179007
Aroclor-1221 (PCB-1221)	FL	8885	10179007
Aroclor-1232 (PCB-1232)	FL	8890	10179007
Aroclor-1242 (PCB-1242)	FL	8895	10179007
Aroclor-1248 (PCB-1248)	FL	8900	10179007
Aroclor-1254 (PCB-1254)	FL	8905	10179007
Aroclor-1260 (PCB-1260)	FL	8910	10179007
PCBs	FL	8870	10179007

Method EPA 8151

Analyte	AB	Analyte ID	Method ID
2,4,5-T	FL	8655	10183003
2,4-D	FL	8545	10183003
2,4-DB	FL	8560	10183003
Dalapon	FL	8555	10183003
Dicamba	FL	8595	10183003
Dichloroprop (Dichlorprop)	FL	8605	10183003
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	FL	8620	10183003
MCPA	FL	7775	10183003
MCPP	FL	7780	10183003
Silvex (2,4,5-TP)	FL	8650	10183003

Method EPA 8260

Analyte	AB	Analyte ID	Method ID
1,1,1,2-Tetrachloroethane	FL	5105	10184404
1,1,1-Trichloroethane	FL	5160	10184404
1,1,2,2-Tetrachloroethane	FL	5110	10184404



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Expiration Date: 10/31/2010
Issue Date: 11/1/2009

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Matrix: *Solid & Hazardous Material*

1,1,2-Trichloroethane	FL	5165	10184404
1,1-Dichloroethane	FL	4630	10184404
1,1-Dichloroethylene (1,1-Dichloroethene)	FL	4640	10184404
1,1-Dichloropropene	FL	4670	10184404
1,2,3-Trichlorobenzene	FL	5150	10184404
1,2,3-Trichloropropane	FL	5180	10184404
1,2,4-Trichlorobenzene	FL	5155	10184404
1,2,4-Trimethylbenzene	FL	5210	10184404
1,2-Dibromo-3-chloropropane (DBCP)	FL	4570	10184404
1,2-Dibromoethane (EDB, Ethylene dibromide)	FL	4585	10184404
1,2-Dichlorobenzene	FL	4610	10184404
1,2-Dichloroethane	FL	4635	10184404
1,2-Dichloropropane	FL	4655	10184404
1,3,5-Trimethylbenzene	FL	5215	10184404
1,3-Dichlorobenzene	FL	4615	10184404
1,3-Dichloropropane	FL	4660	10184404
1,4-Dichlorobenzene	FL	4620	10184404
1,4-Dioxane (1,4- Diethyleneoxide)	FL	4735	10184404
2,2-Dichloropropane	FL	4665	10184404
2-Butanone (Methyl ethyl ketone, MEK)	FL	4410	10184404
2-Chloroethyl vinyl ether	FL	4500	10184404
2-Chlorotoluene	FL	4535	10184404
2-Hexanone	FL	4860	10184404
4-Chlorotoluene	FL	4540	10184404
4-Isopropyltoluene	FL	4915	10184404
4-Methyl-2-pentanone (MIBK)	FL	4995	10184404
Acetone	FL	4315	10184404
Acetonitrile	FL	4320	10184404
Acrolein (Propenal)	FL	4325	10184404
Acrylonitrile	FL	4340	10184404



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Matrix: Solid & Hazardous Material

Allyl chloride (3-Chloropropene)	FL	4355	10184404
Benzene	FL	4375	10184404
Bromobenzene	FL	4385	10184404
Bromochloromethane	FL	4390	10184404
Bromodichloromethane	FL	4395	10184404
Bromoform	FL	4400	10184404
Bromomethane (Methyl bromide)	FL	4950	10184404
Carbon disulfide	FL	4450	10184404
Carbon tetrachloride	FL	4455	10184404
Chlorobenzene	FL	4475	10184404
Chloroethane	FL	4485	10184404
Chloroform	FL	4505	10184404
Chloromethane (Methyl chloride)	FL	4960	10184404
Chloroprene	FL	4525	10184404
cis-1,2-Dichloroethylene	FL	4645	10184404
cis-1,3-Dichloropropylene	FL	4680	10184404
Dibromochloromethane	FL	4575	10184404
Dibromomethane	FL	4595	10184404
Dichlorodifluoromethane	FL	4625	10184404
Diethyl ether	FL	4725	10184404
Ethyl methacrylate	FL	4810	10184404
Ethylbenzene	FL	4765	10184404
Hexachlorobutadiene	FL	4835	10184404
Iodomethane (Methyl iodide)	FL	4870	10184404
Isobutyl alcohol (2-Methyl-1-propanol)	FL	4875	10184404
Isopropylbenzene	FL	4900	10184404
Methacrylonitrile	FL	4925	10184404
Methyl methacrylate	FL	4990	10184404
Methyl tert-butyl ether (MTBE)	FL	5000	10184404
Methylene chloride	FL	4975	10184404



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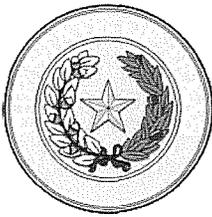
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Matrix: Solid & Hazardous Material

Naphthalene	FL	5005	10184404
n-Butylbenzene	FL	4435	10184404
n-Propylbenzene	FL	5090	10184404
Pentachloroethane	FL	5035	10184404
Propionitrile (Ethyl cyanide)	FL	5080	10184404
sec-Butylbenzene	FL	4440	10184404
Styrene	FL	5100	10184404
tert-Butylbenzene	FL	4445	10184404
Tetrachloroethylene (Perchloroethylene)	FL	5115	10184404
Toluene	FL	5140	10184404
trans-1,2-Dichloroethylene	FL	4700	10184404
trans-1,3-Dichloropropylene	FL	4685	10184404
trans-1,4-Dichloro-2-butene	FL	4605	10184404
Trichloroethene (Trichloroethylene)	FL	5170	10184404
Trichlorotrifluoroethane	FL	5185	10184404
Vinyl acetate	FL	5225	10184404
Vinyl chloride	FL	5235	10184404
Xylene (total)	FL	5260	10184404

Method EPA 8270

Analyte	AB	Analyte ID	Method ID
1,2,4,5-Tetrachlorobenzene	FL	6715	10185203
1,2,4-Trichlorobenzene	FL	5155	10185203
1,2-Dichlorobenzene	FL	4610	10185203
1,2-Diphenylhydrazine	FL	6220	10185203
1,3,5-Trinitrobenzene (1,3,5-TNB)	FL	6885	10185203
1,3-Dichlorobenzene	FL	4615	10185203
1,3-Dinitrobenzene (1,3-DNB)	FL	6160	10185203
1,4-Dichlorobenzene	FL	4620	10185203
1,4-Naphthoquinone	FL	6420	10185203
1,4-Phenylenediamine	FL	6630	10185203
1-Naphthylamine	FL	6425	10185203



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Matrix: *Solid & Hazardous Material*

2,3,4,6-Tetrachlorophenol	FL	6735	10185203
2,4,5-Trichlorophenol	FL	6835	10185203
2,4,6-Trichlorophenol	FL	6840	10185203
2,4-Dichlorophenol	FL	6000	10185203
2,4-Dimethylphenol	FL	6130	10185203
2,4-Dinitrophenol	FL	6175	10185203
2,4-Dinitrotoluene (2,4-DNT)	FL	6185	10185203
2,6-Dichlorophenol	FL	6005	10185203
2,6-Dinitrotoluene (2,6-DNT)	FL	6190	10185203
2-Acetylaminofluorene	FL	5515	10185203
2-Chloronaphthalene	FL	5795	10185203
2-Chlorophenol	FL	5800	10185203
2-Methyl-4,6-dinitrophenol	FL	6360	10185203
2-Methylnaphthalene	FL	6385	10185203
2-Methylphenol (o-Cresol)	FL	6400	10185203
2-Naphthylamine	FL	6430	10185203
2-Nitroaniline	FL	6460	10185203
2-Nitrophenol	FL	6490	10185203
2-Picoline (2-Methylpyridine)	FL	5050	10185203
3,3'-Dichlorobenzidine	FL	5945	10185203
3,3'-Dimethylbenzidine	FL	6120	10185203
3-Methylcholanthrene	FL	6355	10185203
3-Nitroaniline	FL	6465	10185203
4-Aminobiphenyl	FL	5540	10185203
4-Bromophenyl phenyl ether	FL	5660	10185203
4-Chloro-3-methylphenol	FL	5700	10185203
4-Chloroaniline	FL	5745	10185203
4-Chlorophenyl phenylether	FL	5825	10185203
4-Dimethyl aminoazobenzene	FL	6105	10185203
4-Methylphenol (p-Cresol)	FL	6410	10185203



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Matrix: Solid & Hazardous Material

4-Nitroaniline	FL	6470	10185203
4-Nitrophenol	FL	6500	10185203
5-Nitro-o-toluidine	FL	6570	10185203
7,12-Dimethylbenz(a) anthracene	FL	6115	10185203
a-a-Dimethylphenethylamine	FL	6125	10185203
Acenaphthene	FL	5500	10185203
Acenaphthylene	FL	5505	10185203
Acetophenone	FL	5510	10185203
Aniline	FL	5545	10185203
Anthracene	FL	5555	10185203
Aramite	FL	5560	10185203
Benzidine	FL	5595	10185203
Benzo(a)anthracene	FL	5575	10185203
Benzo(a)pyrene	FL	5580	10185203
Benzo(b)fluoranthene	FL	5585	10185203
Benzo(g,h,i)perylene	FL	5590	10185203
Benzo(k)fluoranthene	FL	5600	10185203
Benzoic acid	FL	5610	10185203
Benzyl alcohol	FL	5630	10185203
bis(2-Chloroethoxy)methane	FL	5760	10185203
bis(2-Chloroethyl) ether	FL	5765	10185203
bis(2-Chloroisopropyl) ether	FL	5780	10185203
bis(2-Ethylhexyl) phthalate (DEHP)	FL	6255	10185203
Butyl benzyl phthalate	FL	5670	10185203
Carbazole	FL	5680	10185203
Chlorobenzilate	FL	7260	10185203
Chrysene	FL	5855	10185203
Diallate	FL	7405	10185203
Dibenz(a,h) anthracene	FL	5895	10185203
Dibenzofuran	FL	5905	10185203



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Matrix: Solid & Hazardous Material

Diethyl phthalate	FL	6070	10185203
Dimethoate	FL	7475	10185203
Dimethyl phthalate	FL	6135	10185203
Di-n-butyl phthalate	FL	5925	10185203
Di-n-octyl phthalate	FL	6200	10185203
Ethyl methanesulfonate	FL	6260	10185203
Famphur	FL	7580	10185203
Fluoranthene	FL	6265	10185203
Fluorene	FL	6270	10185203
Hexachlorobenzene	FL	6275	10185203
Hexachlorobutadiene	FL	4835	10185203
Hexachlorocyclopentadiene	FL	6285	10185203
Hexachloroethane	FL	4840	10185203
Hexachloropropene	FL	6295	10185203
Indeno(1,2,3-cd) pyrene	FL	6315	10185203
Isodrin	FL	7725	10185203
Isophorone	FL	6320	10185203
Isosafrole	FL	6325	10185203
Methapyrilene	FL	6345	10185203
Methyl methanesulfonate	FL	6375	10185203
Naphthalene	FL	5005	10185203
Nitrobenzene	FL	5015	10185203
Nitroquinoline-1-oxide	FL	6515	10185203
n-Nitrosodiethylamine	FL	6525	10185203
n-Nitrosodimethylamine	FL	6530	10185203
n-Nitroso-di-n-butylamine	FL	5025	10185203
n-Nitrosodi-n-propylamine	FL	6545	10185203
n-Nitrosodiphenylamine	FL	6535	10185203
n-Nitrosomethylethylamine	FL	6550	10185203
n-Nitrosomorpholine	FL	6555	10185203



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Matrix: Solid & Hazardous Material

n-Nitrosopiperidine	FL	6560	10185203
n-Nitrosopyrrolidine	FL	6565	10185203
o,o,o-Triethyl phosphorothioate	FL	8290	10185203
o-Toluidine	FL	5145	10185203
Parathion, methyl (Methyl parathion)	FL	7825	10185203
Pentachlorobenzene	FL	6590	10185203
Pentachloronitrobenzene	FL	6600	10185203
Pentachlorophenol	FL	6605	10185203
Phenacetin	FL	6610	10185203
Phenanthrene	FL	6615	10185203
Phenol	FL	6625	10185203
Phorate	FL	7985	10185203
Pronamide (Kerb)	FL	6650	10185203
Pyrene	FL	6665	10185203
Pyridine	FL	5095	10185203
Safrole	FL	6685	10185203
Sulfotepp	FL	8155	10185203
Thionazin (Zinophos)	FL	8235	10185203

Method EPA 8330

Analyte	AB	Analyte ID	Method ID
1,3,5-Trinitrobenzene (1,3,5-TNB)	FL	6885	10189807
1,3-Dinitrobenzene (1,3-DNB)	FL	6160	10189807
2,4,6-Trinitrotoluene (2,4,6-TNT)	FL	9651	10189807
2,4-Dinitrotoluene (2,4-DNT)	FL	6185	10189807
2,6-Dinitrotoluene (2,6-DNT)	FL	6190	10189807
2-Amino-4,6-dinitrotoluene (2-am-dnt)	FL	9303	10189807
2-Nitrotoluene	FL	9507	10189807
3-Nitrotoluene	FL	9510	10189807
4-Amino-2,6-dinitrotoluene (4-am-dnt)	FL	9306	10189807
4-Nitrotoluene	FL	9513	10189807
Nitrobenzene	FL	5015	10189807



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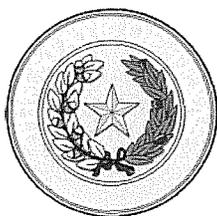
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Matrix: Solid & Hazardous Material

Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	FL	9522	10189807
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	FL	9432	10189807
Tetryl (methyl-2,4,6-trinitrophenyl nitramine)	FL	9633	10189807
Method EPA 8332			
Analyte	AB	Analyte ID	Method ID
Nitroglycerin	FL	6485	10190406
Method EPA 9012			
Analyte	AB	Analyte ID	Method ID
Amenable cyanide	FL	1510	10193201
Total Cyanide	FL	1635	10193201
Method EPA 9038			
Analyte	AB	Analyte ID	Method ID
Sulfate	FL	2000	10196608
Method EPA 9040			
Analyte	AB	Analyte ID	Method ID
pH	FL	1900	10196802
Method EPA 9045			
Analyte	AB	Analyte ID	Method ID
pH	FL	1900	10197805
Method EPA 9056			
Analyte	AB	Analyte ID	Method ID
Bromide	FL	1540	10199209
Chloride	FL	1575	10199209
Nitrate as N	FL	1810	10199209
Nitrate-nitrite	FL	1820	10199209
Nitrite as N	FL	1840	10199209
Orthophosphate as P	FL	1870	10199209
Sulfate	FL	2000	10199209
Method EPA 9060			
Analyte	AB	Analyte ID	Method ID
Total organic carbon	FL	2040	10200201



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Matrix: *Solid & Hazardous Material*

Method	Analyte	AB	Analyte ID	Method ID
Method EPA 9065				
	Total phenolics	FL	1905	10200405
Method EPA 9070				
	n-Hexane Extractable Material (O&G)	FL	1803	10201000
	Silica Gel Treated n-Hexane Extractable Material	FL	10220	10201000
Method EPA 9071				
	n-Hexane Extractable Material (O&G)	FL	1803	10201204
	Silica Gel Treated n-Hexane Extractable Material	FL	10220	10201204
Method EPA 9095				
	Paint Filter Test	FL	10312	10204009
Method EPA 9251				
	Chloride	FL	1575	10207406
Method TCEQ 1005				
	Total Petroleum Hydrocarbons (TPH)	FL	2050	90019208

TITLE: EQUIPMENT MAINTENANCE

Prepared By: Julie Ricardi Date: 8-96

Approved By:

Group Supervisor: _____ Date: _____

Operations Manager: Joh C. Burton Date: 12/15/00

QA Officer: Deborah J. Nadeau Date: 12-13-00

General Manager: JCBurton For D. McGrath Date: 12/15/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Added requirements for recording maintenance in appropriate log. Added Archon maintenance. Modified Lockat maintenance. Attached equipment list.	JN	12-13-00	12-13-00
02	Removed references to General Manager. Removed furnace (GFAA) maintenance from Figure 1 and added ICP-MS. Updated equipment list.	JN	10-22-02	10-22-02
03	Changed supervisor to department manager. Added maintenance for the ASE and general lab maintenance. Updated equipment list.	HRC	06-03-04	06-03-04
04	Added new equipment list.	LAD	01-28-05	01-28-05
05	Added Horizon SPE Extractor & HPLC maintenance to Table 1. Added TOC analyzer, IC & miscellaneous instrument maintenance to Table 2. Updated equipment list.	JN	03-06	03-06

TITLE: EQUIPMENT MAINTENANCE

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **CA-101-08**, titled **EQUIPMENT MAINTENANCE**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **CA-101-08**, titled **EQUIPMENT MAINTENANCE**.

Recipient: _____ Date: _____

TITLE: EQUIPMENT MAINTENANCE

1.0 SCOPE AND APPLICATION

The consistent generation of high quality analytical results requires properly operating analytical equipment. Accordingly, Katahdin Analytical Services, Inc. has an equipment maintenance program, which prescribes the necessary maintenance measures required and also schedules the frequency of conduct and documentation of those measures.

The purpose of this SOP is to describe the routine scheduled maintenance procedures employed for analytical and support equipment and to discuss non-scheduled maintenance of analytical and support equipment utilized by Katahdin Analytical personnel. It is applicable to all equipment utilized by laboratory personnel for analytical and support work. The procedures outlined in this SOP provide general guidance for equipment maintenance and documentation. The appropriate equipment manufacturer's manual(s) and specific analytical SOPs must be available and may provide specific maintenance and cleaning procedures.

1.1 Definitions

1.2 Responsibilities

It is the responsibility of each technical employee to carry out and document the required routine equipment maintenance at the required frequency for the instrumentation that they use.

It is the responsibility of each Department Manager to ensure that maintenance performed in-house is conducted properly by experienced personnel familiar with the equipment. On a routine basis, the department manager or qualified designee reviews equipment maintenance activities and recordkeeping requirements. Deficiencies are brought to the attention of the analyst who is responsible for an unacceptable procedure or entry, and any corrections are made by the appropriate analyst.

The Quality Assurance Officer is responsible for conducting periodic audits of the equipment maintenance program and documentation in each analytical area. Maintenance logs and contracted vendor maintenance records are reviewed for completeness and frequency of maintenance. Deficiencies are immediately brought to the attention of the Supervisor who is responsible for informing his/her staff and initiating the necessary corrective or improved actions in his/her analytical area.

2.0 SUMMARY OF METHOD

Not applicable.

3.0 INTERFERENCES

Refer to specific instrument manufacturer's operating manuals.

TITLE: EQUIPMENT MAINTENANCE

4.0 APPARATUS AND MATERIALS

Not applicable.

5.0 REAGENTS

Not applicable.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Not applicable.

7.0 PROCEDURES

The laboratory is equipped with all equipment needed for analysis and support activities. All equipment is maintained in working condition through a schedule of preventive maintenance and/or outside service contracts or appointments as needed. All maintenance, including internal maintenance and outside service calls, is documented. Any out-of-service equipment is marked as such until it can be repaired and shown by calibration or testing to perform correctly.. In cases where outside equipment is rented or borrowed, any records necessary to demonstrate equipment calibration or maintenance will be requested from the provider.

All equipment must be maintained so that its performance is acceptable for its intended use. For example, an instrument that does not have sufficient sensitivity should not be used for low-level analysis or a balance not verified above 100 grams should not be used to weigh items over 100 grams.

7.1 ROUTINE PREVENTIVE MAINTENANCE

Preventive maintenance (PM) may be conducted by qualified Katahdin personnel or may be performed under contract by authorized service technicians. Between service visits, instruments having PM service contracts are cleaned and maintained according to manufacturer's specifications by experienced Katahdin personnel. Instruments not under service contracts are cleaned and maintained by experienced laboratory personnel according to manufacturer's specifications and based on professional experience. The frequency of maintenance performed depends on the equipment. Preventive maintenance in each analytical section at Katahdin Analytical is scheduled, conducted and documented on a regular basis by the analytical section staff. Many maintenance needs (e.g., accidental breakage, part failure) are not satisfied by scheduled maintenance and are performed as needed. This would apply to routine daily instrument maintenance as well as non-scheduled instrument maintenance (e.g.,

TITLE: EQUIPMENT MAINTENANCE

replacement of GC injection port liners, replacement of volatiles Tekmar traps, etc.). Maintenance procedures indicated in Tables 1 and 2 are documented in appropriate logbooks maintained in each analytical area. Included in the documentation are general remarks describing the maintenance operations performed, the date maintenance was performed, and the signature or initials of the individuals who completed the maintenance. If a specific instrument problem prompted the need for maintenance, the problem and its resolution are documented along with the description of maintenance performed. Specific requirements for maintenance logs are described in Section 7.3.

7.2 PREVENTIVE MAINTENANCE SCHEDULES

Tables 1 and 2 summarize the routine preventive maintenance procedures and schedules followed within Katahdin Analytical for major laboratory instrumentation. These items are required to be documented in the applicable instrument or maintenance log. Refer to Section 7.3 for specific requirements for maintenance log documentation.

7.3 UNSCHEDULED MAINTENANCE & TROUBLESHOOTING

In addition to preventive maintenance, analysts are often required to perform unscheduled maintenance due to sporadic instrument problems. The instrument problems could be obvious, such as those cause by "nasty" samples, or not so obvious. The problem could be more mechanical, such as an autosampler jamming or a needle clogging, or the problems could be more technical such as failing QC for no apparent reason. In all cases, analysts shall proceed through a series of troubleshooting steps to investigate the problem. Analysts should consult the manufacturer's guidelines for troubleshooting certain equipment problems. Changing only one parameter and/or part at a time is critical to ascertain which change may improve or correct a problem.

All troubleshooting steps and outcomes must be recorded in the applicable instrument maintenance logbook for future reference and guidance. The steps taken, which correct the problem should be noted as such. If it is determined that a problem with the equipment exists, and that the problem has had an impact on the quality of past completed data, an immediate Corrective Action Report (CAR) must be initiated. A thorough investigation of all impacted data must be completed and corrective action must be implemented. Refer to the current revision of Katahdin SOP, QA-803, Laboratory Quality Assurance Self Inspection System, for further details.

7.4 MAINTENANCE LOG (or equivalent) REQUIREMENTS

Maintenance must be recorded in either the instrument run log or in a separate maintenance log. For equipment that does not have a run log or does not necessitate a separate maintenance log (i.e. little maintenance required), any maintenance may be recorded in the appropriate logbook (i.e. balance maintenance recorded in the balance calibration log). Attachment A provides a list of analytical equipment for which maintenance activities are recorded. Balances are not listed, although maintenance activities are recorded for these.

TITLE: EQUIPMENT MAINTENANCE

- 7.4.1 Maintenance logs (or equivalent) shall include the type of equipment and the analytical group/analysis for which it is designated, the manufacturer's model number and serial number, as well as any laboratory-assigned identifier (e.g., GC08, MS2, etc.). Each lab-assigned identifier must be unique and each instrument must have only one lab-assigned identifier. As new instrumentation is received, the date acquired, condition when received (new, used, reconditioned), and date put into use should be recorded in the new instrument's maintenance log.
- 7.4.2 Each page of the maintenance logbook (or equivalent) must clearly indicate the type of equipment and laboratory-assigned identifier in the header information. Maintenance entries shall include the date and signature or initials of the individual performing the maintenance as well as a brief explanation of the maintenance performed.
- 7.4.3 If maintenance is conducted as the result of a suspected or known instrument problem, the maintenance record must clearly describe the following:
- Statement of the Problem: brief description of the problem (i.e. calibrations are failing)
 - Cause Analysis: what might be causing the problem. This could be something that occurred just prior to the problem (i.e. electrical outage, analysis of nasty samples) or something that is unknown.
 - Corrective Action Plan: What might fix the problem (i.e replacing liners, tubing or column that could be "damaged" from a nasty sample)
 - CAP Implementation: an indication of when the maintenance is performed
 - Demonstration of CAP Implementation: an indication of whether the problem was resolved and the date when return to control was noted. Document any changes resulting from the CAP.
- 7.4.4 On a routine basis the Department Manager or qualified designee shall review equipment maintenance activities and adherence to recordkeeping requirements. Deficiencies are brought to the attention of the analyst who was responsible for the unacceptable procedure or entry, and any corrections are made by the appropriate analyst. Documentation of the maintenance log review can be made by initialing and dating each page of the log or by indicating "reviewed by", initials and date on the front cover of the maintenance log (or equivalent).
- 7.4.5 On a periodic basis the laboratory QA Officer shall conduct audits of the equipment maintenance program and documentation in the analytical areas. Maintenance logs and contracted vendor maintenance records are reviewed for completeness and adherence to this SOP. Deficiencies are immediately brought to the attention of the Department Manager who is responsible for informing his/her staff and initiating the necessary corrective actions in his/her analytical area.

TITLE: EQUIPMENT MAINTENANCE

7.5 SERVICE CONTRACTS

Katahdin will maintain service contracts for major instrumentation as needed. The need for a service contract will depend on factors such as the cost, the estimated down time, the availability of back-up instrumentation, and the availability of skilled analysts to perform non-routine maintenance. The decision to purchase a service contract is made by the management team of the Vice President, the President, the Operations Manager and the Department Managers.

In cases where a service contract is not in place, and a problem with an instrument or piece of support equipment cannot be corrected in-house, Katahdin will use reputable service companies to perform the repair and/or troubleshooting work. Relationships are maintained with several companies to perform these services.

Occasionally, equipment may need to be sent out to the service company for repair. All equipment must be packaged in large enough boxes to leave room for at least four inches of bubble wrap on every side, including the top and bottom. Any small movable parts must be removed and wrapped separately. When available, a manufacturer's box with appropriate Styrofoam inserts should be used. All equipment returning to the laboratory must be checked and recalibrated to be sure all instrument capabilities and conditions have been maintained.

A preventive maintenance contract is in place for all support equipment and thermometers for cleaning, calibrating to manufacturer's specifications and verifying against NIST traceable sources. The support equipment includes all pH meters, conductivity meters, turbidity meters, balances, spectrophotometers, autoclaves, ovens, and incubators. Field equipment meters are included in this annual verification. All equipment supported under this contract are labeled by the outside calibration company with the last calibration date, and when the next calibration is due. All additional comments of the outside calibration are maintained in files with the QA Officer.

Records of all service contract visits, third party services and preventive maintenance services are maintained to document support equipment performance. All equipment not meeting manufacturer's specifications or those required by the test are removed from service until appropriate corrective actions can be taken.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Not applicable.

9.0 METHOD PERFORMANCE

Not applicable.

TITLE: EQUIPMENT MAINTENANCE

10.0 APPLICABLE DOCUMENTS/REFERENCES

Applicable equipment manufacturer's operating manuals.

LIST OF TABLES & ATTACHMENTS

TABLE 1 – ROUTINE MAINTENANCE SCHEDULE FOR ORGANICS INSTRUMENTATION
TABLE 2 - ROUTINE MAINTENANCE SCHEDULE FOR INORGANICS INSTRUMENTATION
TABLE 3 - ROUTINE MAINTENANCE SCHEDULE FOR GENERAL LABORATORY EQUIPMENT
ATTACHMENT A – ANALYTICAL EQUIPMENT

TITLE: EQUIPMENT MAINTENANCE

TABLE 1

ROUTINE MAINTENANCE SCHEDULE FOR ORGANICS INSTRUMENTATION

GC/MS

Regularly performed maintenance includes, but is not limited to, the following for GC/MS instrumentation:

- ◆ Check to ensure the pressure on the primary regulator never runs below 100 psi
- ◆ Check to ensure the gas supply is sufficient for the day's activity, and delivery pressures are set as described in the SOP.
- ◆ Change septa weekly or as needed.
- ◆ Replace/cut GC column as needed.
- ◆ Replace GC injector glass liner weekly or as needed.
- ◆ Replace glass jet splitter as needed.
- ◆ Replace pump oil as needed.
- ◆ Change gas line dryers as needed.
- ◆ Replace electron multiplier as needed.

GC

Regularly performed maintenance includes, but is not limited to, the following for GC instrumentation:

- ◆ Check to ensure the pressure on the primary regulator never runs below 100 psi.
- ◆ Check to ensure the gas supply is sufficient for the day's activity, and delivery pressures are set as described in the SOP.
- ◆ Change septa weekly or as needed.
- ◆ Replace/cut GC column as needed.
- ◆ Replace GC injector glass liner as needed.
- ◆ Change O₂/moisture traps as needed
- ◆ Clean/replace GC detector as needed.

TITLE: EQUIPMENT MAINTENANCE

TABLE 1, cont'd

ROUTINE MAINTENANCE SCHEDULE FOR ORGANICS INSTRUMENTATION

Purge and Trap Sample Concentrator

Regularly performed maintenance includes, but is not limited to, the following:

- ◆ Check to ensure the gas supply is sufficient for the day's activity and delivery pressures are set as described in the SOP.
- ◆ Replace trap as needed.
- ◆ Decontaminate the system after running high concentration samples or as required by blank analysis.
- ◆ Check system for leaks when problem suspected.

Archons

Regularly performed maintenance includes, but is not limited to, the following:

- ◆ Visually inspect sampler for corrosion or significant water seepage past plunger.
- ◆ Check system pressure (denotes leaks).
- ◆ Check for sufficient standard materials in standard vials.
- ◆ Recalibrate x,y,z components, i.e. robotic arm, as needed.

Accelerated Solvent Extractor (ASE)

Regularly performed maintenance includes, but is not limited to, the following:

- ◆ Check for leaks at the pump solvent reservoir, valves and other components.
- ◆ Inspect needle alignment of source needle.
- ◆ Check alignment of autoseal arms.
- ◆ Change peek seals and o-rings on cell caps after about 50 extractions per cell.
- ◆ Inspect cell edges for nicks and gouges on cell body.
- ◆ Inspect stainless steel frits and sonicate in solvent if needed.

TITLE: EQUIPMENT MAINTENANCE

TABLE 1, cont'd

ROUTINE MAINTENANCE SCHEDULE FOR ORGANICS INSTRUMENTATION

Horizon SPE Automated Extractor System

Regularly performed maintenance includes, but is not limited to, the following:

- ◆ Check and clean sensors with a KIMWipe.
- ◆ Change sensors as needed.
- ◆ Purge system with solvent before use and after use.
- ◆ Clean system with hot water by running method 15 after samples are analyzed and before purging system.

HPLC

Regularly performed maintenance includes, but is not limited to, the following for HPLC instrumentation:

- ◆ Check and sonicate pump valves as needed.
- ◆ Backflush column as needed.
- ◆ Replace analytical column or guard column as needed.
- ◆ Filter and replace solvent with every use.
- ◆ Replace the UV lamp as needed.
- ◆ Perform a column wash at the end of each run
- ◆ Change needle and/or needle seat as needed

GPC

Regularly performed maintenance includes, but is not limited to, the following for GPC instrumentation:

- ◆ Fill solvent reservoir as needed.
- ◆ Empty waste container as needed.

TITLE: EQUIPMENT MAINTENANCE

TABLE 2

ROUTINE MAINTENANCE SCHEDULE FOR INORGANICS INSTRUMENTATION

ICP/MS

Regularly performed maintenance includes, but is not limited to, the following for ICP instrumentation:

- ◆ Clean torch assembly and spray chamber when discolored.
- ◆ Clean nebulizer as needed.
- ◆ Check to ensure that the argon supply is sufficient for the day's activity, and that delivery pressures are set as described in the SOP. Change argon tanks as necessary.
- ◆ Replace peristaltic pump tubing when it becomes stretched or develops flat spots.
- ◆ Check coolant water level weekly; replenish as necessary.
- ◆ Check rinse solution level daily; replenish as necessary.
- ◆ Check waste container level daily; empty as necessary.
- ◆ Check cleanliness of instrument air filters weekly; clean or replace as necessary.
- ◆ Check instrument computer date and time at the start of each day; correct as necessary.
- ◆ Check condition of vacuum pump oil weekly, replace as necessary.
- ◆ Clean sampling and skimmer cones every 2-4 weeks.
- ◆ Clean extraction lenses monthly, or as needed.
- ◆ Clean Einzel lenses every 3-6 months.

ICP

Regularly performed maintenance includes, but is not limited to, the following for ICP instrumentation:

- ◆ Clean torch assembly and spray chamber when discolored.
- ◆ Clean nebulizer as needed.

TITLE: EQUIPMENT MAINTENANCE

TABLE 2, cont'd

ROUTINE MAINTENANCE SCHEDULE FOR INORGANICS INSTRUMENTATION

ICP, cont'd

- ◆ Check to ensure that the argon supply is sufficient for the day's activity, and that delivery pressures are set as described in the SOP. Change argon tanks as necessary.
- ◆ Replace peristaltic pump tubing when it becomes stretched or develops flat spots.
- ◆ Check coolant water level weekly; replenish as necessary.
- ◆ Check rinse solution level daily; replenish as necessary.
- ◆ Check waste container level daily; empty as necessary.
- ◆ Check instrument computer date and time at the start of each day; correct as necessary.

Mercury Analyzer

Regularly performed maintenance includes, but is not limited to, the following:

- ◆ Replace drying tube as necessary
- ◆ Replace peristaltic pump tubing when it becomes stretched or develops flat spots.
- ◆ Replace mercury lamp as necessary.
- ◆ Clean optical cell quarterly or as needed.
- ◆ Clean liquid/gas separator when it becomes cloudy. Replace as needed.
- ◆ Check waste container level before each use; empty as necessary.
- ◆ Check exhaust system integrity before each use; correct as necessary.
- ◆ Check instrument computer date and time at the start of each day; correct as necessary.

TITLE: EQUIPMENT MAINTENANCE

TABLE 2, cont'd

ROUTINE MAINTENANCE SCHEDULE FOR INORGANICS INSTRUMENTATION

Lachat Autoanalyzer

Regularly performed maintenance includes, but is not limited to, the following:

- ◆ Check pump tubing; replace as needed.
- ◆ Clean interference filter with Kimwipe.
- ◆ Check reagent levels and expiration dates; refill or replace as needed.
- ◆ Rinse manifolds with water after analysis.
- ◆ Check manifold board surfaces; clean as needed by running under tap water.
- ◆ Check supplies and reagents; order as needed
- ◆ Check for leaks
- ◆ Check autosampler and autosampler trays; clean and/or lubricate as needed.
- ◆ Check fittings and o-rings on boards; replace as needed.
- ◆ Check peristaltic pump rollers; clean and lubricate as needed.
- ◆ Inspect manifold tubing for kinks and/or stains; replace as needed.
- ◆ Inspect valve flares; clean or replace as needed.
- ◆ Inspect reagent and waste lines; replace as needed.
- ◆ Check flow cell; clean as needed with Kimwipe.

Konelab AutoAnalyzer

Regularly performed maintenance includes, but is not limited to, the following:

- ◆ Rinse and refill distilled water container weekly.

TITLE: EQUIPMENT MAINTENANCE

TABLE 2, cont'd

ROUTINE MAINTENANCE SCHEDULE FOR INORGANICS INSTRUMENTATION

Konelab AutoAnalyzer, cont'd

- ◆ Check cleanness of segments weekly.
- ◆ Wash reagent tubes monthly.
- ◆ Change lamp as needed.
- ◆ Change diluent and wash tubes as needed.
- ◆ Change mixing paddles as needed.
- ◆ Change syringes as needed.
- ◆ Change dispensing needles as needed.
- ◆ Change drain and waste tubes as needed.
- ◆ Check used cuvettes and waste daily.

TOC Combustion Analyzer

Regularly performed maintenance includes, but is not limited to, the following:

- ◆ Check level of dilution water, drain vessel water, humidifier water, autosampler rinse water, and phosphoric acid vessel and fill as needed.
- ◆ Replace oxygen cylinder as needed.

IC

Regularly performed maintenance includes, but is not limited to, the following for IC instrumentation:

- ◆ Check regenerate pump tubing and replace as needed.
- ◆ Clean or regenerate column as needed.

TITLE: EQUIPMENT MAINTENANCE

TABLE 2, cont'd

ROUTINE MAINTENANCE SCHEDULE FOR INORGANICS INSTRUMENTATION

IC, cont'd

- ◆ Replace analytical column or guard column as needed.
- ◆ Change suppressor as needed.

Miscellaneous Support Instrumentation (including field testing equipment)

Regularly performed maintenance includes, but is not limited to, the following:

- ◆ Replace spectrophotometer lamps as needed.
- ◆ Inspect DO probe membrane for tears and check for air bubbles under the membrane.
- ◆ Replace the DO probe membrane cap and electrolyte solution as needed.
- ◆ Clean pH electrode as needed.
- ◆ Change the autotitrator filling solution as needed.
- ◆ Clean, check, calibrate to manufacturers' specifications all pH, DO, conductivity, and turbidity meters annually (minimum); clean, check, calibrate to manufacturers' specifications all spectrophotometers and balances annually. All calibrations must be NIST traceable.

TITLE: EQUIPMENT MAINTENANCE

TABLE 3

ROUTINE MAINTENANCE SCHEDULE FOR GENERAL LABORATORY EQUIPMENT

General Laboratory Areas

- ◆ Clean and calibrate balances annually (minimum).
- ◆ Check balance calibration each day of use.
- ◆ Clean balance pan prior to each use.
- ◆ Calibrate automatic pipettes with each use.
- ◆ Calibrate spirit thermometers yearly against an NIST traceable thermometer; calibrate digital thermometers quarterly.
- ◆ Record refrigerator, freezer, incubator and oven temperatures each day, when appropriate.
- ◆ General housekeeping: keep counter tops, hoods, and floors clean.
- ◆ Check airflow in hoods once a week.

TITLE: EQUIPMENT MAINTENANCE

ATTACHMENT A
ANALYTICAL EQUIPMENT LIST



ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Hewlett Packard 5973 GC/MS with EPC, Archon Autosampler with foam sensor option and EST ENCON Evolution Purge and Trap Concentrator with heated purge capability	2008	5030/8260, 5035/8260, 624, Current versions of CLP SOWs (OLM, OLC, & SOM) (VOA), & 524.2	D
Hewlett Packard 5972 GC/MS with EPC, Centurion Autosampler with foam sensor option and EST ENCON Purge and Trap Concentrator	2006	5030/8260, 624, Current versions of CLP SOWs (OLM & SOM) (VOA)	T
Hewlett Packard 5972 GC/MS with EPC; Tekmar LSC-3100 Purge and Trap concentrator; Archon autosampler capable of low soils per Method 5035.	1998	5030/8260, 5035/8260, 624, Current versions of CLP SOWs (OLM, OLC, & SOM) (VOA), & 524.2	S
Hewlett Packard 5972 GC/MS with EPC; Tekmar LSC - 3000 purge and trap concentrator; Archon autosampler capable of low soils per Method 5035.	1996	5030/8260, 5035/8260, 624, Current versions of CLP SOWs (OLM, OLC, & SOM) (VOA), & 524.2	Z
Hewlett Packard 5972 GC/MS with EPC; Encon Purge and Trap concentrator; Archon autosampler capable of low soils per method 5035.	1995	5030/8260, 5035/8260, 624, Current versions of CLP SOWs (OLM, OLC, & SOM) (VOA), & 524.2	M
Hewlett Packard 5972 GC/MS with EPC; Tekmar LSC-3000 Purge and Trap concentrator; Archon autosampler capable of low soils per Method 5035.	1993	5030/8260, 5035/8260, 624, Current versions of CLP SOWs (OLM, OLC, & SOM) (VOA), & 524.2	F
Hewlett Packard 5973 GC/MS with EPC and Model 6890 GC and Agilent 7683 autosampler.	2008	8270, 625 & Current versions of CLP SOWS (OLM, OLC, & SOM) (SVOA)	G
Hewlett Packard 5973 GC/MS with EPC and Model 6890 GC and Agilent 7683 autosampler.	2006	8270, 625 & Current versions of CLP SOWS (OLM, OLC, & SOM) (SVOA)	R

TITLE: EQUIPMENT MAINTENANCE

ATTACHMENT A
ANALYTICAL EQUIPMENT LIST, cont'd



ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Hewlett Packard 5973 GC/MS with EPC and Model 6890 GC and Agilent 7673 autosampler.	1999/ 2001	8270, 625 & Current versions of CLP SOWS (OLM, OLC, & SOM) (SVOA)	U
Hewlett Packard Model 5890 gas chromatograph with flame ionization detector; Tekmar ALS 2016 autosampler; Tekmar LSC 2000 purge and trap.	1991	8015 MOD., GRO	GC04
Hewlett Packard Model 5890 gas chromatograph with a flameionization detector; Agilent Technologies G1888 Network Headspace Analyzer.	1991/ 2005	Methane, Ethane and Ethene	GC05
Hewlett Packard Model 5890 gas chromatograph with EPC and dual electron capture detectors(ECD); Hewlett Packard Model 7673 autosampler.	1993	8081, 8082, 608	GC06
Agilent Model 6890 gas chromatograph with dual EPC injection ports and micro electron capture detectors; Agilent Model 7683 autosampler	2000	504, 608, 8011, 8081, 8082 (including congeners), 8151, current versions of CLP SOWs (OLM, OLC, & SOM) (P/P)	GC07
Agilent Model 6890 gas chromatograph with one EPC injection ports and micro electron capture detectors; Agilent Model 7683 autosampler	2009	608, 8081, 8082 (including congeners), current versions of CLP SOWs (OLM, OLC, & SOM) (P/P)	GC08
Hewlett Packard Model 5890 gas chromatograph with flame ionization detector and photo ionization detector; Tekmar ALS 2016 autosampler; Tekmar LSC 3000 purge and trap.	1988	8015 MOD., MAVPH, GRO	GC09

TITLE: EQUIPMENT MAINTENANCE

ATTACHMENT A

ANALYTICAL EQUIPMENT LIST, cont'd



ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Hewlett Packard Model 5890 gas chromatograph with EPC and dual flame ionization detectors; Hewlett Packard Model 7673 autosampler.	1993/ 1996	8015 MOD., MAEPH, DRO	GC10
Hewlett Packard Model 5890 gas chromatograph with flame ionization and nitrogen-phosphorous detectors; Hewlett Packard Model 7673 autosampler.	1992/ 1996	ALCOHOLS, DMF, GLYCOLS	GC11
Hewlett Packard Model 5890 gas chromatograph with EPC and dual flame ionization detectors; Hewlett Packard Model 7673 autosampler.	1987	8015 MOD., MAEPH, DRO	GC12
Agilent Model 6890 gas chromatograph with one EPC injection ports and micro electron capture detectors; Agilent Model 7683 autosampler	2008	608, 8081, 8082 (including congeners), current versions of CLP SOWs (OLM, OLC, & SOM) (P/P)	GC01
Hewlett Packard series 1100 HPLC with Quaternary pump, Multiwavelength detector and autosampler	2008	8330, 8332	HPLC03
J2 Scientific AccuPrep MPS™ GPC with internal UV detection	2009	GPC-3640 Current versions of CLP SOWs (OLM, OLC, & SOM)	GPC01
Horizon SPE-DEX 4790 Automated Extractor System equipped with 4 extractors.	2001/ 2005	3535 method development	Horizon #1
Horizon SPE-DEX 3000 Automated Extractor System equipped with 3 extractors.	2006	1664, 9070, 9071	Horizon #2
Thermo ICAP 6500 ICP Emission Spectrometer with autosampler.	2006	6010, 200.7, ILM05.4	I

TITLE: EQUIPMENT MAINTENANCE

ATTACHMENT A

ANALYTICAL EQUIPMENT LIST, cont'd



ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Agilent 7500a ICP-MS with autosampler.	2007	6020, 200.8, ILM05.4	J
CETAC M-6100 Automated Mercury Analyzer with Autosampler	2004	7470/7471, 245.1, 245.5, ILM05.4	H
Tekran Series 2600 automated mercury analyzer with gold amalgam preconcentration and atomic fluorescence detector; Model 2620 autosampler.	2000	Ultra-trace level mercury (1631)	G
CPI ModBlock™ Metals Digestion Unit – Two 48 Place Units.	2000	Metals Aqueous Digestions	Digestion Unit #1 Digestion Unit #2
LACHAT Quickchem 8000 Continuum Series – Automated Ion Analyzer and autosampler.	2000	Various	WC1
Shimadzu TOC-V Combustion Analyzer, PC Controlled, High Sensitivity, Auto-Aqueous TOC Autosampler w/ 40 mL vials; Model SSM-5000A Solid Sample Module.	2002	TOC	WC2
Waters 717 Plus Autosampler, Waters 431 conductivity detector, Spectra System P4000 pump.	1991/ 2004	Ion chromatography – Various Anions	See "Key Instrumentation" column for identification
		Ion Chromatography - Perchlorate	See "Key Instrumentation" column for identification
Scanning Fluorescence Detector	1997	Out of Service	NA
10 position Lab Crest Cyanide Midi-Distillation system.	1998	Cyanide	WC3

TITLE: EQUIPMENT MAINTENANCE

ATTACHMENT A

ANALYTICAL EQUIPMENT LIST, cont'd



ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Spectronic 401 spectrophotometer.	2000	Various	WC4
HACH Turbidimeter, Model 2100A.	1993	Turbidity	WC6
Accumet pH/Conductivity Meter, Model 20.	1998	Various	WC8
Mettler DL25 Autotitrator & Mettler ST20A sample changer.	1993	Alkalinity	WC9
YSI Dissolved Oxygen Meter.	1990	Dissolved Oxygen & Biochemical Oxygen Demand	WC10
Konelab 20 Multi-Wavelength Photometric Analyzer.	2003	Various	Konelab #1
Dionex Accelerated Solvent Extractor ASE 200.	2003	3545	ASE #1
Waters HPLC system with 600E multisolvent delivery system, 717 plus autosampler, Spectra System UV200 detector	2003/2004	Food Testing	HPLC01
Thermo Spectronic Genesys 10uv spectrophotometer	2005	Food Testing	Food #1
DryVap Concentrator System 5000	2005	GC and GC/MS Extractables	DryVap #1

TITLE: **BALANCE CALIBRATION**

Prepared By: Julie Ricardi Date: 12.9.06

Approved By:

Group Supervisor: _____ Date: _____

Operations Manager: John C. Burtow Date: 12.15.00

QA Officer: Deborah J. Madreau Date: 12.13.06

General Manager: JCBurtow For D. McGrath Date: 12/15/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes. Add section about use of ASTM class 1 vs. class 2 weights.	DN	12.13.06	12.13.06
02	Modified to describe current new calibration practices from 7.6 to 7.11 - unique logbooks/acceptance criteria, not doing additive weight calibration. Updated Figure 1. Removed G.U refs.	DN	10.22.02	10.22.02
03	Changed supervisor to department manager. Added Denver Instruments balance. Added weight verification procedures (8.3) and figure 2.	HRC	06.03.04	06.03.04
04	Added new balance in food lab.	DN	01.18.05	01.18.05
05	Changed outside calibration to annually versus semi-annually.	DN	04.06	04.06

TITLE: BALANCE CALIBRATION

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **CA-102-06**, titled **BALANCE CALIBRATION**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **CA-102-06**, titled **BALANCE CALIBRATION**.

Recipient: _____ Date: _____

TITLE: BALANCE CALIBRATION

1.0 SCOPE AND APPLICATION

Accurately operating balances are of paramount importance for the majority of analyses conducted by Katahdin Analytical Services. Specific balance calibration verification procedures must be followed to ensure that laboratory balances are accurate for the intended use and that their accuracy is documented. The purpose of this SOP is to describe procedures utilized to verify calibration of balances in the Katahdin laboratory.

In addition to performing calibration verification on laboratory balances on each day of use, specific analyses (e.g., gravimetric procedures) require verification of balance calibration prior to each use of a balance. Requirements for analysis-specific balance calibration verification procedures are described in the applicable analytical SOPs.

This SOP is applicable to all weighing balances utilized in the laboratory and involves all staff members whose laboratory activities require regular use of balances.

1.1 Definitions

ASTM Class 1 Weights – Those weights meeting the applicable standards set forth in American Society For Testing and Materials, Method E 617-97, 1997 revision.

1.2 Responsibilities

All laboratory personnel whose work requires use of balances are responsible for performing balance calibration verification and documentation procedures as described in the procedures section of this SOP.

It is the responsibility of laboratory managers to ensure that technicians under their supervision are properly trained in balance calibration and documentation and to perform monthly review of balance calibration logbooks for accuracy and completeness.

It is the responsibility of the QA department to periodically review the balance calibration documentation in each laboratory, to arrange for annual preventative maintenance checks on balances by outside vendors, and to arrange for accuracy checks on the working weights every year and on the ASTM Class 1 (formerly designated as 'Class S') weights every five years.

1.3 Safety

NA

1.4 Pollution Prevention/Waste Disposal

NA

TITLE: BALANCE CALIBRATION

2.0 SUMMARY OF METHOD

Not applicable.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

4.1 ASTM Class 1 (formerly designated as Class S) weights that bracket the calibration range of the balance in use

4.2 Working weights verified by ASTM Class I weights

4.3 Balances deemed appropriate for the analytical application and put into service at Katahdin Analytical Services, Inc., including the following (or other comparable balances):

Sartorius A200S/FW Analytical Balance
O'Haus Scout Pro Balance
Mettler PJ400 Balance
Sartorius Basic B310S Balance
O'Haus Galaxy 400 Balance

O'Haus TS400S Balance
Mettler AE200 Balance
Denver Instruments XP-1500
Denver Instruments PK-401

NOTE: The QA department is responsible for maintaining an active list of in-service balances.

5.0 REAGENTS

Not applicable.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Not applicable.

TITLE: BALANCE CALIBRATION

7.0 PROCEDURES

BALANCE CALIBRATION VERIFICATION

The following steps are to be performed on each balance by the analyst preparing to use the balance for sample preparation, standards preparation, or analysis. The steps must be performed prior to the first use of the balance on any given day (prior to each use for gravimetric analyses).

Each department will be equipped with working weights (when supplies permit) to be stored in a desiccator. The working weights are used for daily calibration so as to prevent damage to the ASTM Class 1 weights. The ASTM Class 1 weights will be used to verify the weights of the working weights annually. The QAO will verify the weights and adjust the true values of the working weights as necessary. Refer to section 8 for procedures. The QAO will arrange for outside verification (NIST traceable) of the class 1 weights every five years.

- 7.1 Remove the working weights assigned to your laboratory from the desiccator. Do not touch the weights with your hands. These weights should be handled only with the plastic forceps provided in the weights' case or with vinyl gloves or KIM wipes. The small weights (i.e., ≤ 2 g) are often difficult to pick up with the forceps so use extra care when handling these weights.
- 7.2 Clean the balance pan and surrounding areas.
- 7.3 Check balance level (if applicable to the balance). If the air bubble is not centered in the circle of the level indicator, relevel the balance using the leveling screws. If necessary, ask the QA Officer or your Manager for assistance.
- 7.4 Verify that the balance draft shield is in place or that the balance is free of air currents that could cause balance drift. Draft shields (housing with sliding doors) are permanently attached to the Sartorius A200S/FW analytical balance. The sliding doors should be closed when reading this balance. The Sartorius Basic 310S is equipped with a removable cylindrical shield, and the Mettler PJ400 is used inside a free-standing draft shield. Balances that are not equipped with draft shields must be carefully monitored to ensure they are free of air currents when in use.
- 7.5 Verify balance zero. Readjust or retare if necessary using procedures appropriate for the balance.
- 7.6 Each balance is equipped with a unique Balance Calibration Logbook (Refer to Figure 1 for an example logbook page). Each logbook contains the following information:
 - 7.6.1 Balance Serial #
 - 7.6.2 Location of the balance
 - 7.6.3 Balance Manufacturer's Tolerance – this indicates the \pm error of each reading

TITLE: BALANCE CALIBRATION

7.6.4 Lowest weight to be weighed on the balance – this indicates the lowest weight that can be measured while still meeting the acceptance criteria for accuracy. Do not weigh anything on the balance below this weight. If lower weights need to be measured, another balance that meets the required calibration criteria at a lower weight range must be used.

7.6.5 Date and initials of analyst

7.6.6 True weights to be weighed and acceptance criteria for each weight

7.6.7 Indication of pass/fail, comments and corrective actions

Using the weights indicated in each logbook, perform the balance calibration procedures listed below. The weights have been chosen as appropriate to the range of experimental measurements currently expected. Therefore, it is critical to verify all the weights listed. Weights, other than those indicated in the logbooks, may be used, as appropriate, but must be noted. Record all appropriate information in the logbook.

If you are uncertain of proper operation procedures for any balances, ask the QAO or your Manager for assistance before attempting to use a balance or correct a problem on your own. Note that for some Wet Chemistry analyses, balance calibration verification procedures are also documented in the applicable parameter-specific logbook. Consult with the appropriate Manager or analytical SOP for further information on the applicable parameters. Any unused space on each page of the logbook must be lined-out, initialed and dated.

7.7 Place weight A on the balance pan. Record the measured weight next to the appropriate true weight in the logbook. Remove the weight and be sure the balance returns to zero.

7.8 Place weight B on the balance pan. Record the measured weight next to the appropriate true weight in the logbook. Remove the weight and be sure the balance returns to zero.

7.9 Continue with the remainder of the required weights. Record each measured weight next to its appropriate true weight. Each time, be sure the balance returns to zero before placing the next weight on the pan.

Note: Different variations on the above procedure may be implemented if appropriate for the type of weighing being performed. All weighings must be recorded in the logbook.

7.10 Based on the criteria listed next to each weight in the logbook, determine whether the measured weight passes or fails and indicate this in the appropriate space. If acceptance criteria are not met, do not continue to use the balance. Label the balance as "Out of Service" (initials and date) and immediately notify the QAO or your manager. Document corrective actions taken, maintenance performed, and return to control in the logbook before resuming use of the balance.

TITLE: BALANCE CALIBRATION

- 7.11 Return the weights to their case, then return the case to the desiccator. Always store the weights in the desiccator. If a weight becomes damaged or is missing, you **must** notify your manager or the QAO.
 - 7.12 Preventive maintenance is performed contractually on all laboratory balances annually. The preventive maintenance includes cleaning and calibrating each balance, as well as checking internal mechanisms for wear and proper alignment. A written report is prepared by the contract vendor; a copy of the report is maintained in the laboratory quality assurance files. Non-routine maintenance is generally performed by the contract vendor, except for re-leveling, auto-calibration, weight alignment, and cleaning the pans and exposed areas of the balance which are to be performed by laboratory staff as needed. Staff members uncertain of proper balance operation must ask the QAO or their Manager for assistance before attempting to use a balance or correct a problem on their own.
-

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 Acceptance Criteria for Balances

Acceptance criteria for each balance is listed within each unique Balance Verification Logbook (Refer to Figure 1 for an example).

Acceptance criteria are designed to be appropriate for the intended use of the balance. For example, $\pm 5\%$ is sufficient for weighing initial sample weights but is not appropriate for gravimetric analyses. Likewise, weighed standards must be measured on the analytical balances having tighter acceptance criteria, since $\pm 5\%$ is generally not accurate enough for standards preparation.

8.2 Corrective Action For Non-Conformance

8.2.1 DISCONTINUE BALANCE USE IF ACCEPTANCE CRITERIA ARE EXCEEDED AND IMMEDIATELY NOTIFY THE QAO OR YOUR MANAGER.

8.2.2 Document in the balance calibration logbook that the balance failed to meet acceptance criteria and is out of service as of the date of the failed calibration. Initial and date logbook entry. Place an "Out of Service" tag on the balance, including initials and date.

8.2.3 Contact your manager or the QA Officer who will schedule laboratory or vendor maintenance, as appropriate. Document maintenance performed by Katahdin staff in the "Comments" section of the balance calibration verification log.

8.2.4 After appropriate service has been performed, verify balance calibration (Steps 7.1 - 7.11). Document "return to control" in the balance calibration verification log and remove the "Out of Service" tag from the balance.

TITLE: BALANCE CALIBRATION

8.3 Weight Verification Procedures

- 8.3.1 The QAO will arrange for outside verification (NIST traceable) of the class 1 weights every five years. The ASTM Class 1 weights will be used to verify the weights of the working weights annually. The QAO will verify the weights and adjust the true values of the working weights as necessary. The following procedure must be used.
- 8.3.2 Use the Sartorius A200S/FW balance in the Wet Chemistry balance room for weights of 100 gms or less. Use the Denver Instruments XP-1500 balance in the food lab for weights greater than 100 gms.
- 8.3.3 Obtain the working weights to be verified from the appropriate lab and the Class I weights from the QAO's office.
- 8.3.4 Follow the balance calibration procedures described in steps 7.7 and 7.8 starting with a working weight, followed by the same weight from the Class I set. Repeat each weight four more times (five weighings total for each weight) making sure that the balance door is shut and that the balance returns to zero between each reading.
- 8.3.5 Continue verifying the other working weights against the same weight from the Class I set. If there is not a corresponding Class I weight, several weights may have to be added together to obtain the same weight as the working weight.
- 8.3.6 Record all weights in the Excel spreadsheet for weight verification (Figure 2).
- 8.3.7 Calculate the differences between the working weights and the Class I weights. The working weights are considered acceptable for use if their differences are less than the acceptance range for the balance they will be used on. Acceptance ranges are listed in Figure 2.

9.0 METHOD PERFORMANCE

Not applicable.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Manufacturers' literature

LIST OF FIGURES

FIGURE 1- EXAMPLE OF BALANCE CALIBRATION LOGBOOK PAGE
FIGURE 2 – EXAMPLE WEIGHT VERIFICATION SPREADSHEET

TITLE: BALANCE CALIBRATION

FIGURE 1

EXAMPLE OF BALANCE CALIBRATION LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES, INC.
BALANCE CALIBRATION VERIFICATION LOG

Balance ID: Denver Instruments PK-401 Balance **Serial #:** 04TCF012 **Location:** GC/MS Laboratory
Manufacturer's Tolerance: ± 100 mg **Lowest Weight to be weighed on this balance:** 1000mg or 1g

Corrective Action: If acceptance criteria is not met, do not continue to use the balance. Label the balance as "Out of Service" (initials/date) and notify your supervisor or the QAO. Document corrective actions taken and return to control before using balance. Document any maintenance performed in the "Comments" section of the balance calibration verification log.

Date	Analyst Initials	Weight(g) True Value	Weight(g) Measured Value	Acceptance Criteria (g)	Pass/Fail ?	Comments – Corrective Actions and/or Maintenance
		2.0		± 0.1		
		10.0		± 0.5		
		50.0		± 1.0		
		100.0		± 2.0		
		2.0		± 0.1		
		10.0		± 0.5		
		50.0		± 1.0		
		100.0		± 2.0		
		2.0		± 0.1		
		10.0		± 0.5		
		50.0		± 1.0		
		100.0		± 2.0		
		2.0		± 0.1		
		10.0		± 0.5		
		50.0		± 1.0		
		100.0		± 2.0		

TITLE: BALANCE CALIBRATION

FIGURE 2
 EXAMPLE WEIGHT VERIFICATION SPREADSHEET

WORKING WEIGHTS VERIFICATION

ASTM CLASS 1 WEIGHT SET ID - 10026/70084/70085 INITIALS- DN
 WORKING WEIGHT SET ID - Katahdin #1/66388 DATE- 11/5/2004

WEIGHT	MEASUREMENTS		ACCURACY (%)		PRECISION (%)		P&A ASTM VS WORKING		
	ASTM	WORKING	ASTM	WORKING	ASTM	WORKING	DIFFERENCE	CRITERIA(*)	ACCEPT?
30 G	30.00	30.00							
	30.00	30.05							
	30.00	30.00							
	30.05	30.00							
	30.01	30.01	100.0333	100.0333	0.0224	0.0224	0.0000	0.20	Yes
100 G	100.05	100.05							
	100.05	100.05							
	100.05	100.05							
	100.05	100.05							
	100.05	100.05	100.0500	100.0500	0.0000	0.0000	0.0000	0.30	Yes
500 G	500.35	500.35							
	500.30	500.30							
	500.30	500.30							
	500.30	500.35							
	500.30	500.30	100.0620	100.0640	0.0224	0.0274	0.0100	0.50	Yes

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

Prepared By: GC/MS Group Date: 2/97

Approved By:

Group Supervisor: J. Haley Date: 01/20/01

Operations Manager: J. C. Burton Date: 1/15/01

QA Officer: Rutah J. Nadeau Date: 1.23.01

General Manager: Debra F. Huffman Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03 8260B	Format changes added pollution prevention, changes to calibration section, new limits, added instrument. Other minor changes throughout.	DN	1.23.01	1.23.01
04 8260B	Revised sections 7.5.3.1, 7.5.5, 7.7.1, 7.8.2 + Table 2 to comply with South Carolina. Added NH ₃ oxygenates to calibration.	DN	5.23.01	5.23.01
05 8260B	Updated VOA calibration standard mixes. Added statistical limits for LCS/MS/MSD recoveries and the updated corrective actions.	DN	5.21.02	5.21.02
06 8260B	Reorganization of sections 4, 5, 6 and 7, and Tables and Figures. Added definitions and information for the new data processing system.	MRC	05.03.04	05.03.04
07 8260B	Minor changes rewording of sect. 7.6.3 preservation of calcareous soils	LAD	02.03.05	02.03.05

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-202-10**, titled **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-202-10**, titled **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**.

Recipient: _____ Date: _____

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel to prepare and analyze aqueous and solid matrix samples for purgeable organics by GC/MS in accordance with SW-846 Method 8260, current revision.

This SOP will consolidate all aspects of the analyses in one working document, to be revised as necessary, for the purposes of consistency in data quality.

1.1 Definitions

VOC: Volatile Organic Compounds

VOA: Volatile Organic Analysis

ANALYTICAL BATCH: 20 or fewer samples that are analyzed together with the same method sequence and the same lots of reagents and with the handling practices common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): A quality control sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Laboratory reagent grade water is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing a mid point standard. The calibration check verifies that instrument conditions are sufficiently similar to those at initial calibration.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

INDEPENDANT CALIBRATION STANDARD: A solution prepared from a stock standard solution independent of the standard that is used to calibrate the instrument. This is prepared as an LCS and analyzed after the calibration before any sample analysis.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control and to measure the degree of accuracy of the determination.

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MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions containing target analytes are added to a sample matrix prior to sample extraction, in the case of soils, and/or analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the spiked analytes. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single analyte or mix of certified standards, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition as well as extraction and chromatography characteristics, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate. Surrogates provide an indication of the accuracy for the analytical determination in a discrete sample matrix.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

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 volatile organics by the current revision of EPA Method 8260. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training and Demonstration of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of volatile organics by Method 8260 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate logbook. Any deviations from the test or irregularities with the samples should also be recorded

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in the lab logbook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 **Safety**

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 **Pollution Prevention/Waste Disposal**

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, partially-filled VOA vials and sample jars are returned to the appropriate refrigerators to be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

Sample aliquots used for analysis are disposed of in accordance with SOP SD-903 and the Katahdin Hazardous Waste Management Plan and Safety Manual. The soil samples must be decanted and the soil fraction disposed of separately in compliance with Katahdin's disposal policies.

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There are three general types of waste generated while performing the 8260 method. The "K" waste is a combination of water, sample aliquot (post analysis), as well as internal and surrogate standards. "K" waste is generated when preparing QC, during sample analysis, and procedural cleanup. There are "K" satellites attached to each GC/MS instrument as well as an additional satellite located adjacent to the VOA sample preparation bench. "O" waste consists of methanol (as well as trace amounts of volatile analytes) and is generated when standard preparation syringes are rinsed three times with methanol. The "O" waste stream satellite is located inside the fume hood. Organic soil waste stream "I" consists of any solid left over from sample preparation and/or analysis and is located inside the fume hood. All satellites listed above are stored in a secondary container and are located in the Volatile Organics Laboratory room 111.

2.0 SUMMARY OF METHOD

The general methodology involves purging aqueous and soil samples with helium, an inert gas, for a set period of time to efficiently transfer purgeable organics to the gaseous phase. Soil samples with higher contaminant levels are extracted with methanol prior to the helium purge. These volatile organics are then retained on a cooled trap (commercially available trap suitable for the methodology) before heating causes desorption into a gas chromatograph for compound separation. Detection occurs with an electron impact ionization mass spectrometer.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of VOCs is analyzed immediately after a sample containing high concentrations of VOCs. During initial data review, all analyses are evaluated for potential carryover. Any samples that have suspected carryover are reanalyzed. GC/MS policy is to reanalyze a sample with positive detects greater than the Practical Quantitation Limit (PQL) that has been run immediately after a sample with the same positive detects over the upper limit of the calibration. Typically 2 or 3 rinsing blanks are analyzed at the end of a sequence. Samples are not analyzed on the instrument until a blank with no detects above PQL can be obtained. If the lines are determined to be contaminated, then the entire Tekmar or Archon must be backflushed with warm methanol and water.

4.0 APPARATUS AND MATERIALS

- 4.1 GC: Hewlett Packard 6890 & 5890
- 4.2 Mass Spectrometers (MS): HP5973, HP5972 and HP5970

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- 4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.
- 4.4 Columns: RTX-VMS, 40 meter, 0.18 mm ID or equivalent.
- 4.5 Purge and Traps: Archon 5100, Tekmar 2016 and Centurion auto samplers, and Tekmar 2000, 3000 and Encon concentrators.
- 4.6 Purge tubes: 5 mL fritted and 25 mL fritted purge vessels and 40 mL VOA vials for soil analysis.
- 4.7 Hamilton Gastight syringes: 2.00 uL to 25.00 mL.
- 4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.
- 4.9 Data System: The Target software is used for processing data and generating forms.

5.0 REAGENTS

- 5.1 Purge and trap grade methanol
- 5.2 Organic-free Laboratory reagent grade water: Siemens, Poland Spring, or equivalent. This water may need to be purged with nitrogen to eliminate organic contaminants such as Methylene chloride and Chloroform, which are commonly found at ambient levels in the laboratory.
- 5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 "Standard Preparation and Documentation".
 - 5.3.1 The expiration date for all standards is six months from date of opening the ampule with the following exceptions:

Volatile gases expire within 2 weeks of opening ampule (gases are dichlorodifluoromethane, chloromethane, bromomethane, vinyl chloride, chloroethane, and trichlorofluoromethane).

New standards must be opened if degradation is observed.
 - 5.3.2 Secondary dilution standards

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5.3.2.1 Calibration Mix – Prepare a standard in purge and trap methanol containing the compounds listed below. The final concentration of each compound is 200 ug/mL (some individual analyte concentrations may vary, i.e. Ketones). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 7 days and stored in the VOA standards freezer between uses.

Acetone	Dibromochloromethane	P-Isopropyltoluene
Benzene	1,2-Dibromoethane	Methylene Chloride
Bromobenzene	Dibromomethane	4-Methyl-2-Pentanone
Bromochloromethane	1,2-Dichlorobenzene	Naphthalene
Bromodichloromethane	1,3-Dichlorobenzene	N-Propylbenzene
Bromoform	1,4-Dichlorobenzene	Styrene
Bromomethane	Dichlorodifluoromethane	1,1,1,2-Tetrachloroethane
2-Butanone	1,1-Dichloroethane	1,1,2,2-Tetrachloroethane
n-Butylbenzene	1,2-Dichloroethane	Tetrachloroethene
sec-Butylbenzene	1,1-Dichloroethene	Tetrahydrofuran
tert-Butylbenzene	cis-1,2-Dichloroethene	Toluene
Carbon Disulfide	Trans-1,2-Dichloroethene	1,2,3-Trichlorobenzene
Carbon Tetrachloride	1,2-Dichloropropane	1,2,4-Trichlorobenzene
Chlorobenzene	1,3-Dichloropropane	1,1,1-Trichloroethane
Chloroethane	2,2-Dichloropropane	1,1,2-Trichloroethane
2-Chloroethylvinyl Ether	1,1-Dichloropropene	Trichloroethene
Chloroform	Cis-1,3-Dichloropropene	Trichlorofluoromethane
Chloromethane	Trans-1,3-Dichloropropene	1,2,3-Trichloropropane
2-Chlorotoluene	Ethylbenzene	1,2,4-Trimethylbenzene
4-Chlorotoluene	Hexachlorobutadiene	Vinyl Acetate
Cyclohexane	2-Hexanone	Vinyl Chloride
1,2-Dibromo-3-Chloropropane	Idomethane	1,3,5-Trimethylbenzene
Isopropylbenzene	Methyl Tert-Butyl Ether	1-Chlorohexane

5.3.2.2 Extras mix – Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 200 ug/mL. The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

Acetonitrile	Isobutyl Alcohol
Acrolein	Methacrylonitrile
Acrylonitrile	Methylcyclohexane
Allyl Chloride	Methyl Acetate
Chloroprene	Methyl Methacrylate
Diethyl Ether	Methyl Tert-Butyl Ether
Cis-1,4-Dichloro-2-Butene	Pentachloroethane
Trans-1,4-Dichloro-2-Butene	Propionitrile
1,4-Dioxane	Tertiary-Amyl Methyl Ether
Di-Isopropyl Ether	Tertiary-Butyl Alcohol
Ethyl Methacrylate	1,3,5-Trichlorobenzene
Ethyl Tertiary-Butyl Ether	1,2,3-Trimethylbenzene
Freon-113	

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5.3.2.3 Independent Calibration Verification Standard, Laboratory Control Spike and MS/MSD Mixture - Prepare a standard as above containing the compounds listed in Table 3. The final concentration of each compound is 200 ug/mL (some individual analyte concentrations may vary, i.e. Ketones). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 7 days and stored in the VOA standards freezer between uses.

5.3.2.4 Surrogate Spiking Solution - Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 250 ug/mL or 50 ug/mL depending on which autosampler you will be using. The standard must be prepared every 14 days and stored on the Archon and/or the Centurion autosampler in a pressurized vial or in the VOA standards freezer between uses.

4-Bromofluorobenzene
1,2-Dichloroethane-D₄
Toluene-D₈
Dibromofluoromethane

5.3.2.5 Internal Standard Solution - Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 250 ug/mL or 50 ug/mL depending on which autosampler you will be using. The standard must be prepared every 14 days and stored on the Archon and/or the Centurion autosampler in a pressurized vial or in the VOA standards freezer between uses.

Pentafluorobenzene
1,4-Difluorobenzene
Chlorobenzene-D₅
1,4-Dichlorobenzene-D₄

5.3.2.6 BFB Solution - Prepare a standard as above containing 4-BFB. The final concentration is 25 ug/mL. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

5.3.2.7 See Table 4 for a complete list of standards, concentration, and vendors.

NOTE: The concentrations of standards may vary depending on the type of autosampler being used.

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6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All aqueous samples must be analyzed within 14 days from sample collection if preserved (by addition of HCl to pH <2) or within 7 days from sample collection if unpreserved. All soil/sediments must be analyzed within 14 days from sample collection. For specific projects, soil may be received in pre-weighed vials containing methanol, with an aliquot of the methanol used for analysis. For these projects, the methanol aliquot must be analyzed within 14 days from sample collection. Samples must be stored at $4\text{ C} \pm 2\text{ C}$ from the time of receipt at the lab until analysis.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition: C:\HPCHEM\1\DATA

Tune file: BFB.U

Method files: I826AXX.M (all samples and standards)

Where:

XX = the calibration number in chronological order

I = instrument ID (F,M,S,T,or Z)

A = matrix (A for water, S for soil and SB for sodium bisulfate soils)

BFB288AQ.M (waters) or BFB288SL.M (soils) (BFB tuning acquisition)

Data files for BFB: IB___.D where ___ is a number in chronological order from 000 to 999, and I is the instrument ID (F,M,S,T,or Z).

All other data files: I____.D where _____ is a number in chronological order from 0000 to 9999, and I is the instrument ID (F,M,S,T,or Z). This file also contains the Quantitation output file.

7.3 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks, or samples, the GC/MS system must be shown to meet the mass spectral ion abundance criteria for a 50 ng injection of p-Bromofluorobenzene (p-BFB), tabulated below:

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<u>Mass</u>	<u>Criteria</u>
50	15.0-40.0% of mass 95
75	30.0-60% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	less than 2.0% of mass 174
174	greater than 50.0% of mass 95
175	5.0-9.0% of mass 174
176	greater than 95.0%, but less than 101.0% of mass 174
177	5.0-9.0% of mass 176

7.3.1 The following are the GC/MS operating conditions for injection of BFB.

GC/MS type: 5970

Column: RTX-VMS, 40 meter, 0.18 mm ID
Temperatures: Injection port: 170°
 Transfer line: 150°
Source: 170°
Analyzer: 170°
Isothermal temperature: 150°
Run time: 10 minutes
Scan start time: 4 minutes
Scan parameters: not to exceed 2 sec per scan
Mass range: 35-300
Number of A/D samples: 8
GC peak threshold: 1000 counts
Threshold: 10 counts

GC/MS type: 5972 and 5973

Column: RTX-624, 40 meter, 0.18 mm I.D or RTX-VMS,
 40 meter, 0.18 mm ID.
Temperatures: Injection port: 200°
 Transfer line: 150°
 Detector: 240°
Isothermal temperature: 150°
Run time: 8 minutes
Scan start time: 3 minutes
Scan parameters: not to exceed 2 sec per scan
Mass range: 35-300
Number of A/D samples: 8
GC peak threshold: 1000 counts
Threshold: 10 counts

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The BFB solution must be analyzed once at the beginning of each 12-hour period, the time stamp of the injection of the BFB is the beginning of the 12-hour clock. All calibrations and samples must be run within the 12-hour clock as the method specifies.

When the BFB run has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The BFB run is processed using the current algorithms in the Target software.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, BFB must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument BFB tune is not in criteria.

7.4 INSTRUMENT CONFIGURATION / CALIBRATION

7.4.1 Tekmar LSC 3000/Archon 5100/ Tekmar 2016, Setup/Operation: Please refer to the Tekmar or Archon Manuals for more detailed operations for these instruments.

To begin, set the Tekmar LSC 2000/3000 to the specification listed in section 2-12 of the Archon manual. Edit method 14 as follows:

Method 14 should include:

Standby:	35°
Prepurge:	0 min
Preheat Temp:	0°
Sample Temp:	0°
Purge:	11 min
Dry purge:	2-4 min
Desorb preheat:	245°
Desorb Temp:	250°
Desorb time:	2-5 min
Dry purge:	2-4 min
Bake Time:	10 min
Bake Temp:	260°
Auto drain:	On
Bake gas by pass:	Off
Valve Temp:	120°
Line Temp:	120°
Runs per sample:	1

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The above temperature settings are for a Vocab 3000 trap, these temperatures may vary with the use of alternative traps. Temperature settings may also vary to optimize system performance.

The Archon autosampler should be set up according to the specifications in the manual. The setting of particular concern, with regards to keeping the Tekmar and Archon in coordination with each other, is the desorb time. There are several other programmable features on the Archon; the settings for this feature will depend on the sample matrix and method of analysis. Please refer to the Archon manual for more specifics on its programming features.

7.4.2 Encon/Centurion, Setup/Operation

Please refer to the Encon or Centurion manuals for more detailed operations for the instruments.

To begin, the Encon operation method should contain:

Purge Conditions: Purge Gas: Helium
 Purge Time: 11.0 ±0.1 minute
 Purge Flow Rate: approx. 24-40 mL/min
 Purge Temperature: Ambient (water)

Desorb Conditions: DesorbTemp: 250°C
 Desorb Flow rate: 15 mL/min
 Desorb Time: 2.0 ± 0.1 min
 Bake Time: 10 min
 Bake Temperature: 260°C

The above temperature settings are for a Vocab 3000 trap, these temperatures may vary with the use of alternative traps. Temperature settings may also vary to optimize system performance.

The Centurion autosampler should be set up according to the specifications in the manual.

7.4.3 Initial Calibration for Method 8260

Once the instrument has achieved BFB tuning criteria, calibration of the instrument can begin.

To determine the linearity of response, the GC/MS must be initially calibrated at six different levels.

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For aqueous calibration, target analytes and surrogate are prepared at the following concentrations; 1.0, 5.0, 20, 50, 100 and 200 ug/L. The curve is analyzed at ambient temperature.

For a soil calibration target analytes and surrogates are prepped at the following concentrations: 5.0, 10, 20, 50, 100 and 200 ug/L. The calibration standards are stirred and heated to 40°C.

The following amounts standards should be added to 100 mL of organic-free laboratory reagent grade water in order to generate a 6-point initial calibration curve:

	STD. ID	CAL. Mix 200 ug/mL	Extras Mix 200 ug/mL	Surr. Mix 250 ug/mL Archon	Surr. Mix 50 ug/mL Centurion
AQ curve only	VSTD001	0.5 uL	0.5 uL	0.4 uL	2.0 uL
	VSTD005	2.5 uL	2.5 uL	2.0 uL	10 uL
SL curve only	VSTD010	5.0 uL	5.0 uL	4.0 uL	20 uL
	VSTD020	10 uL	10 uL	8.0 uL	40 uL
CC	VSTD050	25 uL	25 uL	20 uL	100 uL
	VSTD100	50 uL	50 uL	40 uL	200 uL
	VSTD200	100 uL	100 uL	80 uL	400 uL

The internal standard is spiked by the autosampler. Due to different spike amounts separate standards are used depending on which autosampler is being used.

After analysis of the six points, the standard analyses must be quantitated and evaluated for adherence to QC criteria, as follows. Minimum requirements for method files are use of specific quantitation ions and quantitating a specific set of target compound and surrogates with a specified internal standard. These requirements are found in Tables 3 and 5.

7.4.4 Initial Calibration Criteria

The percent (%) RSD for six calibration check compounds (CCC) must be less than or equal to 30%. CCCs are 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene, and Vinyl Chloride.

A system performance check must be performed as part of initial calibration. The five system performance check compounds (SPCC) and the minimum acceptable average relative response factors (RRF) for these compounds are as follows (taken from 8260B):

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SPCC	RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

The SPCCs are used to check both the standard and instrument stability.

7.4.4.1 Linearity of Target Analytes

If the RSD of any target analyte is 15% or less using the average response factor, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15% using the average response factor, then a calibration option outlined in section 7.0 of method 8000 will need to be employed. Please note that some options may not be allowable for certain states, federal programs, or clients.

Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. For linear models, Target calculates the correlation coefficient and then squares it (r^2). This is what is reported on all Target forms. The value for r^2 must be greater than or equal to 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order (seven calibration points required) polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.99.

7.4.5 Independent Calibration Verification

Immediately following an initial calibration, an independent calibration standard must be analyzed. This standard contains all target compounds, internal standards and surrogates at a concentration of 50 ug/L and is obtained from a source independent of the initial calibration source. Please refer to section 8.1 and Table 1 for acceptance criteria and corrective action for this standard.

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For projects or clients requiring DoD QSM 4.1 all project analytes must fall between 80-120% of the true value. No samples may be run until the ICV criteria are met.

7.4.6 Calibration Verification

Once a valid initial calibration curve has been achieved, a continuing calibration standard containing all the target compounds, internal standards and surrogates at a concentration of 50 ppb must be analyzed every 12-hour clock for Method 8260, timed from the injection of BFB. The relative response factor from the 50 ppb continuing calibration check standard must be compared to the average response factor data from the initial calibration.

The EICP (extracted ion current profile) area for any of the internal standards in the calibration verification must not change by more than a factor of two (-50% to +100%) from the same level standard in the last initial calibration. The retention time for any internal standard cannot shift by more than 30 seconds from the same level standard in the last initial calibration.

For Method 8260, if the percent difference for each CCC is less than or equal to 20%, and all of the SPCCs have a relative response factor greater than or equal to those listed in Section 7.4.3, the continuing calibration is considered valid.

For projects or clients requiring DoD QSM 4.1 all project analytes must have $\pm 20\%D$.

Continuing calibration check criteria must be met before sample analysis can proceed.

7.4.7 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.

For projects or clients requiring DoD QSM 4.1, IS responses and retention time windows for QC and samples are compared to the midpoint of the most recent ICAL.

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7.5 QUALITY CONTROL SAMPLE ANALYSIS

When preparing standards in water or spiking samples with internal standards/surrogates or matrix spike solution, be sure to rinse all syringes a minimum of three times with purge and trap grade methanol between uses. Failure to do this will result in cross-contamination of samples and standards.

7.5.1 Laboratory Control Sample (LCS)

The LCS mix is prepared from a secondary source vendor (i.e. different vendor from the calibration standards). The LCS is analyzed immediately after the initial calibration curve or calibration check and prior to the method blank to minimize any analyte carryover possibilities in samples. Acceptance criteria for the LCS are outlined in Section 8.0.

To prepare the water and medium-level soil LCS, 25 uL of the LCS standard mix at 200 ug/mL are spiked into 100 mL of analyte-free laboratory reagent grade water for a final concentration of 50 ug/L. The Archon autosampler adds 1 uL of internal and 1 uL of surrogate standard to a 5 mL aliquot of this preparation for analysis. The Centurion autosampler adds 5 uL of both surrogates and internal standards to a 5 mL aliquot. To prepare the low-level soil LCS, a stir bar is added to 5 mL of the above solution in a VOA vial. The Archon unit adds an additional 10 mL of water to which the internal and surrogate standards have been added; this preparation is then heated, stirred and purged.

To prepare the water and medium-level soil LCS for analysis on the LSC 2000 / 2016 autosampler, 1.25 uL of the LCS standard mix at 200 ug/mL are spiked into 5 mL of analyte-free laboratory reagent grade water for a final concentration of 50 ug/L.

7.5.2 Method Blank Analysis

After calibration criteria have been met, a method blank must be analyzed before sample analysis can proceed. A method blank analysis must be performed once for each 12-hour calibration immediately after analysis of the calibration standard(s) and prior to sample analysis.

The aqueous method blank is a volume of analyte free laboratory reagent grade water spiked with internal and surrogate standards.

The low-level soil method blank is a volume of analyte free laboratory reagent grade water spiked with internal and surrogate standards. This method blank is analyzed using the low soil specification.

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The method blank must contain less than the Practical Quantitation Level (PQL) for all analytes of interest for the samples associated with the blank.

For projects requiring DoD QSM 4.1 no analytes may be detected >1/2 the PQL and > than the 1/10th the measured amount in any sample or 1/10th the regulatory limit, whichever is larger. Except for common laboratory contaminants which may not be detected > than the PQL.

7.5.3 Surrogate Recovery Limits

Laboratory established limits are derived for each of the surrogates. Please refer to the current revision of Katahdin Analytical Services SOP # QA-808 for further information on statistical limits. All samples including blanks, laboratory control samples, matrix spikes and client samples, must meet the statistical limits for the analysis to be considered valid. If surrogate recoveries do not meet these limits, reanalysis must occur to confirm matrix interference.

7.5.4 Internal Standard Area Recoveries / Retention Times.

The internal standard responses and retention times in the method blank must be evaluated immediately after or during data acquisition. If the EICP (extracted ion current profile) area for any of the internal standard changes by a factor of two (-50% to +100%), from the last daily calibration standard, the GC/MS must be inspected, and corrective action taken. If the retention time for any internal standard has shifted by more than 30 seconds from the mid-point standard level of the most recent calibration sequence, the GC/MS must be inspected, and corrective action taken. All samples and QC must also meet the EICP area and retention time criteria or must be reanalyzed.

7.5.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

An MS/MSD must be analyzed every twenty samples of a similar matrix. The MS/MSD is prepared in a manner similar to the LCS, except that 40 mL aliquots (aqueous) or 5 g aliquots (soil), of environmental samples are used in place of the analyte-free laboratory reagent grade water. Note that trip blanks and field/equipment blanks should not be used for MS/MSD analyses. The spike solution (section 7.5.1) is added to the sample at a concentration of 50 ppb. Acceptance criteria for the MS/MSD are outlined in Section 8.0.

7.6 SAMPLE ANALYSIS

When new samples are received, they should be checked for past sample history. If sample history cannot be located or the sites are different than past sites, the project manager should be consulted. He/she may be able to provide more information about the sample. Sample history is used to determine what order in which to run the

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samples and at what dilution. Refer to Katahdin Analytical Services SOPCA-106, "Basic Laboratory Technique", current revision for information on subsampling.

Samples are removed from the VOA refrigerator and appropriate chain of custody form is completed. Remove only the vials that have not been opened yet (opened vials will be upside down). Note in sample run log any bubbles, and significant discoloration or sediment in the sample vials.

7.6.1 SAMPLE ANALYSIS FOR 8260B WATER

7.6.1.1 Tekmar LSC 2000 / 2016 units

Rinse a 5.0 mL gas-tight syringe a minimum of three times with analyte-free laboratory reagent grade water (e.g., Poland Spring or equivalent). Pour sample at ambient temperature into the syringe until nearly overflowing. Carefully insert and adjust plunger to sample volume of 5.0 mL. While adjusting plunger to final volume, expel extra volume of sample onto pH paper for sample pH verification. Add 1.0 uL of the internal and surrogate mixtures (250 ug/mL). Immediately inject contents of the syringe into the ALS sparger.

Record the sample pH in the injection logbook. Continue as above for each sample, ensuring that the 5.0 mL gas-tight syringe is rinsed a minimum of three times with laboratory reagent grade water between each sample.

7.6.1.2 Tekmar LSC 3000 / Archon 5100 units

Place the sample vials into the Archon sample tray and program the Archon for the appropriate sample volume and or dilution for the sample. The Archon unit will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standard. The Archon can be programmed to run as many samples as will fit in the twelve-hour window. The auto sampler hot water rinses the sparge vessel, transfer lines, purge needle, and syringe between samples to minimize possible carryover.

Record the sample pH in the injection logbook after sample analysis is complete (usually the day after the analysis is done) and return the sample vial to the sample refrigerator.

7.6.1.3 Centurion/Encon unit

Place the sample vials into the Centurion sample tray and program the Centurion for the proper sequence. The Centurion will automatically

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transfer the sample to the sparge vessel while adding the internal and surrogate standards. Using the Centurion software, the analyst can program the Centurion to run as many samples that will fit into a 12 hour clock. The autosampler uses hot water to rinse the sparge vessel, transfer lines, purge needle and sample needle to minimize carryover.

Record the sample pH in the injection logbook after sample analysis is complete (usually the day after the analysis is done) and return the sample vial to the sample refrigerator.

Make sure that all entries in the injection log have been made in a complete, neat, and legible manner. Corrections in any logbook must be crossed through with a single line, dated, initialed and have a written explanation or the applicable error code.

If for any reason a sample needs to be rerun, diluted or duplicated, a note in the comments field of the injection logbook must be entered, addressing the reason why in the logbook to facilitate answering any questions that may arise during the review process.

To minimize carryover from samples that contain a target compound at a level exceeding the upper limit of the calibration curve, the following must be done: monitor both the samples immediately after the contaminated sample as well as the next run of the contaminated sample in the same purge inlet for the target(s) in question; both must have levels <PQL.

7.6.2 ANALYSIS OF LOW-LEVEL SOIL SAMPLES

Method 5035 Closed System Purge & Trap procedure for low level soils
(5 ug/Kg -200 ug/Kg)

Selecting the appropriate technique may depend on cleanup goals, confidence levels, and anticipated levels of contamination. Field sampling activities typically result in Encore or Encore-like devices being submitted to the lab. These devices must be extruded within 48 hours. It is the laboratory's standard policy to extrude soil samples into 5 mL of Laboratory reagent free laboratory reagent grade water that contains a magnetic stir bar. The sample is subsequently frozen until analysis within 14 days. Note that the sample must be extruded and frozen within 48 hours of sampling, until analysis can begin. This approach is preferred over extrusion into sodium bisulfate because it is believed that the sodium bisulfate reacts with calcium carbonate in highly calcareous soils causing effervescence and driving the volatile analytes out of solution. There is also anecdotal

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information to suggest that acetone may be generated when bisulfate preservation occurs. The Katahdin sample ID, extrusion date, and time are recorded in the GC/MS extrusion logbook. Please refer to the Katahdin method 5035 SOP, CA-214 for more detail.

In lieu of the use of Encore samplers, the lab may pre-weigh 40 mL VOA vials containing 5 mL of laboratory reagent grade water or a 20% sodium bisulfate solution and a magnetic stir bar and ship these to the field. The vial is assigned a vial specific number prior to shipment to the field. The vial and weight will be recorded with its vial specific number in the methanol soil logbook. If possible the field sampler should weigh the sealed vial to ensure that 5 +/- 0.5 grams of sample were added in the field. When the lab receives the vials back from the field, the vials will be weighed and the weight recorded. The samples must be frozen within 48 hours of sampling, until analysis can begin.

The subsequent analysis is performed on a specially developed autosampler that heats, stirs, and purges the sample simultaneously without exposing the contents of the vial to the atmosphere. This procedure will help to minimize the loss of VOC's due to transport, handling, and analysis and may help minimize ambient lab contribution. The expected detection limits are consistent with the traditional low soil technique from method 5030. The Archon is programmed to heat each vial to 40°C during the purge time. Initiate purging for 11.0 minutes; the sample must be heated to 40°C ± 1°C before purging can begin. If you have questions concerning setting up the Tekmar or initiating a GC/MS batch run, consult the Organic Department Manager, or senior chemist within the group.

If the client does not require method 5035, method 5030 for analysis of low-level soils may be followed. This means that the Tekmar ALS 2016 unit may be used for the preparative step, as well as the Archon units.

7.6.3 ANALYSIS OF MEDIUM-LEVEL SOIL SAMPLES

Method 5030 Procedure for higher concentration soils (> 200 ug/Kg)

Higher concentration soils may be sampled as either a bulk sample or field preserved with a water miscible solvent such as methanol. If sampled in an Encore unit, the soil is extruded into methanol upon receipt at the lab.

Bulk Sample- A sample is placed in a glass jar or vial and returned to the lab for extraction and analysis. In this approach the lab takes an aliquot of soil and extracts with purge & trap grade methanol, a portion of the methanol is then analyzed for volatile analytes.

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Extraction

Calibrate the balance properly (See SOP CA-102) and note it in the appropriate logbook. Place 5.0 grams of thoroughly mixed, undecanted soil sample in a 40.0 mL vial. Add 5.0 mL reagent grade methanol. Shake for 2 minutes. Let stand for 3 minutes. Record extraction in soil prep logbook.

Methanol Field Preservation - A 5 gram sample is added to a VOA vial that has been previously charged with purge and trap grade methanol (the volume of methanol is dependent upon client request). The vial with methanol has been previously weighed in the lab and assigned a vial specific number prior to shipment to the field. The vial and methanol weight will be recorded with its vial specific number in the VOA vial prep logbook. If possible the field sampler should weigh the sealed vial to ensure that 5 +/- 0.5 grams of sample were added in the field. When the lab receives the vials back from the field, the vials will be weighed and the weight recorded. A portion of the methanol is then analyzed for volatile analytes.

For analysis on Archon or Centurion autosamplers, add 400 uL of the extract into 20 mL of organic-free laboratory reagent grade water (e.g., Poland Spring or equivalent). IS and SS is added by the Archon and/or Centurion autosampler for analysis. This will give an estimated calibration range between 500-10000 ug/Kg.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

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

- Surrogate recoveries
- stability of internal standard responses
- LCS spike recoveries
- method blank acceptance
- chromatography
- target compound detection/quantitation / review for false positives

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC

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elements, the chromatography and quantitation of target analytes must be reviewed.

7.7.1.1 Chromatography

The chromatography should be examined for the presence or absence of any "ghost" peaks and can also be used as an indication of whether or not matrix interferences might be influencing surrogate recoveries and/or ISTD area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g., Surrogate recoveries) to determine the necessity of reanalyses.

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

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. An "M" qualifier will automatically be printed on the quantitation report summary.

This manual integration package must then be submitted to the Organic Department Manager or his/her designee, who will review each manual integration.

For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

7.7.1.2 Target Compound Detection/Quantitation

The method files have been set up to error on the side of false positives, that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- all ions present in the standard mass spectra at a relative intensity > 25% must be present in the sample spectrum.
- the relative intensities of primary and secondary ions must agree within $\pm 20\%$ between the standard and sample spectra.
- ions greater than 25% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

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If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the response of the largest target compound hit in the upper half of the initial calibration range.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial data review should be accomplished at the beginning of a work shift for the previous set of analyses. After the analyst has completed his or her initial data review, the data should immediately be forwarded to the Organic Department Manager, or his/her designee.

7.7.1.3 Tentatively Identified Compounds (TIC)

TIC's may be requested by certain clients for samples. Refer to SOP CA-207 "GC/MS Library Search and Quantitation".

7.7.2 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into 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 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

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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 Independent Calibration Verification, LCS and MS/MSD Criteria

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 30 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Organic Department Manager, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states.

The LCS recoveries for all analytes are evaluated. 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

TITLE: **ANALYSIS OF VOA_s BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

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.

For projects or clients requiring DoD QSM 4.1 all project analytes in the ICV must fall between 80-120% of the true value. No samples may be run until the ICV criteria is met. Laboratory established recovery limits for LCS and MS/MSDs must be within 3 standard deviations of the mean LCS recovery. MS/MSD pairs must be run once per analytical/preparatory batch. RPDs must be less than or equal to 30% between MS and MSDs.

For analytes with no available DoD acceptance criteria, laboratory established limits shall be used.

8.2 Surrogate Recovery Criteria

Statistical limits are compiled annually for surrogate recoveries (archived in QA office). Statistical limits are only calculated when at least 30 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Organic Department Manager, Laboratory Operations Manager and Quality Assurance Officer. The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states.

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

8.3 QC Requirements

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Due to the 14-day hold time associated with this method, samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs 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 8260 for other method performance parameters and requirements.

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 8260B, current revision.

"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.

Katahdin SOP CA-101, Equipment Maintenance, current revision.

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TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

TABLE 1

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.3 of this SOP	Retune instrument, and verify
Six-point calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥ 0.30 , except chloromethane, 1,1-DCA and bromoform ≥ 0.10 ; RSD for RFs $\leq 30\%$ for CCCs. Refer to section 7.4.3 also.	Repeat initial calibration
Independent Calibration Verification	Once, immediately following calibration	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.1 of this SOP for more information on allowable exceedances For projects requiring DoD QSM 4.1, all target analytes must have $\pm 20\%$ recovery of true value.	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. For projects requiring DoD QSM 4.1, no samples may be run until a passing ICV is run.
Calibration verification	Once per each 12 hours, prior to sample analysis in absence of initial cal	SPCCs minimum RF ≥ 0.30 , except chloromethane, 1,1-DCA and bromoform ≥ 0.10 ; RF for CCC analytes $\leq 20\%$ (%D) of average initial multipoint RF For projects requiring DoD QSM 4.1, the %Difference/Drift for all target analytes must be less than or equal to 20%	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
IS	During data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS For projects requiring DoD QSM 4.1, IS responses and retention times are compared to the midpoint of the most recent ICAL for all samples and QC.	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning

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TABLE 1 (cont.)

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method Blank	One per batch of 20 or fewer samples. For projects requiring DoD QSM 4.1, one per preparatory batch.	No analytes of interest detected > PQL with the exception of Methylene Chloride See section 7.5.2 of this SOP for additional DoD acceptance requirements.	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS	One per batch of 20 or fewer samples.	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.4 of this SOP for more information on allowable exceedances. For projects requiring DoD QSM 4.1, DoD limits shall be used, unless otherwise specified by the project QAPP.	Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Surrogate spike	Every sample, control, standard and method blank	Statistically derived limits. For projects requiring DoD QSM 4.1, DoD limits shall be used, if available. Otherwise lab limits.	Reprep and reanalyze for confirmation of matrix interference when appropriate.
MS/MSD	One MS/MSD per every 20 samples. For projects requiring DoD QSM 4.1, one MS/MSD pair will be analyzed per preparatory batch per matrix if supplied with sufficient sample.	Statistically derived from lab data or nominal limits depending on the project. Statistical limits are used as default limits. For projects requiring DoD QSM 4.1, DoD limits shall be used, unless otherwise specified by the project QAPP.	(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.		
Demonstrate ability to generate acceptable P & A using 4 replicate analyses of a QC check standard	Once per year for each analyst; 4 reps	All recoveries within method QC acceptance limits	Recalculate results; locate and fix problem; rerun P & A study for those analytes that did not meet criteria prior to sample analysis

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

TABLE 2
 SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-202-10	METHOD 8260, current revision
Apparatus/Materials	None	
Reagents	None	
Sample preservation/ handling	Preserved samples analyzed within 14 days. Unpreserved samples analyzed within 7 days.	Preserved samples analyzed within 14 days. No criteria for unpreserved samples.
Procedures	(1) Use laboratory reagent grade water for low level soil calibration, method blanks, and laboratory control samples to minimize clogging of archon soil needles with sand. (2) Internal Standards- pentafluorobenzene, 1,4-difluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4	(1) Use an aliquot of a clean (control) matrix similar to the sample matrix. (2) Recommended internal standards – fluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4
QC - Spikes	None	
QC - LCS	None	
QC - Accuracy/Precision	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL
QC - MDL	None	

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 3

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Acetone	43	58
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl Chloride	76	41, 39
Benzene	78	-
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
2-Butanone	43	72
n-Butylbenzene	91	92, 134
Sec-Butylbenzene	105	134
Tert-Butylbenzene	119	91, 134
Carbon Disulfide	76	78
Carbon Tetrachloride	117	119
Chlorobenzene	112	77, 114
Chloroethane	64	66
2-Chloroethylvinyl Ether	63	65, 106
Chloroform	83	85
Chloromethane	50	52
Chloroprene	53	88, 90
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
Cyclohexane	56	84, 60
1,2-Dibromo-3-Chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
Diethyl Ether	74	45, 59
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
Cis-1,2-Dichloroethene	96	61, 98
Trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 3 (cont.)

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
Cis-1,3-Dichloropropene	75	77, 39
Trans-1,3-Dichloropropene	75	77, 39
Cis-1,4-Dichloro-2-butene	75	53, 77
Trans-1,4-Dichloro-2-butene	53	88, 75
1,4-Dioxane	88	58, 43
Di-Isopropyl Ether	45	43, 87
Ethylbenzene	91	106
Ethyl Methacrylate	69	41, 99
Ethyl Tertiary-Butyl Ether	59	87, 57
Freon-113	151	101
Hexachlorobutadiene	225	223, 227
2-Hexanone	43	58, 57, 100
Idomethane	142	127, 141
Isobutyl Alcohol	43	41, 42
Isopropylbenzene	105	120
P-ISOPROPYLTOLUENE	119	134, 91
Methacrylonitrile	41	67, 39
Methylcyclohexane	83	55, 98
Methylene Chloride	84	86, 49
Methyl Acetate	43	74
Methyl Methacrylate	69	41, 100
4-Methyl-2-Pentanone	43	58, 85, 100
Methyl Tert-Butyl Ether	73	57, 41
Naphthalene	128	-
Pentachloroethane	167	130, 132
Propionitrile	54	52, 55
N-PROPYLBENZENE	91	120
Styrene	104	78
Tertiary-Amyl Methyl Ether	73	55, 87, 71
Tertiary-Butyl Alcohol	59	41, 43
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Tetrahydrofuran	42	72, 71
Toluene	92	91
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,3,5-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132

TITLE: **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**

TABLE 3 (cont.)

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,3-Trimethylbenzene	105	120
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl Acetate	43	86
Vinyl Chloride	62	64
Xylenes (Total)	106	91
1-Chlorohexane	91	55,43

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 4
ANALYTE QUANTITATION AND INTERNAL STANDARDS

Pentafluorobenze	1,4-Difluorobenzene	Chlorobenzene - d5	1,4-Dichlorobenzene - d4
Dichlorodifluoromethane	1,2-Dichloroethane	1,3-Dichloropropane	1,1,2,2-Tetrachloroethane
Chloromethane	1,1-Dichloropropene	Tetrachloroethene	1,2,3-Trichloropropane
Bromomethane	Carbon tetrachloride	Dibromochloromethane	Isopropylbenzene
Vinyl chloride	Benzene	Chlorobenzene	Bromobenzene
Chloroethane	1,2-Dichloropropane	1,1,1,2-Tetrachloroethane	2-Chlorotoluene
Trichlorofluoromethane	Trichloroethene	Ethylbenzene	4-Chlorotoluene
Methylene Chloride	Dibromomethane	Xylenes (total)	1,3,5-Trimethylbenzene
Acetone	Bromodichloromethane	Bromoform	Tert-Butylbenzene
1,1-Dichloroethene	cis -1,3-Dichloropropene	Styrene	1,2,4-Trimethylbenzene
1,1-Dichloroethane	4-Methyl-2-pentanone	2-Hexanone	Sec-Butylbenzene
cis-1,2-Dichloroethene	Toluene-d8 (surr.)	Bromoform	1,3-Dichlorobenzene
trans-1,2-Dichloroethene	Toluene		P-Isopropyltoluene
Chloroform	trans-1,3-Dichloropropene		1,4-Dichlorobenzene
2,2-Dichloropropane	1,1,2-Trichloroethane		1,2-Dichlorobenzene
2-Butanone	1,2-Dibromoethane		N-Propylbenzene
Methyl-tert-butylether (MTBE)	Vinyl Acetate		1,2-Dibromo-3-chloropropane
Tetrahydrofuran	Methyl Methacrylate		1,2,4-Trichlorobenzene
Bromochloromethane	Ethyl Methacrylate		Naphthalene
1,1,1-Trichloroethane	1,4-Dioxane		Hexachlorobutadiene
Tertiary-butyl alcohol (TBA)	2-Chloroethylvinyl ether		1,2,3-Trichlorobenzene
Di-isopropyl ether (DIPE)	Bromofluorobenzene (surr.)		cis-1,4-Dichloro-2-butene
Ethyl-tert-butylether (ETBE)			trans-1,4-Dichloro-2-butene
Tertiary-amyl methyl ether			Pentachloroethane
Diethyl Ether			n-Butylbenzene
Carbon Disulfide			1,3,5-Trichlorobenzene
Freon-113			1,2,3-Trimethylbenzene
Iodomethane			
Acrolein			
Isobutyl Alcohol			
Allyl Chloride			
Chloroprene			
Propionitrile			
Methacrylonitrile			
Acrylonitrile			
Cyclohexane			
Methyl Acetate			
Methylcyclohexane			
1-Chlorohexane			
Dibromofluoromethane (surr.)			
1,2-Dichloroethane-d4 (surr.)			

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 1
 EXAMPLE OF VOA RUNLOG PAGE

KATAHDIN ANALYTICAL SERVICES
 GCMS-T INSTRUMENT RUNLOG

DATE/TIME OF BFB INJECTION: 8/6/09 08:52
 Reviewed by/Date:

SAMPLE NAME	DATAFILE	DF	ALS #	METHOD	PREP METHOD			Y/N	MS/MSD	PH	ANALYST	COMMENTS
					5030	5035	1311					
SO N6 BFB	TR906	-	-	BFB2889Q				Y			HCG	
VSTWST06A	TSS33	1	1	T826A31				Y				
LCSA W067144-1	TSS34	1	2					Y				
VAKA	TSS35	1	3					N				
↓ B W067144-2	TSS36	1	4					Y				target wide
54268-1	A TSS37	1	5		X			Y	1	<2		
-6	B TSS38	1	6					Y	2	<2		
5435-1	B TSS39	1	7					Y	3	<2		
-6	A TSS40	1	8					Y	4	<2		
54337-7	A TSS41	1	9					Y	5	<2		
54258-2	B TSS42	1	10					Y	6	<2		
-4	B TSS43	1	11					Y	7	<2		
54351-2	B TSS44	1	12					N	8	<2		156A RR
-4	B TSS45	1	13					Y	9	<2		
-7	B TSS46	1	14					Y	10	<2		
54337-1	A TSS47	1	15					Y	11	<2		
-2	A TSS48	1	16					Y	12	<2		
-3	A TSS49	1	17					Y	13	<2		
-4	C TSS50	1	18					Y	14	<2		
5435-6	C TSS51	1	19					Y	15	<2		
5435-5	E TSS52	1	20					Y	16	<2		
54422-1	A TSS53	1	21					Y	17	<2		
-2	A TSS54	1	22					Y	18	<2		20:29 ✓
RINSE	TSS55	2	23					Y				
↓	TSS56	2	24					Y				

STANDARD	CODE	STANDARD	CODE
BFB	V2802	IS MIX	V2810
CAL. STD.	V2808	SS MIX	V2811
LCS/MS MIX	V2809		
EXTRAS MIX	V2797		

Circle-Methods:
 SW846 8260 OLM 04.2
 EPA 624 OLM 03.1
 EPA 524 OLC 02.1
 SIM OLC 03.2

QAMS413

00000017

8/7/09

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GCMS LABORATORY
REVIEWED BY/DATE:

AMP1514 1519 1520 1521	1-Chlorohexane (EPA-1208) Lot: CB-R341A Ultra Exp: 11/30/07 1000µg/ml	Rec'd 11/07/07
AMP1522 1523 1524 1525	 Calif 80434 MA VPI Standard 600 - 1500 µg/ml in Purge and Trap Methanol Lot # A040187 Exp: 11/12 Store: Freezer Potech Corporation - 110 Berner Circle - Belafonte, PA 16823	Rec'd 11/10/07 FKL ↓
AMP1526 1527	 Calif 80465 RL Dithane Argonate Glass #1 2.00 - 10.00 µg/ml each in F11 Methanol Lot # A048252 Exp: 12/11 Store: Freezer Potech Corporation - 110 Berner Circle - Belafonte, PA 16823	Rec'd DMF 11/20/07
AMP1528 1529	 AS-E0285 Diethyl ether 5000 µg/ml in MeOH Lot: B4070099-1A Exp: Jan 19, 2010	FOR LABORATORY USE ONLY STORAGE Ambient POISON Rec'd DMF 11/21/07 ↓
AMP1530 1531	 CLP-LC4S-100X Laboratory Control Sample - Internal Standard Mix 2500 µg/ml in MeOH Lot: B2090027 Exp: Sep 6, 2012 3 comps.	FOR LABORATORY USE ONLY STORAGE Ambient POISON ↓
AMP1532 1533	 Calif 80624 RL DEE #11 ret. (80) hex-toluene standard 50 µg/ml each in Methanol Lot # A050631 Exp: 3/10 Store: Freezer Potech Corporation - 110 Berner Circle - Belafonte, PA 16823	Rec'd DMF 11/26/07

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 3

EXAMPLE OF VOA STANDARDS PREPARATION LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES
 GC/MS VOA STANDARD PREP LOG BOOK

CODE	STOCK NAME	STOCK	STOCK CONC ug/ml	VOLUME ADD. uL
VJ809	502.2 Cal Mix #1	AMP 2187	2000	150
STANDARD: 8260 LCS	MIBK	AMP 2121		
FINAL CONC (ug/mL): 200	502.2 Cal Meas Mix	AMP 2113		
FINAL VOLUME (mL): 1.5	S-4575-10x()	AMP 2147		
PREP DATE: 8/4/09	M-8260-ADD-10x	AMP 2180		
EXPIRATION DATE: 8/18/09	γ-chloro-xylene	AMP 2109	1000	500
MEOH VOLUME (uL): 150	cyclohexane	AMP 2183		
MEOH LOT #: E25806				
INITIALS: HCG				
8/14/09 (HCG)				
VJ810	Custom 8260 IS Mix	AMP 2071	5000	50
STANDARD: 8260 IS "1"				
FINAL CONC (ug/mL): 50				
FINAL VOLUME (mL): 5				
PREP DATE: 8/15/09				
EXPIRATION DATE: 8/19/09				
MEOH VOLUME (uL): 4950				
MEOH LOT #: E25806				
INITIALS: HCG				
HCG 8/15/09				
VJ811	8260 surrogate Mix	AMP 2223	2500	100
STANDARD: 8260 46 "1"				
FINAL CONC (ug/mL): 50				
FINAL VOLUME (mL): 5				
PREP DATE: 8/15/09				
EXPIRATION DATE: 8/19/09				
MEOH VOLUME (uL): 4900				
MEOH LOT #: E25806				
INITIALS: HCG				

Reviewed by/Date:

8/15/09

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 4
 STANDARD INFORMATION

VOA Standards

Standard	Concentration	Manufacturer	Catalog Number
1,2,3 Trimethylbenzene	2000 ug/mL	Restek	58733
1,2,3 Trichlorobenzene	2000 ug/mL	Accustandard	M-502-47-10X
1,2,4 Trimethylbenzene	2000 ug/mL	Accustandard	M-502-54-10X
1,3,5 Trichlorobenzene	neat	Supelco	44-2235
1,3,5 Trimethylbenzene	2000 ug/mL	Accustandard	M502-55-10X
2-CEVE	2000 ug/mL	Accustandard	M-601C-10X
502.2 Cal Mix #1 (gases)	2000 ug/mL	Restek	30042
502.2 Cal2000 Mega Mix	2000 ug/mL	Restek	30431
504.1 Cal Mix	200 ug/mL	Accustandard	M-504.1-CSS
Acrolein & Acrylonitrile	5000 ug/mL	Accustandard	M-603-M-5X
Appendix IX Volatiles Mix	various	Accustandard	M-8240C-R3-10X
Bromochloromethane	2000 ug/mL	Accustandard	M-502-03-10X
California Oxygenates Mix #1	2000 - 10,000 ug/mL	Restek	30465
Carbon Disulfide	2000 ug/mL	Restek	30258
Chloroprene	2000 ug/mL	Accustandard	APPX9-048-R1
Custom GC Std	2000 ug/mL	Accustandard	S-11160
Custom VOC mix	various	Accustandard	S-7920-R1
Custom Volatile GC/MS Std	2000 ug/mL	Accustandard	S-3432B
Custom Volatiles GC/MS	2000 ug/mL	Accustandard	S-3432A
Diethyl Ether	5000 ug/mL	Accustandard	AS E0285
Freon 113	2000 ug/mL	Supelco	4-7944
Method 8260 Additions	2000 ug/mL	Accustandard	M-8260-ADD-10X
Method 8260B-Revision	2000 ug/mL	Accustandard	M-8240B-R-10X
MTBE	2000 ug/mL	Supelco	4-8483
Napthalene	2000 ug/mL	Accustandard	M-502-40-10X
THF	2000 ug/mL	Accustandard	S-4575-10X
Vinyl Acetate	2000 ug/mL	Restek	30216
Vinyl Acetate	2000 ug/mL	Accustandard	APPX9-211-20X
VOA Calibration Mix #1 (Ketones)	5000 ug/mL	Restek	30006
TCL Ketone Mix	5000 ug/mL	Accustandard	CLP-022-25X
VOC Liquid Mix	2000 ug/mL	Accustandard	M-502A-R2-10X
Volatile Organic Compounds (gases)	2000 ug/mL	Accustandard	M-502B-10X
IS/SS/Tune			
Custom 8260 IS	5000 ug/mL	Restek	54577
Custom 8260 SS	5000 ug/mL	Restek	54578
4-BFB	2000 ug/mL	Supelco	48083
VOA Tuning Compound (BFB)	5000 ug/mL	Restek	30003
1,2 Dichlorobenzene-D4	2000 ug/mL	Supelco	48952-U
Fluorobenzene	2000 ug/mL	Supelco	
VOA IS (CLP)	2500 ug/mL	Restek	30004
VOA SS (CLP)	2000 ug/mL	Supelco	48943
624 IS	1500 ug/mL	Restek	30023
4-BFB/Fluorobenzene/Pentafl. (EPA 624)	20000 ug/mL	Accustandard	M-624-SS-M
8260A SS	2500 ug/mL	Restek	30240
CLP Only			
04.1 CLP VOA Cal 2000	2000 ug/mL	Restek	30456
LCS-IS	2500 ug/mL	Accustandard	CLP-LCS-IS-100X
LCS-Volatiles	200 ug/mL	Accustandard	CLP-LCS-V
CLP Volatiles DMC Stock Solution	deuterated compds	Cambridge Isotope	ES 5038
3.2 OLC mix	1000 - 2000 ug/mL	Restek	30492

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Prepared By: Casarin/Millett/Galaszyn Date: 2/97

Approved By:

Group Supervisor: JJ Galaz Date: 01/20/01

Operations Manager: Joh C Bente Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1-23-01

General Manager: Debra P. Keegan Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03 8270C	Format changes added pollution prevention, changes to calibration section, new limits, added instrument. Other minor changes throughout.	DN	1-23-01	1-23-01
04 8270C	updated sections 7.5.1.1 and 7.6.1 to be compliant with South Carolina requirements.	DN	5-22-01	5-22-01
05 8270C	Added statistical limits for LCS & MS/MSD recoveries. Modified corrective actions for these limits. Added 5973 instrument.	DN	5-21-02	5-21-02
06 8270C	major reorg. of sect's 4, 5, 6, 7 and Tables added definitions and information for new processing system. added wording to sect. 8 minor changes throughout	LA D	02/05	02/05
07 8270C	Sect. 5.3.2.1 changed 200 µg/l to 1500 µg/l Sect. 7.2 changed Instrument "K" to Instr. "R" Sect. 7.5.1 updated table sect. Table 5: minor edits	LAN	04/06	04/06

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN
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SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
08 8270C	Added Icv Criteria to Definitions, 7.5.2.2 and Table 1 Added RT window clarification to 7.5.2.3 Added Allowable Exceedances criteria to 8.4 and Table 1 Removed Grande Mean Option from 7.5.2.1 updated Std. Prep in 5.3.2.1 and 7.5.1 Reworded correlation coefficient criteria	LAD	05/07 07/07	05/07 07/07
09 8270C	Added new instrument (G). updated Section 10. Fixed grammatical errors.	LAD	02/08	02/08
10 8270C	Minor change to Section 5.3.2.1 - regarding the prep of the ICV.	LAD	04/09	04/09
11 8270C	updated throughout to address new calibration levels and DoDQSM Ver. 4.1 compliance	LAD	08/09	08/09

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN
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Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-204-11, titled "Analysis of Semivolatile Organic Compounds by Capillary Column GC/MS: SW 846 Method 8270".

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-204-11, titled "Analysis of Semivolatile Organic Compounds by Capillary Column GC/MS: SW 846 Method 8270".

Recipient: _____ Date: _____

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN
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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.

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, laboratory reagent grade water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount; a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution, which is different from the stock used to prepare standards.

INDEPENDANT CALIBRATION STANDARD: A solution prepared from a stock standard solution independent of the standard that is used to calibrate the instrument. Analyzed immediately after calibration,

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

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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.

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1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory.

Each analyst should know the location of 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 Analysis", SOP CA-512, "Preparation Of Sediment/Soil Samples By Sonication Using Method 3550 For Subsequent Extractable Semi-Volatiles Analysis" and SOP CA-526, "Preparation Of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For

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Subsequent Extractable Semivolatile Analysis”). An aliquot of the final extract is injected into the gas chromatograph for compound separation by capillary column, followed by the electron impact mass spectrometer for identification and quantitation.

Target and surrogate compounds are identified and compared to the mass spectra obtained from the analysis of standard solutions containing the same compounds. A relative response factor is established for each target compound and surrogate against an internal standard during the most recent initial or continuing calibrations. The identified compound is then quantitated using the relative response factor, the amount of internal standard in the sample, the initial volume of sample, and any other factors, such as dilutions.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of SVOCs is analyzed immediately after a sample containing high concentrations of SVOCs. Any samples that have suspected carryover must be reanalyzed.

4.0 APPARATUS AND MATERIALS

- 4.1 GC: Hewlett Packard 5890 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 constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.
- 4.4 Autosamplers: HP 7673As and HP 7683s
- 4.5 Hamilton syringes: 2.00 uL to 10 mL
- 4.6 Volumetric glassware: Grade A or equivalent
- 4.7 Columns: DB-5MS 30m, 0.25mm I.D., 25um film thickness, columns (J&W Scientific) or equivalent.
- 4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.
- 4.9 Data System: The Target software is used for processing data and generating forms.
- 4.10 1.8 mL vials with 350uL inserts

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4.11 Crimp tops with Teflon lined septa

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

The standards are prepared on an as needed basis (but not greater than every 6 months) and stored in screw cap amber bottles with Teflon liners in the BNA standards freezer between uses. Standards prepared from various stock solutions must always use the first expiration date of any of the solutions used for preparation.

5.3.2.1 Calibration Mix – Prepare a standard stock mix that contains those compounds commonly considered 8270 and those compounds commonly considered Appendix IX compounds. The compound dinoseb should not be added to this stock as it is only available in methanol. This will be added separately to each calibration level. Use Table 3 as a guide. The stock should be prepared at 125 ug/mL.

5.3.2.2 Independent Calibration Verification (ICV) Standard – From a source other than that used to make the calibration standards, prepare separate standards mixes (A and B) such that Standard Mix A contains those compounds commonly considered 8270 and Standard B Mix contains those compounds commonly considered Appendix IX compounds. Use Table 3 as a guide. Each stock should be prepared at 100 ug/mL.

5.3.2.3 DFTPP Solution – Prepare standard in methylene chloride containing DFTPP, Pentachlorophenol, Benzidine and DDT at a final concentration of 25 ug/mL.

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6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All semivolatile sample extracts should be refrigerated until analysis. Extracts must be analyzed within forty days following the date of extraction.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition and storage: C:\HPCHEM\1\DATA

Tune file: DFTPP.U

Method files: L8270CXX.M (all samples and standards)

Where:

XX = the calibration number in chronological order

L = instrument ID (R, U, or G)

DFTPP tuning acquisition: DFTPP390.M

NOTE: All acquisition parameters must be identical for L8270CXX.M and DFTPP390. M.

Data Files: L___.D, where ___ is a number in chronological order from 0001 to 9999 and L is the instrument ID (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.

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- 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.

<u>Mass</u>	<u>Criteria</u>
51	30.0 to 60.0 percent of mass 198
68	less than 2.0 percent of mass 69
69	present
70	less than 2.0 percent of mass 69
127	40.0-60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent relative abundance
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.

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

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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 approximately 60 mL/minute
Mass range	35 to 500 amu
Number of A/D samples	4
GC Peak threshold	500 counts
Threshold	10 counts

Set up the run on the Enviroquant system using "Edit Sample Log Table". For a more detailed explanation of the Enviroquant software, consult the appropriate manual, Department Manager, or senior chemist within the GC/MS group.

When the DFTPP has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The DFTPP run is processed using the current algorithms in the Target software.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, DFTPP must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument DFTPP is not in criteria.

The DFTPP tuning standard should also be used to assess the column performance and injection port inertness. Calculate the degradation of DDT to DDE and DDD; it should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, with no evidence of peak tailing. For clients requiring DOD criteria, the tailing factors for these two compounds should not exceed 2.

In order to document the performance of benzidine, pentachlorophenol and DDT, the following procedure must be followed. At the PC, which operates the instrument, load the method TUNETAIL.M into the ENVDA screen. Go into the quant drop down menu and select *calculate/generate report*. When that finishes, select *Qedit quant result*. Each compound can now be evaluated. Double click on benzidine and select *ChromEval* and then *Evaluate tailing*. Follow the instructions given on the screen to evaluate tailing. Send the report to the printer. Repeat the procedure for pentachlorophenol. Repeat the procedure for DDT, selecting *Evaluate degradation*. Follow the instructions given on the screen and then send the report to the printer. The report should be filed with the tune raw data.

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;

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conceivably, the run-time of an injection could end after the twelve hours.

7.5 INSTRUMENT CALIBRATION

7.5.1 Initial Calibration for Method 8270

Prior to the analysis of samples and required method blanks, and after the instrument DFTPP tuning criteria have been met, the GC/MS system must be calibrated. The calibration consists of a six point curve. The calibration levels are 10, 25, 50, 75, 100 and 125 ng/uL Calibration is done to determine instrument sensitivity and the linearity of GC/MS response for the semivolatiles target and surrogate compounds.

Final conc. (ng/uL)	SVOA Stock Soln Added (uL)	1000 ug/mL dinoseb Standard (uL)	MeCl ₂ Added (uL)	Final Vol (uL)	IS Added (uL)
10	16	2	182	200	2
25	40	5	155	200	2
50	80	10	110	200	2
75	120	15	65	200	2
100	160	20	20	200	2
125	100	0	0	100	1

If additional compound mixtures are added, the volume of MeCl₂ is adjusted to maintain a final volume of 200 or 100 uL. A 100 uL aliquot of each of the standards above is spiked as above with 4000 ng/uL Internal Standard stock and analyzed.

Internal Standards
1,4-Dichlorobenzene-d4
Naphthalene-d8
Acenaphthene-d10
Phenanthrene-d10
Chrysene-d12
Perylene-d12

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

Column Temperature Program	40°C for 3 minutes to 300°C at 10°/minute
Final Column Temperature hold	300°C
Run Time	34-36 minutes
Scan Start Time	1.8 minutes (time may vary dependent upon column length)
Injection volume	1 uL

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The conditions are set up in the method files L8270CXX.M.

After analysis of the six calibration points, they must be processed and evaluated for adherence to QC criteria. Minimum requirements of ID files are the use of specific quantitation ions and quantitating a specific set of targets and surrogates with a set internal standard. These requirements are found in Tables 3 and 5.

7.5.2 Initial Calibration Criteria

Relative response factors (RRFs) must be calculated and evaluated for each target compound and surrogate. The RRF is defined as follows:

$$\text{RRF} = \frac{A_x}{A_{IS}} \times \frac{C_{IS}}{C_x}$$

where: A_x = 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 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 thirteen calibration check compounds: Acenaphthene, 1,4-Dichlorobenzene, Hexachlorobutadiene, N-Nitrosodiphenylamine, Di-n-octylphthalate, Fluoranthene, Benzo(a)pyrene, 4-Chloro-3-Methylphenol, 2,4-Dichlorophenol, 2-Nitrophenol, Phenol, Pentachlorophenol and 2,4,6-Trichlorophenol. This is also applicable to clients that request DOD criteria.

There are also four system performance check compounds (SPCCs). They have no maximum percent RSD but they must meet a minimum RRF criterion of 0.050. The four SPCCs are N-Nitroso-di-n-propylamine, Hexachlorocyclopentadiene, 2,4-Dinitrophenol and 4-Nitrophenol. The SPCCs are used to check the stability of both the standard and the instrument. This is also applicable to clients that request DOD criteria.

Achieving CCC and SPCC criteria is not a substitute for performing a calibration for all target analytes.

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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. Please note that some options may not be allowable for certain states, federal programs, or clients. South Carolina does not allow option 1 or option 3 for compliance work originating in their state.

Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. For linear models, Target reports r^2 . This is calculated by either calculating r and squaring the result or by calculating the coefficient of determination. For a linear calibration, the equation for either is the same. The value for r^2 must be greater than or equal to 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order (seven calibration points required) polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.99.

Internal standard (IS) responses and retention times in all standards must be evaluated immediately after data acquisition; if the RT for any IS changes by more than 0.50 minutes from the latest daily calibration standard, corrections must be made to the chromatographic system. If the extracted ion current profile (EICP) area for any IS changes by more than a factor of two (-50% to +100%), corrective action must be performed.

Each GC/MS system must be calibrated following system corrective action, including ion source cleaning or repair and column removal or replacement.

If time remains in the clock after meeting the initial calibration acceptance criteria, samples may be analyzed. The calibration must be verified each twelve hour time period (time period starts from the moment of the DFTPP injection) for Method 8270. The SSTD050 in

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the curve may be used as the calibration verification standard as long as it meets the calibration verification acceptance criteria. All sample results must be quantitated using the initial calibration response factors.

7.5.2.2 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.

7.5.2.3 Retention Time Windows

Retention time windows are set at the midpoint standard of the calibration curve, following every ICAL. When a CV is analyzed (and not an ICAL), the retention time windows of the daily CV must be within 30 seconds of the midpoint calibration standard of the most recent ICAL. The samples analyzed following the daily CV must have retention times within 30 seconds of those for the daily CV. Each successive daily CV must be compared to the most recent ICAL midpoint standard.

7.5.3 Continuing Calibration

A calibration verification check standard must be performed once every twelve hours immediately following analysis of the tuning compound DFTPP. This check contains all target compounds and surrogates at a concentration of 50 ng/uL.

After quantitation of the 50 ng/uL continuing calibration check, response factors must be calculated and compared to the average response factors in the initial calibration. The Target program calculates the calibration check response factors and compares them to the average RFs in the calibration curve by calculating percent differences. The method 8270 CCC's must have a % difference of +/- 20%D in order to be considered in criteria. The method 8270 SPCC's must meet a minimum RRF criterion of 0.050 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%.

The area for the internal standards in the calibration verification must be within a factor of two (-50% to 100%) from the mid-point standard of the most recent initial calibration. This is listed in the ISTD monitor report.

If the calibration verification does not meet criteria, corrective action must be taken. Depending on the situation, corrective action may be as follows:

- Re-analyze the 50 ng/uL continuing calibration check.
- Change the septum; clean the injection port; install a clean, silanized

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quartz liner; cut off a small portion (1" to 3") of the front end of the capillary column (this is usually performed when acid RFs are low and/or chromatography is poor).

- Analyze a new initial calibration curve.

The last option, the generation of a new initial calibration curve, is usually chosen when percent difference are >30%. In these instances, there is little or no chance of a continuing calibration reanalysis meeting criteria. If there is any doubt concerning which corrective action to undertake, consult the Department Manager or a senior chemist within the group.

If the calibration verification does meet the criteria specified above then analysis may proceed using initial calibration response factors.

7.6 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 4000 ng/uL IS stock to the vial and then cap. This gives a 40 ng/uL final concentration for the internal standard compounds. The samples are topped with Teflon lined crimp top caps.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

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

- Surrogate Recoveries

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- 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 Department Manager.

7.7.2 Chromatography

The chromatography should be examined for the presence or absence of any ghost peaks and can also be used as an indication of whether or not matrix interferences might be affecting surrogate recoveries and/or Istd area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g. surrogate recoveries) to determine the necessity of reanalyzing.

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

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an "m" qualifier will automatically be printed on the quantitation report summary. All manual integrations are initialed, dated and given a code which describes the reason for the manual integration.

This manual integration package must then be submitted to the Department Manager or his/her designee, who will review each manual integration. For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

7.7.3 Target Compound Detection/Quantitation

The semivolatile ID files have been set up to err on the side of false positives; that is, to identify and quantitate peaks as target compounds that may not

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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 $\pm 20\%$ between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial review should be accomplished at the beginning of a work shift for the previous set of analyses.

7.7.3.1 Tentatively Identified Compounds (TIC)

TIC's may be requested by certain clients for samples. Refer current Katahdin to SOP CA-207 "GC/MS Library Search and Quantitation.

7.7.4 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into 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

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errors. When completed, the package is sent to the department manager for final review. A complete review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

7.8 Injection Port Liner Cleaning And Silanizing Procedure

- 7.8.1 Remove the rubber o-ring from the liner and place the liner in a large Erlenmeyer flask.
- 7.8.2 In the hood, pour nitric acid into the flask until the liner is covered. Place the flask on a hotplate and boil for 2-3 hours.
- 7.8.3 Let cool; drain nitric acid and thoroughly flush the liner with water.
- 7.8.4 Bake briefly in the muffle oven until liner is dry and cool to room temperature.
- 7.8.5 Place the liner in a beaker, fill with Sylon and let it soak for at least two hours.
- 7.8.6 Take out the liner and rinse it thoroughly with toluene.
- 7.8.7 Rinse the liner thoroughly with purge and trap grade methanol.
- 7.8.8 Bake the liner in the muffle oven for a minimum of three hours.

7.9 Instrument Maintenance

Instrument preventative maintenance is performed on a semi-annual basis by GC/MS chemists. This maintenance includes a thorough inspection and cleaning of all parts, including changing rough and turbopump oils. GC/MS analysts perform other maintenance on an as-needed basis. Typically, routine maintenance involves clipping off the front end of the DB-5MS column, replacing the injection port septum, and installing a freshly silanized quartz liner after sample analysis.

All maintenance must be documented in the instrument-specific maintenance log, whether it is routine or not. The Department Manager must authorize any maintenance over and above a routine source cleaning.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to 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

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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 (laboratory reagent grade water for water samples, baked organic-free sand for soil/sediment matrices) that is carried through the entire analytical procedure. One method blank must be extracted with each group of samples of a similar matrix and must be analyzed on the GC/MS system that was used to analyze the samples.

An acceptable method blank must contain less than or equal to the PQL of any target compound. For clients requiring DOD criteria, no analytes detected at $> \frac{1}{2}$ PQL and $> \frac{1}{10}$ the amount measured in any sample or $\frac{1}{10}$ the regulatory limit.

If the method blank exceeds these contamination levels, the analytical system is considered out of control and corrective action must be taken before sample analysis.

Reanalysis of the blank is the first step of the corrective action; if that does not solve the problem, a Katahdin Corrective Action Report (CAR) will be initiated.

Corrective action will be specified after consultation including the Department Manager, Operations Manager, and QA Officer.

8.2 Surrogate Recoveries

There are six surrogates, which can be divided as follows:

- B/N - Nitrobenzene-d5, 2-Fluorobiphenyl and Terphenyl-d14

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- Acid - Phenol-d5, 2-Fluorophenol and 2,4,6-Tribromophenol

The surrogates have laboratory derived statistical limits that are updated on an annual basis and are available in the QA office. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

If specifications are not met, the sample (or blank) should be reanalyzed. If specifications are met in the reanalysis, this reanalysis should only be submitted. If surrogate specifications are not met in the sample or method blank reanalysis, a Corrective Action Report (CAR) should be initiated. Corrective action will be specified after consultation including the Department Manager and Operations Manager.

For further information regarding the acceptance of surrogate recoveries, consult the Department Manager.

8.3 Internal Standard Responses

Internal standard responses and retention times (RT) in all samples and blanks must be evaluated as part of the technical data review. The method files have been set up to only detect compounds that fall within a set RT window. For Method 8270 analysis, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the daily continuing calibration standard, reanalysis must occur. If the reanalysis meets criteria, only the in-criteria run should be reported. If the reanalysis is still out-of-criteria, both analyses should be included in the sample package set.

MS/MSD samples that do not meet the EICP area criteria above do not have to be reanalyzed.

8.4 Laboratory Control Sample (LCS)

An LCS must be performed for each group of samples of a similar matrix, for the following, whichever is more frequent:

- Every 20 samples of a similar matrix or similar concentration, or
- Every batch of samples extracted.

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Department Manager, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts", current revision.

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The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states (i.e. South Carolina). For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits with the following sporadic exceedance allowances.

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. Nominal limits of 60-140% are used for all other compounds. Generally, corrective action is only taken for the short list of the spiked compounds. The specific corrective actions will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control

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indicators and/or the ability to reanalyze a sample within holding time. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

8.6 QC Requirements

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all of the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in 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. Samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs 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.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846, 2nd edition, 1982 (revised 1984), 3rd edition, 1986, and Updates I, II, IIA, III, IIIA, and IIIB 1996, 1998 & 2004, Office of Solid Waste and Emergency Response, U.S. EPA, Method 8270C, current revision.

Katahdin SOP CA-101, Equipment Maintenance, current revision.

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

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TABLE 1
QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.4	Retune instrument, and verify
Six-point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥ 0.05 ; RSD ≤ 30 for RFs of the CCCs; Average %RSD $< 15\%$ for all compounds. Refer to section 7.5.2.1 also.	Repeat calibration if criterion is not met
Independent calibration verification	Once after Initial calibration	$\pm 20\% D$	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 $\leq 20\%D$; SPCCs RF ≥ 0.05	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
ISs	Immediately after or during data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to $+100\%$ of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Demonstration of ability to generate acceptable accuracy and precision	Once per analyst and annually 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.

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TABLE 1, (cont.)

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
LCS for all analytes	One LCS per prep batch	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.4 of this SOP for more information on allowable exceedances.	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. 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.
Surrogate spike	Every sample, control, standard, and method blank	current statistical limits	(1) Check chromatogram for interference; if found, flag data (2) If not found, check instrument performance; if problem is found, correct and reanalyze (3) If still out reextract and analyze sample (4) If reanalysis is out, flag data
MS/MSD	One MS/MSD per every 20 samples	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits and section 8.5 of this SOP.	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) 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.		

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TABLE 2
SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-204-11	Method 8270, current revision
Apparatus/Materials	none	
Reagents	none	
Sample preservation/ handling	none	
Procedures	none	
QC - Spikes	none	
QC - LCS	none	
QC - Accuracy/Precision	none	
QC - MDL	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL.

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TABLE 3

Analyte Quantitation and Internal Standards

Internal Standard: 1,4-dichlorobenzene-d4	2,6-Dichlorophenol (8270 C)
	1,2,4-Trichlorobenzene
Target and Surrogates:	a, a-Dimethyl-phenethylamine (8270 C)
Pyridine	Naphthalene
N-Nitrosodimethylamine (not on TCL list)	4-Chloroaniline (not on PP list)
Aniline (not on TCL list)	Hexachlorobutadiene
Phenol	4-Chloro-3-methylphenol
Bis (2-chloroethyl) ether	2-Methylnaphthalene
2-Chlorophenol	N-Nitrosodi-n-butylamine (8270 C)
1,3-Dichlorobenzene	N-Nitrosopiperidine (8270 C)
1,4-Dichlorobenzene	o-toluidine (Appendix IX)
1,2-Dichlorobenzene	o, o, o-Triethylphosphorothioate (Appendix IX)
Benzyl alcohol (not on PP list)	Hexachloropropene (Appendix IX)
2-Methylphenol (not on PP list)	Isosafrole (Appendix IX)
2,2'-oxybis(1-chloropropane) (also known as Bis (2-Chloroisopropyl) ether)	Nitrobenzene-d5 (surrogate)
4-Methylphenol (not on PP list)	Internal Standard: Acenaphthene-d10
N-Nitroso-di-n-propylamine	Target and Surrogates:
Hexachloroethane	Hexachlorocyclopentadiene
Ethyl methanesulfonate (8270 C)	2,4,6-Trichlorophenol
Methyl methanesulfonate (8270 C)	2,4,5-Trichlorophenol (not on PP list)
2-Picoline (8270 C)	1-Chloronaphthalene (8270 C)
N-Nitrosomethylethylamine (Appendix IX)	2-Chloronaphthalene
N-Nitrosodiethylamine (Appendix IX)	2-Nitroaniline (not on PP list)
N-Nitrosopyrrolidine (Appendix IX)	Dimethyl phthalate
N-Nitrosomorpholine (Appendix IX)	Acenaphthylene
2-Fluorophenol (surrogate)	3-Nitroaniline (not on PP list)
Phenol-d6 (surrogate)	Acenaphthene
Internal Standard: Naphthalene-d8	2,4-Dinitrophenol
Target and Surrogates:	4-Nitrophenol
Nitrobenzene	Dibenzofuran (not on PP list)
Isophorone	2,4-Dinitrotoluene
2-Nitrophenol	2,6-Dinitrotoluene
2,4-Dimethylphenol	Diethyl phthalate
Acetophenone (8270 C)	4-Chlorophenylphenyl ether
Benzoic acid (not on PP list)	Fluorene
Bis (2-chloroethoxy) methane	4-Nitroaniline (not on PP list)
2,4-Dichlorophenol	1-Naphthylamine (8270 C)
	2-Naphthylamine (8270 C)
	Pentachlorobenzene (8270 C)

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TABLE 3 (cont.)

Analyte Quantitation and Internal Standards

1, 2, 4, 5-Tetrachlorobenzene (8270 C)
2, 3, 4, 6-Tetrachlorophenol (8270 C)
p-Phenylenediamine (Appendix IX)
Safrole (Appendix IX)
1,4-Naphthoquinone (Appendix IX)
Thionazine (Appendix IX)
5-Nitro-o-toluidine (Appendix IX)
1,2-Diphenylhydrazine (not on TCL list)
2-Fluorobiphenyl (surrogate)
2,4,6-Tribromophenol (surrogate)

Internal Standard: Phenanthrene-d10

Target and Surrogates:

4,6-Dinitro-2-methylphenol
N-Nitrosodiphenylamine
Diphenylamine (8270 C)
4-Bromophenylphenyl ether
Phenacetin (8270 C)
Hexachlorobenzene
4-Aminobiphenyl (8270 C)
Pentachlorophenol
Pentachloronitrobenzene (8270 C)
Pronamide (8270 C)
Phenanthrene
Anthracene
Di-n-butylphthalate
Carbazole (8270 B)
Fluoranthene
Sym-Trinitrobenzene (Appendix IX)
Diallate (Appendix IX)
4-Nitroquinoline-1-oxide (Appendix IX)
Methapyrilene (Appendix IX)
Isodrin (Appendix IX)
Dinoseb (Appendix IX)

Internal Standard: Chrysene-d12

Target and Surrogates:

Benzidine (not on TCL list)
Pyrene
Butylbenzyl phthalate
3,3'-Dichlorobenzidine
p-Dimethylaminoazobenzene (8270 C)
Benzo (a) Anthracene
Bis (2-ethylhexyl) phthalate
Chrysene
3-Methylcholanthrene (8270 C)
Aramite (Appendix IX)
Chlorobenzilate (Appendix IX)
3,3'-Dimethylbenzidine (Appendix IX)
2-Acetylaminofluorene (Appendix IX)
Terphenyl-d14 (surrogate)

Internal Standard: Perylene-d12

Target and Surrogates:

Di-n-octyl phthalate
Benzo (b) fluoranthene
Benzo (k) fluoranthene
Benzo (a) pyrene
Indeno (1,2,3-cd) pyrene
Dibenz (a, h) anthracene
Dibenz (a, j) acridine (8270 C)
Benzo (ghi) perylene
7,12-Dimethylbenz (a) anthracene (8270 C)
Hexachlorophene (Appendix IX)

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TABLE 4

PROCEDURE CONDENSATION

Clock

12 hours from injection of 50ng DFTPP.

Calibration Curve Criteria

<30% RSD for CCCS
minimum RF criteria for SPCCs
<15% RSD average for all analytes in calibration standard

Continuing Calibration Check Criteria

<20% D for CCCS
minimum RF criteria for SPCCs

Additional QC

LCS every extraction batch
MS/MSD every 20 samples

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TABLE 5

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
2-Picoline	93	66,92
Aniline	93	66,65
N-Nitrosodimethylamine	42	74,43
Phenol	94	65,66
Bis(2-Chloroethyl)ether	93	63,95
2-Chlorophenol	128	64,130
1,3-Dichlorobenzene	146	148,111
1,4-Dichlorobenzene	146	148,111
1,2-Dichlorobenzene	146	148,111
N-Nitrosomethylethylamine	88	42,43,56
Benzyl alcohol	108	77,79
2-Methylphenol	107	108,77,79,90
Bis(2-Chloroisopropyl)ether	45	77,121
4-Methylphenol	107	108,77,79,90
N-Nitroso-di-n-propylamine	70	42,101,130
Hexachloroethane	117	201,199
Nitrobenzene	77	123,65
Isophorone	82	95,138
2-Nitrophenol	139	65,109
2,4-Dimethylphenol	122	121,107
Benzoic acid	122	105,77
Bis(2-chloroethoxy)methane	93	95,123
2,4-Dichlorophenol	162	164,98
1,2,4-Trichlorobenzene	180	182,145
Naphthalene	128	129,127
4-Chloroaniline	127	129,65,92
Hexachlorobutadiene	225	223,227
4-Chloro-3-methylphenol	107	144,142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235,272
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,97,132,99
2-Chloronaphthalene	162	164,127
2-Nitroaniline	65	92,138
Dimethyl phthalate	163	194,164
Acenaphthylene	152	151,153
3-Nitroaniline	138	108,92
Acenaphthene	153	152,154
2,4-Dinitrophenol	184	63,154
4-Nitrophenol	109	139,65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63,89
2,6-Dinitrotoluene	165	89,63
Diethyl phthalate	149	177,150
4-Chlorophenylphenylether	204	206,141
Fluorene	166	165,167
4-Nitroaniline	138	92,108,65,80,39
4,6-Dinitro-2-methylphenol	198	105,51
N-Nitrosodiphenylamine	169	168,167
4-Bromophenylphenylether	248	250,141

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN
GC/MS: SW 846 METHOD 8270.

TABLE 5 (cont.)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
Hexachlorobenzene	284	142,249
1,2-Diphenylhydrazine	184	77,92
Pentachlorophenol	266	264,268
Phenanthrene	178	179,176
Di-n-butyl phthalate	149	150,104
Carbazole	167	166,139
Fluoranthene	202	101,203
Benidine	184	92,185
Pyrene	202	200,203
Butylbenzylphthalate	149	91,206
3,3-Dichlorobenzidine	252	254,126
Benzo(a)anthracene	228	229,226
Bis(2-ethylhexyl)phthalate	149	167,279
Chrysene	228	229,226
Di-n-octyl phthalate	149	167,43
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,125
Indeno(1,2,3-cd)pyrene	276	138,277
Dibenz(ah)anthracene	278	139,279
Benzo(ghi)perylene	276	138,277
N-Nitrosodiethylamine	102	42,57,44,56
N-Nitrosopyrrolidine	100	41,42,68,69
N-Nitrosomorpholine	56	116,86
Acetophenone	105	71,51,120
2,6-Dichlorophenol	162	63,98
α,α -Dimethylphenethylamine	58	91,65,134,42
N-Nitrosodi-n-butylamine	84	57,41,116,158
N-Nitrosopiperidine	114	42,55,56,41
O-toluidine	106	107,77,51,79
O,O-Triethylphosphorothioate	198	121,97,65
Hexachloropropene	213	211,215,117,106,141
Isosafrole	162	131,104,77,51
1-Chloronaphthalene	162	127,164
1-Naphthylamine	143	115,89,63
2-Naphthylamine	143	115,116
Pentachlorobenzene	250	252,108,248,215,254
1,2,4,5-Tetrachlorobenzene	216	214,179,108,143,218
2,3,4,6-Tetrachlorophenol	232	131,230,166,234,168
p-Phenylenediamene	108	80,53,54,52
Safrole	162	104,77,103,135
1,4-Naphthquinone	158	104,102,76,50,130
Thionazine	107	96,97,143,79,68
5-Nitro-o-toluidine	152	77,79,106,94
4-Aminobiphenyl	169	168,170,115
Diphenylamine	169	168,167
Pentachloronitrobenzene	237	142,214,249,295,265
Phenacetin	108	180,179,109,137,80
Pronamide	173	175,145,109,147
sym-Trinitrobenzene	75	213,120
Dinoseb	211	163,240

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

TABLE 5 (cont.)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	PRIMARY ION	SECONDARY ION(S)
Diallate	86	234,43,70
4-Nitroquinoline-1-oxide	174	101,128,75,116
Methapyrilene	97	50,191,71
Isodrin	193	66,195,263,265,147
p-Dimethylaminoazobenzene	225	120,77,105,148,42
7,12-Dimethylbenz(a)anthracene	256	241,239,120
3-Methylcholanthrene	268	252,253,126,134,113
Aramite	185	191,319,334,197,321
Chlorobenzilate	251	139,253,111,141
3,3'-Dimethylbenzidine	212	106,196,180
2-Acetylaminofluorene	181	180,223,152
Dibenz(a,j)acridine	279	280,277,250
Hexachlorophene	196	198,209,21,406,408
Phenol-d6 (surrogate)	99	42,71
2-Fluorophenol (surrogate)	112	64
2,4,6-Tribromophenol (surrogate)	330	332,141
Nitrobenzene-d5 (surrogate)	82	128,54
2-Fluorobiphenyl (surrogate)	172	171
Terphenyl-d14 (surrogate)	244	122,212
1,4-Dichlorobenzene-d4 (istd.)	152	115,150
Naphthalene-d8 (istd.)	136	68
Acenaphthene-d10 (istd.)	164	162,160
Phenanthrene-d10 (istd.)	188	94,80
Chrysene-d12 (istd.)	240	120,236
Perylene-d12 (istd.)	264	260,265

Primary ions must not be changed except in unusual instances where interference occurs with a co-eluting non-target analyte. In this case, a secondary ion may be used for quantitation with the following rules:

- (1) The corresponding standard(s) (initial calibration curve and continuing calibration standard) must be re-quantitated with the secondary ion.
- (2) Approval must be obtained from the Department Manager or the laboratory Operations Manager.

The quantitation ion must then be changed back to the one specified in Table 3 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 CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

FIGURE 1

EXAMPLE OF RUNLOG LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES
GC/MS SVOA INJ LOG INSTRUMENT: 5973-U

DATE OF DFTPP INJECTION: 010708

JOB	SAMPLE	DATAFILE	DF	ALS #	METHOD	UL INJ	CHEMIST	COMMENTS
	SD Wg DFTPP	UD626	1	1	DFTPP390	2.0	JLH	OK
B	SS1005DU0107	UD563		2	US270C02	1.0		✓
	010	64		3				✓
	025	65		4				✓ <i>Run OK</i>
	100	66		5				✓
	150	67		6				✓
	200	68		7				✓
A	SS1005DU0107	69 (L)		8				✓ (L) = UTCLP02 files
	010	70 (L)		9				✓
	025	71 (L)		10				✓
	100	72 (L)		11				✓
	150	73 (L)		12				✓
	200	74 (L)		13				✓
	8270 WND CHK	75 (L)		14				✓
J-010308								

QAMS364

STANDARD	CODE
DFTPP	S1176
CAL. STD.	S1181 S1182
IS MIX	S17C-3505

REVIEWED AND APPROVED BY: _____
DATE: _____

0000015

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN
GC/MS: SW 846 METHOD 8270.

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK ENTRY

KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GCMS LABORATORY
REVIEWED BY/DATE:

<i>Am20946</i>	 155 Market St., New Haven, CT 06513 - USA Tel. 203-785-5200 www.accustandard.com	FOR LABORATORY USE ONLY WARNING: This product contains a presence known to the State of California to cause cancer.
	APP-9-176-D-20X Pentachlorophenol 2.0 mg/mL in CH ₂ Cl ₂ Lot: B3010100 Exp. Jan 10, 2013	1 mL STORAGE Ambient POISON
		<i>Deal</i> <i>3/16/09</i> <i>JL</i>
<i>Am20947</i>	 155 Market St., New Haven, CT 06513 - USA Tel. 203-785-5200 www.accustandard.com	FOR LABORATORY USE ONLY
	APP-9-090-50X 4,6-Dinitro-o-cresol 5.0 mg/mL in MeOH Lot: B1100296 Exp. Aug 16, 2012	1 mL STORAGE Ambient FLAMMABLE
<i>Am20948</i>	 155 Market St., New Haven, CT 06513 - USA Tel. 203-785-5200 www.accustandard.com	FOR LABORATORY USE ONLY
	APP-9-145-50X p-Nitrophenol 5.0 mg/mL in MeOH Lot: B5050205 Exp. May 18, 2015	1 mL STORAGE Ambient POISON

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

FIGURE 3

EXAMPLE OF SVOA STANDARDS PREPARATION LOGBOOK ENTRY

GC/MS SVOA STANDARD PREP LOGBOOK

50863	8270 Stock (w/o MeCl ₂)	3-15-06	7-7-06	JLW	AMP0884	8270 meq/hup	300	2-22-07	4.2ml	150 ug/ml
					AMP0887	↓	300	3-17-07		
					AMP0911	APP IX # 2	600	3-2-07		
					AMP0910	↓ 1	100	3-9-07		
					AMP0870	↓ 1	200	7-7-06		
					AMP0699	Organophos pest	300	5-11-06		
					AMP0928	Benzoic Acid	↓	3-9-07		
					AMP0917	Hexachlorophene	↓	2-22-07		
					AMP0906	Propylene	↓	3-9-07		
					AMP0936	33'-Dichlorobenzene	↓	3-14-07		
					AMP0932	8270 Surv	150	3-9-07		
					50861	DEA	300	3-13-07		
					B43890	MeCl ₂	500	-		
50864	8270 Level 1	3-15-06	7-7-06	JLW	50863	8270 Stock	70	7-7-06	1.05ml	10 ug/ml
					B43890	MeCl ₂	980			
50865	8270 Level 2	3-15-06	7-7-06	JLW	50863	8270 Stock	150	7-7-06	0.90ml	25 ug/ml
					B43890	MeCl ₂	750			
50866	8270 Level 3	3-15-06	7-7-06	JLW	50863	8270 Stock	600	7-7-06	1.8ml	50 ug/ml
					B43890	MeCl ₂	1200			
50867	8270 Level 4	3-15-06	7-7-06	JLW	50864	8270 Stock	700	7-7-06	1.05ml	100 ug/ml
					B43890	MeCl ₂	350			

0000057

Reviewed by/Date:

TITLE: ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311

Prepared By: GC/MS Group Date: 7/97

Approved By:

Group Supervisor: J. Haley Date: 02/30/01

Operations Manager: John C. Benton Date: 2/13/01

QA Officer: Deborah J. Nadeau Date: 2/12/01

General Manager: Dennis F. Neff Date: 4/13/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 1311	Format changes, added pollution prevention. Changed wording around for section 7 and updated MS spiking.	DN	2/12/01	2/12/01
02 1311	added wording to section 8 minor changes to sections 8.2, 8.3 and table 1.	LAD	4/04	4/04
03 1311	grammatical and formatting correction	LAD	04/06	04/06
04 1311	Section 5.4 changed location of pH Cal. 4.3 removed acid washing procedure (req. for metals only). 1.4 added waste stream information and proper disposal. Updated Tables 1, Figs. Added QC table, Analyte List (Table 3), Rotary Extractor-Verification LB (Fig 2), Rotary Extractor (Fig 3) and ZHE Vessel (Fig 4). Added wording to Sect. 10 and 9. Added DDC info to 8(1,8)	LAD	03/07	03/07
05	Sect. 7.1b - corrected room temperature criteria. Table 2 - added method modifications for procedure of adding extraction fluid and the pressure the ZHE's are tumbled at. Updated logbook page.	LAD	11/08	11/08

TITLE: ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311

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-209-05**, titled **Zero Headspace Extraction (ZHE) of Volatile Samples for Toxicity Characteristic Leaching Procedure (TCLP) Method 1311**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **SOP CA-209-05**, titled **Zero Headspace Extraction (ZHE) of Volatile Samples for Toxicity Characteristic Leaching Procedure (TCLP) Method 1311**.

Recipient: _____ Date: _____

TITLE: ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services, Inc. technical personnel to extract solid matrix samples for volatile organics per EPA Method 1311, TCLP, using Zero Headspace apparatus.

1.1 Definitions

TCLP EXTRACTION BATCH: 20 or fewer samples, which are prepared together with the same method.

TCLP BLANK: An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Baked organic-free sand is used as a blank matrix. The same extraction fluid used for the samples is used for the associated TCLP blank. The blank is taken through the appropriate steps of the process.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of all TCLP compounds are added to a sample matrix and a sample matrix duplicate after filtration of the TCLP extracts and prior to sample analysis. 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. The purpose of the matrix spike is to monitor the performance of the analytical methods used.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the Toxicity Characteristic Leaching Procedure by EPA Method 1311. 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 TCLP Method 1311 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 Supervisor 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.

TITLE: ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311

1.3 Safety

When expressing the initial liquid from the waste to determine the percent solids, or when filtering the final TCLP extract from the ZHE after agitation, it is advisable to place the ZHE behind an explosion proof shield and to place the preweighed gas tight syringe on the liquid inlet/outlet valve without the plunger in the syringe. If the plunger is left in the syringe and the piston in the ZHE moves suddenly during pressurization, the plunger can become a dangerous projectile and/or the syringe could explode. Pressurize the ZHE slowly; if the pressure increases too much without the internal piston moving, carefully tap the outside of the ZHE to initiate movement. Do not exceed 20 psi if the piston does not move. In this event, vent the bottom flange and restart pressurization procedure. Too much pressure and a sudden release of the piston will force the liquid through the glass filter too fast, possibly rupturing the glass filter and/or blowing the syringe from the liquid inlet/outlet valve.

Always wear gloves, safety glasses and lab coat when handling the ZHE, sample or extract.

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 Hazardous Waste Plan and follow appropriate procedures such as: wearing safety glasses and gloves when working with chemicals or near an instrument; not taking food or drink into the laboratory; each analyst should know the location of a respirator and be trained on how to use it properly and 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 Plan for further details on pollution prevention techniques.

Waste accumulated from the ZHE vessel is classified as organic soil waste stream "I," which consists of leftover solids and used Borosilicate filters. The satellite for organic soil waste stream "I" is located inside the fume hood in the Volatile Organics Laboratory (room 111).

TITLE: ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD (taken from Method 1311)

For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis. The particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter.

For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP extract. If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

Potential interferences that may be encountered during analysis are discussed in the individual analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1 The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 revolutions per minute (rpm) – see Figure 3. Each of the laboratory's rotary extractors is equipped with a device that displays the actual rotation rate in rpm. The rotation rate of each extractor is monitored before each use, and the measured rotation rates are recorded in a logbook maintained for that purpose (see Figure 2). If the measured rotation rate of an extractor is outside the range 30 ± 2 rpm, it must be taken out of service until it can be repaired. (Associated Design and Manufacturing Co. or equivalent)

4.2 ZHE Vessels - The ZHE allows for initial liquid/solid separation, extraction and final extract filtration without opening the vessel – see Figure 4. The vessels have an internal volume of 500-600 mL and are equipped to accommodate a 90-110 mm filter. (Associated Design and Manufacturing Co. Model 3745-ZHE or equivalent).

TITLE: ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311

- 4.3 Filters - Borosilicate glass fiber containing no binder materials having an effective pore size of 0.6 to 0.8 μm . (Environmental Express Cat. #FG7590MM, size - 90 mm, pore size - 0.7 μm , or equivalent). Prefilters must not be used. Glass fiber filters are fragile and should be handled with care.
- 4.4 pH Meters accurate to ± 0.05 units at 25°C.
- 4.5 60 mL Gas-tight B-D Syringe - Collection device for initial liquid phase and the final extract of the waste when using the ZHE device.
- 4.6 ZHE Extraction Fluid Transfer Device - A 500 mL graduated cylinder which is capable of transferring the extraction fluid without changing the nature of it. (Associated Design and Manufacturing Co. Model #3775 or equivalent)
- 4.7 Laboratory Balance accurate to within ± 0.1 grams (all weight measurements are to be within ± 0.1 grams).
- 4.8 Beaker or Erlenmeyer flask, glass, 500 mL.
- 4.9 Magnetic stirrer.
- 4.10 Nitrogen tank complete with gauge as appropriate.

5.0 REAGENTS

Reagent grade chemicals shall be used in all tests. Other grades may be used only if it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. The purity of all chemicals must be known or evaluated before use to minimize any laboratory contamination.

- 5.1 Reagent water – Laboratory grade reagent water containing no interferent. Reagent water should be monitored periodically for impurities.
- 5.2 Sodium hydroxide (10N), NaOH, made from ACS reagent grade.
- 5.3 Glacial acetic acid, $\text{CH}_3\text{CH}_2\text{OOH}$, ACS reagent grade.
- 5.4 Extraction fluid 1 – Prepared and documented in Metals Laboratory. Please refer to the current revision of Katahdin Analytical Services SOP CA-510 for further information.

NOTE: The extraction fluid should be monitored frequently for impurities. The pH must be checked and documented prior to use to ensure that these fluids are made up accurately. Documentation of pH meter calibration prior to use is to be maintained

TITLE: ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311

in the pH Meter Calibration Logbook (metals prep lab). If impurities are found in the extraction fluid or the pH of the fluid is not within 4.93 ± 0.05 , the fluid shall be discarded and fresh extraction fluid prepared and documented.

5.5 Analytical standards shall be prepared according to the appropriate analytical method.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All samples shall be collected in a soil jar using an appropriate sampling plan.

6.1 Sufficient sample must be collected to support the preliminary determinations and to provide an extract volume adequate for analytical and quality control purposes. The necessary sample size will depend on the solids content of the waste, but in no instance should less than 30 g of waste be provided to the laboratory.

6.2 Preservatives shall not be added to samples before extraction. Samples should be stored at 4°C ($\pm 2^\circ\text{C}$) and opened only immediately prior to TCLP extraction.

6.3 TCLP extracts should be prepared for analyses and analyzed as soon as possible following TCLP extraction. Sample holding times for Volatile TCLP extraction and analysis are as follows:

Date of sampling to TCLP extraction: 14 days
TCLP extraction to analysis: 14 days

7.0 PROCEDURES

SAMPLE PREPARATION

7.1 Adjust the piston within the ZHE to a height that will minimize the distance it will have to move once the ZHE is charged with sample. It may be necessary to moisten the O-rings with extraction fluid to adjust the piston.

7.2 Weigh out a 10 g subsample of the waste and record the weight in the Volatile TCLP Extraction Logbook (Figure 1).

7.3 If the waste will obviously yield no liquid when subjected to pressure filtration, i.e., appears to be 100% solids, proceed to 7.12.

7.4 If the sample appears to contain low total solids (high degree of moisture but greater than .5% solids), approximate the amount of waste necessary so that after the liquid has been expressed there will be approximately 10 g of solid waste in the vessel and continue with step 7.7. The vessel can only be charged once.

TITLE: ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311

$$\text{Weight of waste to charge ZHE} = \frac{10}{\% \text{ solids}} \times 100$$

Generally, the TCLP Metals group will determine the percent solid of the sample and those results can be used in lieu of proceeding with the determination in step 7.7.

- 7.5 If the sample is less than .5% solids, it will be run as an aqueous sample. If the sample appears to be less than 0.5 % dry solids, this filtrate (or sample) is defined as the TCLP extract and is analyzed directly (See section 7.18).
- 7.6 If the sample appears to be an oil, extract as a medium level soil.

ZHE WITH PRELIMINARY DETERMINATION OF PERCENT SOLIDS

- 7.7 Quantitatively transfer the entire sample (both liquid and solid phases) quickly to the ZHE. Place the filter and support screens onto the top flange of the device and tighten. If it appears that more than 1% of the original sample weight has adhered to the container, determine the weight of this residue and subtract it from the sample weight.
- 7.8 When expressing the initial liquid from the waste to determine the percent solids, or when filtering the final TCLP extract from the ZHE after agitation, it is advisable to place the ZHE behind an explosion proof shield and to place the preweighed gas tight syringe on the liquid inlet/outlet valve without the plunger in the syringe. If the plunger is left in the syringe and the piston in the ZHE moves suddenly during pressurization, the plunger can become a dangerous projectile and/or the syringe could explode. Pressurize the ZHE slowly; if the pressure increases too much without the internal piston moving, carefully tap the outside of the ZHE to initiate movement. Do not exceed 20 psi if the piston does not move. In this event, vent the bottom flange and restart pressurization procedure. Too much pressure and a sudden release of the piston will force the liquid through the glass filter too fast, possibly rupturing the glass filter and/or blowing the syringe from the liquid inlet/outlet valve.
- 7.9 Attach a preweighed collection syringe to the liquid inlet/outlet valve (top flange) and open the valve. Attach the gas line to the gas inlet/outlet valve and pressurize to 1-10 psi slowly. Carefully increase the pressure to 50 psi at 10 psi increments (monitor collection syringe to prevent excessive pressure buildup which could detach or break the syringe). At each 10 psi increment, wait 2 minutes for additional liquid flow. Stop filtration when liquid flow has ceased within a 2 minute period at 50 psi.

CAUTION: Too much pressure at once can degrade the glass fiber filter and may cause premature plugging.

TITLE: ZERO HEADSPACE EXTRACTION (ZHE) OF VOLATILE SAMPLES FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) METHOD 1311

- 7.10 Reweigh the collection syringe to determine the percent solid. The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

$$\% \text{ Solids} = \frac{\text{Weight of initial sample} - \text{liquid}}{\text{Total weight of waste}} \times 100$$

The liquid phase may be analyzed immediately or stored at 4°C until time of analysis.

- 7.11 The particle size of a sample must be smaller than 1 cm in diameter prior to extraction. If particle size reduction is necessary, it can be accomplished by crushing, grinding or cutting particles that do not meet the size criteria. However, the sample and reduction equipment must be refrigerated to 4°C before size reduction.

Sieving the waste may cause volatiles to be lost and thus is not recommended. Proceed with step 7.15.

ZHE FOR WASTES WITH 100% PERCENT SOLIDS

- 7.12 The particle size of a sample must be smaller than 1 cm in diameter prior to extraction. If particle size reduction is necessary, it can be accomplished by crushing, grinding or cutting particles that do not meet the size criteria. However, the sample and reduction equipment must be refrigerated to 4°C before size reduction.

Sieving the waste may cause volatiles to be lost and thus is not recommended.

- 7.13 Quantitatively transfer the entire sample quickly to the ZHE. Place the filter and support screens onto the top flange of the device and tighten. If it appears that more than 1% of the original sample weight has adhered to the container, determine the weight of this residue and subtract it from the sample weight.
- 7.14 Determine the amount of extraction fluid #1 to add to the ZHE. If the waste appears to be less than 0.5 % liquid, or basically dry, use 10 g waste and 200 mL extraction fluid #1.

$$\text{amt. of extraction fluid} = \frac{20 (\% \text{ solids}) (\text{weight of waste filtered})}{100}$$

SAMPLE EXTRACTION

- 7.15 If the ZHE has been pressurized (determination of percent solids), release gas pressure on the ZHE piston. Add the appropriate amount of extraction fluid #1 using the 500 mL graduated cylinder. If the extraction fluid was prepared on the same day as sample extraction, ensure that fluid prep information has been recorded on the

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bench sheet in the Volatile TCLP Extraction Logbook. Otherwise, reference the date of fluid preparation on the bench sheet. Check the ZHE to ensure that there are no leaks. Pressurize the ZHE with 5-10 psi and check again for leaks. When the pressure is at 10 psi and no leaks appear, slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced during the addition of extraction fluid. Stop the bleeding at the first appearance of liquid from the valve since the appearance of liquid is an indicator of no headspace.

- 7.16 Place the ZHE in the rotary agitation apparatus and rotate at 30 ± 2 rpm for 18 ± 2 hours. Record the TCLP extraction start and end time in the Volatile TCLP Extraction log. Room temperature must be maintained and documented to be at $23 \pm 2^\circ\text{C}$ during agitation.
- 7.17 After the 18 ± 2 hour agitation period, check the pressure gauge on the ZHE to ensure there were no leaks over that time period. If the pressure within the device has been maintained, the sample in the ZHE vessel is separated into its component liquid and solid phases, as discussed in Steps 7.8 - 7.9. The liquid is carefully dispensed from the collection syringe into a VOA vial at a rate that precludes effervescence.

If the original waste contained no initial liquid phase, the filtered liquid material obtained is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained and the initial liquid phase are collectively defined as the TCLP extract.

If the individual phases are analyzed separately determine the volume of the individual phase (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume weighted average, as follows:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where: V_1 = The volume of the first phases (L).

C_1 = The concentration of the analyte of concern in the first phase (mg/L).

V_2 = The volume of the second phase (L).

C_2 = The concentration of the analyte of concern in the second phase (mg/L).

If the individual liquid phases are compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the TCLP extract and these are analyzed together.

- 7.18 Refer to Katahdin SOP CA-202, Analysis of VOAs By SW-846 Method 8260, current revision, for detailed procedure for GC/MS calibration and analysis.
-

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8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to the current revision of Katahdin SOP CA-202, Analysis of VOAs by Method 8260, for applicable quality control criteria. The following QC samples are prepared with each TCLP extraction batch:

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 Blanks: 1 blank is run per set per day. 1 ZHE vessel is loaded with 10 grams of baked sand, 200 mL extraction fluid #1, and analyzed as if it were a regular sample.
- 8.2 One Matrix Spike and Matrix Spike Duplicate extraction is performed for every 20 extractions. Matrix spike solution is added after filtration of the TCLP extract. All TCLP compounds are spiked.

Matrix spikes are added at a concentration equivalent to the corresponding regulatory limit, but not less than 5 times the method detection limit.

Matrix spike recoveries are calculated by the following formula:

$$\%R (\% \text{ Recovery}) = 100 (X_s - X_u)/K$$

Where: X_s = measured value for the spiked sample,
 X_u = measured value for the unspiked sample, and
 K = known value of the spike in the sample.

Measured sample values are reported without correction for analytical bias (based on the matrix spike recovery).

- 8.2.1 Preparation of Matrix Spike: The matrix spike is prepared with the method 8260 LCS mix. Refer to the 8260 SOP CA-202, current revision for further details.
- 8.2.2 Each new analyst must demonstrate her/his ability to perform the method acceptably by while being witnessed by an analyst who is experience in performing the method. To successfully demonstrate the method, the analyst must perform the method in conformance with all the requirements of the

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SOP, referring to the SOP for guidance as necessary. In addition, each analyst must demonstrate the ability to produce TCLP Extraction Blanks that are free of contamination. This demonstration will require the analyst to collect and file the analytical results from four Extraction Blanks that he/she has generated

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 Department Manager and with the QAO.

Refer to the current revisions of USEPA Method 1311 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Final Update III, Method 1311, US EPA, 12/96 or current revision

LIST OF TABLES AND FIGURES

Table 1	QC Requirements
Table 2	Summary of Method Modifications
Table 3	Toxicity Characteristic Constituents and Regulatory Levels
Figure 1	Example of Volatile TCLP Extraction Logbook Page
Figure 2	Example Page from Rotary Extractor RPM Verification Logbook
Figure 3	Rotary Agitation Apparatus
Figure 4	ZHE Vessel

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TABLE 1
 QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Toxicity Characteristic Leaching Procedure (TCLP)/ EPA 1311	Method Blanks	One per 20 samples extracted using a particular batch of extraction fluid.	Refer to individual analytical method.	Prepare fresh extraction fluid and repeat TCLP extraction of all associated samples.
		One per 20 samples extracted in a particular extraction vessel.	Refer to individual analytical methods.	Remove extraction vessel from service.
	Matrix Spike	One per 20 TCLP extractions performed (required). One per waste type (suggested, left to discretion of client).	Refer to individual analytical method.	Refer to individual analytical method.
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	New analyst's performance of the method is witnessed by an experienced analyst. New analyst must produce method blanks that meet all method and laboratory acceptance criteria.	Repeat analysis until able to demonstrate acceptable performance of the method to witnessing analyst and by producing acceptable method blanks; document successful performance in personal training file.

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TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-209-05	METHOD 1311, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling	Leachate is drawn into a 60 mL gas tight syringe and is dispensed into VOA vials at a rate that precludes effervescence.	Method recommends the use of TEDLAR bags and or 600 mL gas tight syringes.
Procedures	<p>Preliminary TCLP evaluations done on 10 g aliquot of waste; this subsample is also used for TCLP extraction.</p> <p>Extraction fluid is added to the ZHE prior to addition of sample</p> <p>ZHEs are tumbled at 40 ± 2 psi</p>	<p>Preliminary TCLP evaluations done on minimum 100 g aliquot of waste, which may not actually undergo TCLP extraction. Sample size for TCLP extraction is based on 25 g of solid in the waste subsample.</p> <p>Extraction fluid is added through the inlet valve.</p> <p>ZHEs are tumbled at 5 - 10 psi</p>
QC - Method Blanks	Frequency of one method blank per 20 extractions or each batch.	Frequency of one method blank per 20 extractions performed in a particular extraction vessel.
QC – Spikes	One Matrix Spike and Matrix Spike Duplicate extraction is performed for every 20 extractions.	A matrix spike shall be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. As a minimum, follow the matrix spike addition guidance provided in each analytical method.
QC – LCS		
QC – Accuracy/Precision		
QC – MDL		

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TABLE 3

TOXICITY CHARACTERISTIC CONSTITUENTS AND REGULATORY LEVELS

Constituent	Regulatory Level (mg/L)
Benzene	0.5
Carbon tetrachloride	0.5
Chlorobenzene	100.0
Chloroform	6.0
1,2-Dichloroethane	0.5
1,1-Dichloroethene	0.7
Methyl ethyl ketone	200.0
Tetrachloroethene	0.7
Trichloroethene	0.5
Vinyl Chloride	0.2

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FIGURE 1

EXAMPLE OF VOLATILE TCLP EXTRACTION LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES, INC.
VOLATILE TCLP EXTRACTION BENCH SHEET FOR 586578-6A

100% Dry Weight Determination <0.5% %Wet Solids _____

Refer to non-volatile TCLP extraction sheet, page # _____ for _____

Sample description: _____ Homogeneous _____ Non-homogeneous _____

1) Weight Container 1 + Residue _____ g	7) Weight of Filter _____ g
2) Weight Container 1 + Waste _____ g	8) Wt of Filter and Wet Solid _____ g
3) Weight of Waste ((2) - (1)) _____ g	9) Wt of Wet Solid Phase ((8) - (7)) _____ g
4) Weight of Container 2 _____ g	10) %Wet Solids (100*(9)/(3)) _____
5) Weight of Container 2 and Filtrate _____ g	11) Wt of Filter + Solid Phase Dry _____ g
6) Wt(g)/Vol(mL) of Filtrate ((5) - (4)) <u>1</u>	12) Wt of Solid Phase Dry ((11) - (7)) _____ g
	13) %Dry Solids (100*(12)/(3)) _____

Analyst JTC Date 11/19/08

Extraction Fluid Preparation and pH Check Fluid #1 Prepared Today _____ (Complete Prep Info Below)
Date prepared (if previously prepared) 11/11/08 Refer to Prep Info on Page 11-203A
Prep

_____ mL glacial acetic acid, manuf./lot # _____, added to 500 mL reagent water; _____ mL _____ N NaOH, manuf./lot # _____ added, then solution diluted to 1 L with reagent water.

Analyst JTC Date 11/19/08 pH of Fluid Today 4.93 Extraction Fluid Batch # 878

Extract Use Filter Paper Lot #: 5350401

<0.5% Solids (Filter adequate sample volume) _____ mL Filtered.
 100% Solids

10 g of Waste added to 200 mL of fluid (fluid volume = 20 times wt of solid sample)
 Free Liquid present (See Equation A for amount of waste to filter)

(A) X = Desired weight of solid phase on filter * 100 / (%Wet Solid)

1) Weight Container 1 + Waste _____ g	
2) Weight Container 1 + Residue _____ g	
3) Weight of Waste Charged to ZHE ((1) - (2), or weighed directly into vessel) _____ g	
4) Weight of Pre-extraction Collection Device _____ g	
5) Weight of Device and Filtrate _____ g	
6) Weight ((5) - (4))/Volume (mL) of Filtrate _____ g	6a _____ / 6b _____
7) _____ g of Solid Phase was added to 8) _____ mL of Fluid #1. ((3) - (6a)) (20 * (7a))	

Pressure before Tumbling 37 Pressure after Tumbling: 39
Time Started Tumbling 15:52 Time Stopped 16:40 Hours Extracted 18.48
Room Temp @ Start 21 Room Temp @ End _____

Was pre-extracted filtrate recombined with extract _____ YES _____ NO
If "NO" enter volume of filtrate (6) _____
Amount of Fluid (8) _____

Analyst JTC Date 11/20/08 Rev 6/08

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FIGURE 2

EXAMPLE PAGE FROM ROTARY EXTRACTOR RPM VERIFICATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

ROTARY EXTRACTOR RPM VERIFICATION LOGBOOK

EXTRACTOR #1: TOP SHELF SERIAL NUMBER: NONE
 EXTRACTOR #2: MIDDLE SHELF SERIAL NUMBER: 1173
 EXTRACTOR #3: BOTTOM SHELF SERIAL NUMBER: 1169

Please record the number of RPMs for each extractor each time they are used.

Date	Initials	Extractor #1	Extractor #2	Extractor #3	Comments
09/26/06	ATB	out of service	50	30	Replaced fuse in Ext. #2
10/1	ALL	out of service	29	Not use	
10/10/06	DMF	↓	30	↓	
11/30/06	DMF	↓	not in use	30	
12/27/06	DMF	↓	↓	30	
01/09/07	DMF	↓	↓	30	
01/11/07	DMF	↓	↓	30	
01/22/07	DMF	↓	↓	29	
01/25/07	DMF	↓	↓	30	
01/29/07	DMF	↓	↓	30	
01/31/07	DMF	↓	↓	30	
02/06/07	DMF	↓	↓	30	
02/12/07	DMF	↓	↓	30	

Acceptance Range is 28-32 RPMS.
 Meters Should Be Verified Against A Wrist Watch Annually And Recorded In The Comments Section.

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ROTARY AGITATION APPARATUS

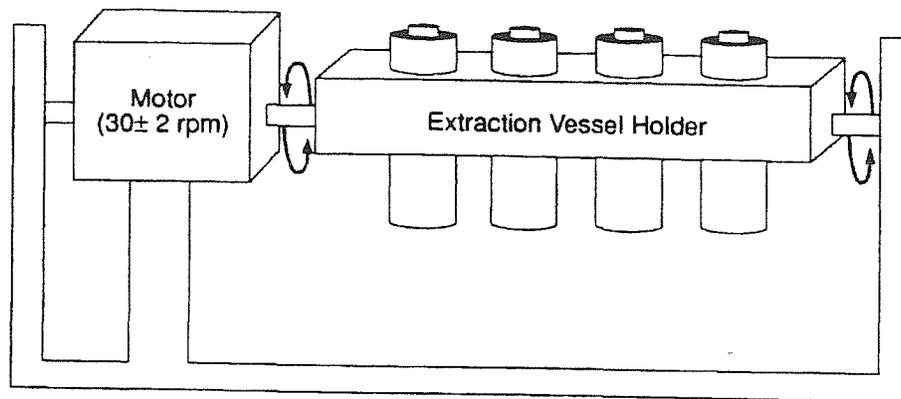


Figure 1. Rotary Agitation Apparatus

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FIGURE 4
ZHE VESSEL

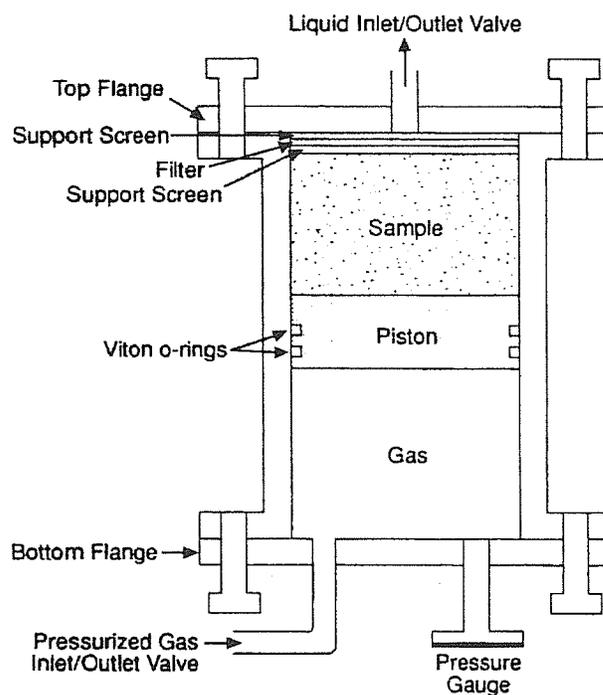


Figure 2. Zero-Headspace Extractor (ZHE)

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– Modified for Selected Ion Monitoring (SIM)**

Prepared By: GC/MS Department Date: 6/98

Approved By:

Group Supervisor: A. Galay Date: 020101

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

QA Officer: Deborah J. Nadeau Date: 1.31.01

General Manager: Dennis F. Kufan 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.	EN	1.31.01	1.31.01
02 8270C	Many changes in formatting. Some additions to section 8 & Table 1 to comply with NAVY.	EN	09.30.04	09.30.04
03 8270C	Sect. 7.2: Removed "K" Instrument & added "R" instrument. Added Pentafluorophenol surr. to Tables 3, 5 and Sect. 8.2. Removed all references to TIC'S.	LAD	04/06	04/06
04 8270C	Sect. 8.2 - changed 5 to 4 and removed pentachlorophenol. Table 3 and 5 - removed pentachlorophenol. changed linear regression correlation coefficient criteria. Added MI SOP reference. Added LCS exceedance criteria. Added ICV requirement and criteria. Added RT window procedure.	LAD	06/07	06/07
05 8270C	Added "G" instrument, Removed "X" instrument Edited section 7.5.1 - initial cal table	LAD	02/08	02/08

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270
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Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-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.

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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.

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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

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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 constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.
 - 4.4 Autosamplers: HP 7673As
 - 4.5 Hamilton syringes: 2.00 uL to 10 mL
 - 4.6 Volumetric glassware: Grade A or equivalent
 - 4.7 Columns: 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

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- 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.

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7.0 PROCEDURES

- 7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.
- 7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition and storage: C:\HPCHEM1\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.

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-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

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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 semivolatiles 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

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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.

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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:

$$\text{RRF} = \frac{A_x}{A_{IS}} \times \frac{C_{IS}}{C_x}$$

where: A_x = 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

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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.

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- 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 semivolatiles 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.

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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

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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 $\pm 20\%$ 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

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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.

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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)

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that is carried through the entire analytical procedure. One method blank must be extracted with each group of samples of a similar matrix and must be analyzed on the GC/MS system that was used to analyze the samples.

An acceptable method blank must contain less than or equal to the PQL of any target compound. **For clients requiring DOD criteria, no analytes detected at > ½ PQL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit.**

If the method blank exceeds these contamination levels, the analytical system is considered out of control and corrective action must be taken before sample analysis.

Reanalysis of the blank is the first step of the corrective action; if that does not solve the problem, a Katahdin Corrective Action Report (CAR) will be initiated. Corrective action will be specified after consultation including the Department Manager, Operations Manager, and QA Officer.

8.2 Surrogate Recoveries

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.

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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

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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.

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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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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 \leq 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	\pm 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 \leq 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 \pm 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Demonstration of ability to generate acceptable accuracy and precision	Once per analyst initially and annually thereafter	All recoveries within method QC acceptance limits.	Recalculate results; locate and fix problem; reextract/reanalyze P&A study for those analytes that did not meet criteria
Method blank	One per prep batch	No analytes detected > PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e.If the blank results are above the PQL, report samples that are <PQL or > 10X the blank result. Reprep a blank and the remaining samples.
LCS for all analytes	One LCS per prep batch	Statistically derived from lab data or nominal limits depending on the project. See also section 8.4 of this SOP for more information on allowable exceedances.	(1) Evaluate the samples and associated QC: i.e.If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS rec. is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.

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TABLE 1
QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogate spike	Every sample, control, standard, and method blank	Statistically derived limits.	(1) Check chromatogram for interference; if found, flag data (2) If not found, check instrument performance; if problem is found, correct and reanalyze (3) If still out reextract and analyze sample (4) If reanalysis is out, flag data
MS/MSD	One MS/MSD per every 20 samples	Statistically derived from lab data or nominal limits depending on the project. Nominal limits are used as default limits.	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

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TABLE 2
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-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 QUANTITATION 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 Dimethylnaphthalene (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

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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

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270
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TABLE 5
SVOA COMPOUNDS AND CHARACTERISTIC 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:

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(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.

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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
	SD wa DFTPP	X0600	1	1	DFTPP390	2.0	JR	OK
	SSTD060X0310	X9437		2	X 625A022	1.0		✓
	150	36		3				✓
	100	34		4				✓
	30	40		5				✓ curve JR
	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				
	BUNK 1	51		16				
	2	52		17				

STANDARD	CODE
DFTPP	S0856
CAL. STD.	S089-30 S089-49
IS MIX	AHP0937

REVIEWED AND APPROVED BY: _____
DATE: _____

QAMS302

0000034

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:

APP-9-176-D-20X Pentachlorophenol 2.0 mg/mL in CH ₂ Cl ₂ Lot: B3010100 Exp. Jan 10, 2013	1 mL STORAGE Ambient POISON	FOR LABORATORY USE ONLY WARNING: This product contains a chemical(s) known to the State of California to cause cancer JEA 8/16/09
APP-9-090-50X 4,6-Dinitro-o-cresol 5.0 mg/mL in MeOH Lot: B1100296 Exp. Aug 16, 2012	1 mL STORAGE Ambient FLAMMABLE	FOR LABORATORY USE ONLY
APP-9-145-50X p-Nitrophenol 5.0 mg/mL in MeOH Lot: B5050205 Exp. May 18, 2015	1 mL STORAGE Ambient POISON	FOR LABORATORY USE ONLY

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270
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FIGURE 3

EXAMPLE OF SVOA STANDARDS PREPARATION LOGBOOK ENTRY

GC/MS SVOA STANDARD PREP LOGBOOK

50863	8270 Stock (w/o MeOH)	3-15-06	7-7-06	JLW	AMP084	8270 hexaphenyl	300	2-22-07	4.2ml	150 ug/ml
					AMP087	↓	300	3-17-07		
					AMP091	APP IX # 2	600	3-2-07		
					AMP090	↓ 1	100	3-9-07		
					AMP080	↓ 1	200	7-7-06		
					AMP069	Organophos pest	300	5-11-06		
					AMP083	Propox Resid	↓	3-9-07		
					AMP087	Hexachlorophene	↓	7-22-07		
					AMP086	Propylene	↓	3-9-07		
					AMP036	3,3'-Dichlorobenzene	↓	3-14-07		
					AMP072	8270 Surr	150	3-9-07		
					50861	DEA	350	3-13-07		
B43890	MeCl ₂	550	-							
50864	8270 Level 1	3-15-06	7-7-06	JLW	50863	8270 Stock	70	7-7-06	1.05ml	10 ug/ml
					B43890	MeCl ₂	980			
50865	8270 Level 2	3-15-06	7-7-06	JLW	50863	8270 Stock	150	7-7-06	0.90ml	25 ug/ml
					B43890	MeCl ₂	750			
50866	8270 Level 3	3-15-06	7-7-06	JLW	50863	8270 Stock	600	7-7-06	1.8ml	50 ug/ml
					B43890	MeCl ₂	1200			
50867	8270 Level 4	3-15-06	7-7-06	JLW	50864	8270 Stock	700	7-7-06	1.05ml	100 ug/ml
					B43890	MeCl ₂	350			

Reviewed by/Date:

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: Dorothy J. Nadeau Date: 1.22.01

General Manager: Deanna F. Kufner 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.	HRC	05.04.04	05.04.04
06 8081A	added alternative CV Conc. changed data checklist minor changes throughout added wording to section 8	LAD	3/02/05	3/02/05

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 wastestreams. Sect. 4.5 - removed balance changed makeup gas from N ₂ to Ar Me. Changed TR 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 375. 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

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: _____

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

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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.

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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.

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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.

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- 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.

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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.

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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 $\pm 20\%$, 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

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- 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):

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

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

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 $\pm 15\%$. DoD allows a difference of $\pm 20\%$. 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 after a group of samples exhibits a response for an analyte that is above 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 was 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

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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.

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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

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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 quantitation report summary and assign a code that indicates

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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 $\pm 40\%$, 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

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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

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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.

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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.

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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 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.	$\pm 15\% D$ DoD: 20%D	(1) Evaluate the samples: If the %D>+15% and sample results are <PQL, narrate. If %D> $\pm 15\%$ only on one channel, narrate. If %D> $\pm 15\%$ 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	$\pm 20\% D$	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.		

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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 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 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

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FIGURE 1

EXAMPLE OF INSTRUMENT RUN LOG

Katahdin Analytical Services, Inc. GC Laboratory Instrument Runlog
 Instrument: GC08
 Amount Injected 2ul Method: 608 / 8081 / 8082
 (circle)
 Reviewed by/ Date: _____

Date	Init.	Result File	Sample ID	Y/N	Method	Column	Comments
6-13-07	SK	8AF1/2 154	WG39913-1 3510	Y	PSTA/B 2.74A	293/294	
			155 -2				
			156 -3				
			157 -4				TCLP blank PCB on PCB
			158 SA 2894-1 3550				
			159 -2				
			160 SA 2750-1 3510				
			161 Hexane	N			
			162 INDAB 0.025ppm	Y			P4228
			163 SA 2799-7 3510	Y			
			164 SA 2883-2 3550				
			165 SA 2753-1 3510				
			166 -2				10CB on B
			167 SA 2799-2 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 EVAL	Y			P4188
			178 INDAB 0.05ppm	N			most low P4229
			179 INDAB 0.05ppm				
			179 0.005ppm	Y	PSTA/B 2.75A		P4226
			180 0.01				P4227
			181 0.025				P4228
			182 0.05				P4229

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FIGURE 2

DATA REVIEW CHECKLIST

Verbal Due Date _____ Due Date _____

Client:	Primary	Secondary
Method:	Date:	Date:
SDG No: Level:	Initials:	Initials:
KAS No:	Approved : <input type="checkbox"/> 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 .

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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

TITLE: ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION
DERIVATIZATION: SW-846 METHOD 8151

Prepared By: Peter Lemay Date: 6/98
 Approved By: _____
 Group Supervisor: Peter Lemay Date: 1/24/01
 Operations Manager: John C. Burtis Date: 1/24/01
 QA Officer: Deborah J. Nadreau Date: 1.24.01
 General Manager: Deanna F. Kaufman Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8151A	Format changes, added pollution prevention, other minor changes to sections 7 + 8 and QA Table	DN	1.24.01	1/24/01
02	minor changes in sections 5 and 7	DN	4.9.02	4.9.02
03	Revised SOP to indicate Turbochrom is being used as instrument control and data collection software. Included Target related definitions. Changes to sections 7.5.3, 7.5.4, 7.6 and Table 1.	MRC	08.27.04	08.27.04
04	added Penta chloro phenol added alternating CV conc. (7.4.2) Table 1 - added CV conc. added Manual Int. SOP changed data review checklist	LAD	030405	030405
05	minor changes to reflect current practices, fix grammatical errors and formatting.	LAD	04/06	04/06

**TITLE: ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION
DERIVATIZATION: SW-846 METHOD 8151**

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-305-08**, titled **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

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TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION
DERIVATIZATION: SW-846 METHOD 8151**

1.0 SCOPE AND APPLICATION

This SOP details the procedure used by Katahdin Analytical Services, Inc. personnel for the analysis of soil and water extracts for Herbicides, Method 8151. It is applicable to the following compounds: 2,4-D, 2,4,5-TP (Silvex), 2,4,5-T, 2,4-DB, Dalapon, Dinoseb, Dichloroprop, Dicamba, MCPA, and MCPP. Extracts are analyzed by Gas Chromatography-Electron Capture Detector. Detection limits achievable by this method are listed in Table 3.

The analyte Pentachlorophenol has also been 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; 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.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution which is different from the stock used to prepare standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

INDEPENDENT CALIBRATION 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.

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

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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.

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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 herbicides by EPA Method 8151. 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 Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis by method 8151 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 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 the 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 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.

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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 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 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.

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

This SOP provides gas chromatographic conditions for the analysis of chlorinated acid herbicides in water, soil and waste samples. Water samples are extracted with diethyl ether and then esterified with diazomethane. Soil and waste samples are extracted and esterified with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector. Spiked samples are used to verify the applicability of the chosen extraction technique to each new sample type. The esters are analyzed by gas chromatography using an electron capture detector. The results are reported as the acid equivalent.

3.0 INTERFERENCES

Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis, by analyzing reagent blanks.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph

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- 4.1.1 GC Hewlett Packard 5890 series I or II or 6890 connected to the Turbochrom data system or Enviroquant data system, or equivalent.
- 4.1.2 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: DB-608 30M x 0.53 mm ID, DB-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.
- 4.5 Balances: Analytical, 0.0001 g
- 4.6 Refrigerator for storage of extracts and standards.

5.0 REAGENTS

- 5.1 Solvent: Hexane - herbicide quality or equivalent for diluting samples and standards.
- 5.2 Standards
 - 5.2.1 Stock standard solutions: Solutions purchased from suppliers like AccuStandard or other acceptable retailers as certified solutions of the methyl esters. 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. The standard mix contains the following compounds: Dalapon, MCPP, Dicamba, MCPA, Dinoseb, Dichloroprop, 2,4-D, Silvex, 2,4,5-T, 2,4-DB and DCAA as the surrogate. Pentachlorophenol is also present in the standard mix.
 - 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. The herbicide mix is diluted with hexane from concentrated stocks to give mixes at the following concentrations: 0.1/10,

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0.25/25, 0.5/50, 0.75/75, 1.0/100, and 2.0/200 ug/ml. Note the compounds MCPA and MCPP are 100 times more concentrated than the other eight, hence the ratio 0.1/10, etc. Pentachlorophenol is present in the standards at the lower of the two concentrations stated. Standards should be stored at 4°C.

5.2.3 Independent Calibration Verification Standard: Prepared at a mid point concentration using a standard independent of the calibration standards.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Extracts must be stored under refrigeration at 4°C (± 2°C) and analyzed within 40 days of extraction.

7.0 PROCEDURES

7.1 Instrument conditions

Refer to the instrument logbook for the current column and conditions. Typical conditions are as follows:

- Column flow: 2 ml/min He
- Makeup flow: 60 ml/min Ar/Methane or Nitrogen
- Run time: 31 min
- Injector temp: 200
- Detector temp: 325
- Oven ramp: 70° (0) 5° /min - 195° (0) - 11° /min - 261° (0)
- Injector size: 2 ul split

7.2 CALIBRATION

7.2.1 The GC system is calibrated using the external standard calibration procedure. A six-point calibration standard mix containing the analytes and surrogate listed in section 5.2.1 at the concentrations listed in section 5.2.2 is prepared.

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

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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. (Pentachlorophenol is a very responsive analyte and it may be necessary to omit the highest concentration standard if it appears the detector has been saturated at the high concentration. If this is necessary, a linear fit equation should be used for quantitation of this analyte.) 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

- 7.2.2 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 $\pm 20\%$, a new independent source standard must be analyzed or a new calibration curve must be prepared and/or analyzed.
- 7.2.2 The working calibration curve must be verified every 10 samples by injecting the 0.5/50ppm calibration standard. If the response for any analyte varies from the expected response by more than $\pm 15\%$, a new calibration curve must be prepared for that analyte. DoD allows a difference of $\pm 20\%$.
- 7.3 Retention time windows
- 7.3.1 Make three injections of all single component standard mixtures over 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.
- 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.
- 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.

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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: ± 0.07 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 by carefully evaluating the chromatograms.

7.4 Gas chromatographic analysis

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

7.4.2 Samples are analyzed in a set referred to as an analytical 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 0.5/50ppm standard (calibration verification standard). If a CV is run, the calculated concentration must not exceed a difference of $\pm 15\%$. DoD allows a difference of $\pm 20\%$. Each sample analysis must be bracketed with an acceptable initial calibration or an opening CV and a closing CV. The 0.25/25ppm standard should be used for the closing CV. If a second window of samples is run immediately after the closing CV, the concentration of the calibration standard at the completion of this window would be 0.5/50ppm. 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. Another CV is analyzed or the instrument is recalibrated and then samples are injected. All samples that were injected after the standard exceeding the criterion must be re-injected to avoid errors in quantitation, if the initial analysis indicated the presence of a specific target analyte that exceeded the criterion.

However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above 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 was 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

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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.

- 7.4.3 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.
- 7.4.4 The identification of Herbicides 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 retention time window. Each tentative identification must be confirmed using a second GC column of dissimilar stationary phase. 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.
- 7.4.5 If the response for an analyte exceeds the calibration range of the system, the sample must be diluted and reanalyzed.
- 7.4.6 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 a column, an ECD or an electronic board, this information is written in the instrument maintenance logbook. Refer to Katahdin SOP CA-101, Equipment Maintenance.
- 7.4.7 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration when the file is processed through the appropriate calibrated method.
- 7.4.8 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

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

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

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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: 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 7.6.

7.5.2 Surrogate recovery

All recoveries must meet the most recently established laboratory acceptance limits.

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.

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For method blanks, if the recovery of the surrogate is low or high, and the blank does not contain any target analytes above the PQL, and the recovery of the surrogate 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 the surrogate 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.

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 quantitation report summary and assign a code that indicates the reason for the manual integration. Refer to the current revision of 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

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present on both channels and the concentration is within the calibration range, and the quantitation from both chromatograms agrees within $\pm 40\%$, 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.

In order to avoid reporting false positives, identified peaks on a chromatogram may need to be undetected electronically in Target. The possible scenarios are: If an analyte is present on one column but its concentration is below the PQL, if an analyte is present on one column but does not confirm on the other channel, if an analyte is present on both columns but the concentrations differ by more than 40%, or if an analyte is present but its retention time is ± 0.04 minutes or more than the retention time of the analyte in the preceding CV.

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 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

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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 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 ten single component herbicides at the same concentration. The spike concentrations are:

	WATER ug/L	SOILS mg/Kg
All Herbicides except	5.0	0.17
MCPA and MCPP	500	17

The surrogate spike concentration in the final extracts is:

	WATER ug/ml	SOILS ug/ml
2,4-Dichlorophenylacetic acid (DCAA)	0.5	0.5

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,"

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current revision. For DoD work, the recoveries are compared to DoD QSM acceptance limits.

Dinoseb in a soil matrix recovers poorly. SW846 Method 8151 states that the soil hydrolysis step may result in the loss of dinoseb and the formation of aldol condensation products if any residual acetone remains from the extraction of solids. Dinoseb is also listed as a poor performer in the Department of Defense Quality Assurance Manual, current revision. For these reasons Katahdin Analytical Services defaults to the DoD QSM limits of 5-130% for this compound.

If any spike compound in the laboratory control sample falls outside of the established recovery acceptance limit window, the QC sample is considered to be out of control and any sample that is associated should be reextracted. However, if the recovery is high and the associated samples do not contain the specific compound(s), the data can possibly be accepted with narration.

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.

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.
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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 Organics 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. The Practical Quantitation Limit (PQL) concentrations for all the target analytes are listed in Table 3.

Refer to the current revision of Method 8151 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. Promulgated Update III dated December, 1996, Method 8151A.

Katahdin Analytical Services, Inc., SOP CA-106, Standard Preparation, Documentation and Traceability.

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: ie. 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	Laboratory statistically derived limits Dinoseb in soil – DoD QSM limits. 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 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	One after every 10 samples: alternating between 0.5/50ppm and 0.25/25ppm concentration	± 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% 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: ie. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable, reprep the 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 (2) Else, reanalyze
6pt calibration of Herbicide Mix	Initial cal prior to sample analysis	6 pt calibration – coefficient of determination ≥ 0.990	(1) Repeat Initial calibration
Demonstration of analyst proficiency – 4 replicates	Once per analyst initially and annually thereafter	P&A meet method criteria	Repeat P&A study
MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

**TITLE: ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION
DERIVATIZATION: SW-846 METHOD 8151**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-305-07	METHOD EPA 8151, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures	7.5.5 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: ± 0.07 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.	7.5.2.1 Plus or minus three times the standard deviation of the retention times for each standard will be used to define the retention time window. 7.5.2.2 In those cases where the standard deviation for a particular standard is zero, the laboratory must substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION
DERIVATIZATION: SW-846 METHOD 8151**

TABLE 3

PQLS FOR METHOD SW8151

Parameter/Method	Analyte	Practical Quantitation Limit (PQL)	
		(ug/L)	(ug/kg)
Herbicide/ SW846 8151	2,4-D	1.0	33
	2,4,5-TP	1.0	33
	2,4-DB	1.0	33
	2,4,5-T	1.0	33
	Dalapon	5.0	170
	Dicamba	1.0	33
	Dichloroprop	2.0	67
	Dinoseb	5.0	170
	MCPA	150	5000
	MCPP	100	3300

TITLE: ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

Katahdin Analytical Services, Inc.

GC Laboratory Instrument Runlog

Instrument: GC07

Method:
(circle)

SW846 8082 / SW846 8081

8151

Amount Injected 2ul

Reviewed by/ Date: Jurison

OLM04.3 / OLM03.2 / SOM01.1

Date	Init.	Result File	Sample ID	Y/N	Method	Column	Comments
5-21-07	JUP	7AE031	Hexane	N	HERB01	301/302	
5-22-07	JUP		32				
			41				TEST
			42				
			43				
			44				
			45				
			46				
			214.5-T	Y			
5-22-07	JUP	7AE047	ICAL 0.5ug/mL	Y	HERB01		P3957
			48				P3958
			49				P3959
			50				P3960
			51				P3961
			52				P3962
			53				P3994
			54				
			55				
			56				
			57				
			58				
			59				
			60				
			61				
			62				
			63				P3957
			64				
			65				
			66				
			67				
			68				

TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION
 DERIVATIZATION: SW-846 METHOD 8151**

FIGURE 2
 REVIEW CHECKLIST

Verbal Due Date _____ Due Date _____

Client:	Primary	Secondary
Method:	Date:	Date:
SDG No: Level:	Initials:	Initials:
KAS No:	Approved :	<input type="checkbox"/> 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 .

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: *[Signature]* Date: 6-23-06

QA Officer: *[Signature]* 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 LCS/D, 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 BN 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

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

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

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 semivolatiles 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

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

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 $\text{pH} \leq 2$ and extracted with methylene chloride using a continuous liquid-liquid extractor. The pH is then adjusted to $\text{pH} \geq 11$ 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 $\text{pH} \geq 11$ and then to $\text{pH} \leq 2$. 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 semivolatiles 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 interferences, 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.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

- 4.11 Water bath - heated, with concentric ring cover, capable of temperature control ($\pm 20^{\circ}\text{C}$). 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 $\frac{3}{4}$ " 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.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

5.5 Standard Preparation - For all standard preparations, see current revision of the following Katahdin Analytical SOPs:

- "Standards Preparation, Documentation and Traceability", (CA-106, current revision)
- "Balance Calibration," (CA-102, current revision)

5.5.1. 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-d ₆	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-d ₅	50 ug/mL
p-terphenyl-d ₁₄	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-d ₁₀	2.0 ug/mL
2-Methylnaphthalene-d ₁₀	2.0 ug/mL
Pyrene-d ₁₀ .	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.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

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 applicable
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

- Initial and final volumes or weight
- Surrogate and spike amounts
- Any sample cleanup performed
- 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.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

- 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 H₂SO₄ 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).

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

- 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 ≤ 2 in the same manner mentioned in 7.1.6. If the pH has changed, more acid should be added to make the pH ≤ 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 ≥ 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 ≥ 11 in the same manner mentioned in 7.1.6. If the pH has changed, more NaOH should be added to make the pH ≥ 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.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

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.

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- 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 $\text{pH} \geq 11$ 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 ≥ 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

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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 ≥ 11 . If the pH has changed back to neutral range, it must be readjusted to ≥ 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 ≥ 11 , the pH is then adjusted to ≤ 2 with 1:1 H₂SO₄. Add enough 1:1 H₂SO₄ to adjust the pH to ≤ 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” satalite.

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

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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 N₂ 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 $\frac{3}{4}$ " 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.

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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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TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-502-06	METHOD 3510, current revision
Apparatus/Materials	<ol style="list-style-type: none"> 1) 250 mL amber bottle or flask 2) 1.0 mL syringe 3) short stem funnels 	<ol style="list-style-type: none"> 1) 250 mL Erlenmeyer flask 2) 5.0 mL syringe 3) drying columns
Reagents		
Sample preservation/handling		
Procedures	<ol style="list-style-type: none"> 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 Na₂SO₄ 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 	<ol style="list-style-type: none"> 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 Na₂SO₄ 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	<ol style="list-style-type: none"> 1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL 	<ol style="list-style-type: none"> 1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	<ol style="list-style-type: none"> 1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL 	<ol style="list-style-type: none"> 1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL

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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	<ol style="list-style-type: none"> 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 Na₂SO₄ 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 	<ol style="list-style-type: none"> 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 Na₂SO₄ 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

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FIGURE 1

EXAMPLE OF SEMIVOLATILES LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - AQUEOUS SEMI-VOLATILES

SV SEM SEP

Extraction Method: (check one)	SWB46 3520 (CLLE)	SWB46 3510 (SEP) <input checked="" type="checkbox"/>	SWB46 3535 (SPE)
Analytical Method: (check one)	SWB46 8270 <input checked="" type="checkbox"/> EPA 825	CLP OLM04.2	CLP OLC02.1
Standards	Surrogate ID (1): <i>SV2350</i>	Spike ID (1): <i>SV2352</i>	Spike ID (3):
	Surrogate ID (2): <i>SV2349</i>	Spike ID (2): <i>SV2351</i>	
Solvents/Acid/Base	Solvent Lot # (Meq2): <i>H10621</i>	H ₂ SO ₄ # <i>635026</i>	NaOH Lot # <i>H22101</i>
Consumables	Filter Paper Lot # <i>K1147236</i>	Na ₂ SO ₄ Lot # <i>27969005</i>	Vial Lot # <i>00091428</i>
Nitrogen Water Bath Temperature	<i>35°C</i>	pH (1 st Extraction) <i>≥ 11</i>	pH (2 nd Extraction) <i>≤ 2</i>
Prep Start Time: <i>0930</i>	Prep End Time: <i>1230</i>	CLLE Acid Start:	CLLE Acid End:
		CLLE B/N Start:	CLLE B/N End:

Date Extracted	Ext. Inlt.	Sample ID	Initial Vol. ml	Surr. Vol.	Spike Vol.	Fraction	Final Vol. ml	Date Conc.	Tray Location	Initials	Comments
<i>10/24/09</i>	<i>CB</i>	<i>W670484-1</i>	<i>1000</i>	<i>1 mL</i>	<i>NR</i>	<input checked="" type="checkbox"/>	<i>1 mL</i>	<i>10/26/09</i>	<i>SV2352</i>	<i>CB</i>	<i>E1+R112490</i>
		<i>W670485-2</i>	<i>980</i>	<i>1 mL</i>	<i>1 mL</i>				<i>B9</i>		<i>R112487</i>
		<i>-3</i>	<i>980</i>						<i>B10</i>		<i>M5C6556-11 K</i>
		<i>-4</i>	<i>1000</i>						<i>B1</i>		<i>M5D ↓ -11 L</i>
		<i>W670484-2</i>	<i>1000</i>			<input checked="" type="checkbox"/>			<i>C2</i>		
		<i>-3</i>	<i>↓</i>			<input checked="" type="checkbox"/>			<i>C3</i>		
<i>CB 10/26/09</i>											

Date Extracted	Ext. Inlt.	Sample ID	Initial Vol. ml	Surr. Vol.	Spike Vol.	Fraction	Final Vol. ml	Date Conc.	Tray Location	Initials	Comments
<i>10/24/09</i>	<i>CB</i>	<i>5C6524-1a</i>	<i>1060</i>	<i>1 mL</i>	<i>NR</i>	<input checked="" type="checkbox"/>	<i>1 mL</i>	<i>10/26/09</i>	<i>SV2352</i>	<i>CB</i>	
		<i>-3 c</i>	<i>1010</i>						<i>C5</i>		
		<i>-5 d</i>	<i>1050</i>						<i>C6</i>		
		<i>-7 c</i>	<i>1060</i>						<i>C7</i>		<i>res double surr.</i>
		<i>-9 c</i>	<i>1030</i>						<i>C8</i>		
		<i>-11 h</i>	<i>1030</i>						<i>C9</i>		<i>MSD</i>
		<i>-13 c</i>	<i>1020</i>						<i>C10</i>		
		<i>-15 d</i>	<i>1010</i>						<i>D1</i>		
		<i>-17 c</i>	<i>1060</i>						<i>D2</i>		
		<i>-19 d</i>	<i>↓</i>						<i>D3</i>		
		<i>-21 c</i>	<i>↓</i>						<i>D4</i>		
		<i>-23 c</i>	<i>1070</i>						<i>D5</i>		
		<i>-25 c</i>	<i>1040</i>						<i>D6</i>		
		<i>5C6525-2 e</i>	<i>1060</i>			<input checked="" type="checkbox"/>			<i>D7</i>		
		<i>-3 c</i>	<i>↓</i>						<i>D8</i>		
		<i>-4 e</i>	<i>↓</i>						<i>D9</i>		
		<i>-5 d</i>	<i>1040</i>						<i>D10</i>		
		<i>-6 d</i>	<i>1040</i>						<i>E1</i>		

0000000

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: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

Prepared By: George Brewer Date: 12/97

Approved By:

Group Supervisor: George Brewer Date: 02/01/01

Operations Manager: John C. Banton Date: 2/2/01

QA Officer: Deborah J. Kadeau Date: 2.1.01

General Manager: Deborah F. Keefe Date: 2/08/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 1311	Changed figures, inserted database references. Format changes, added pollution prevention.	gn	2.1.01	2/1/01
02 1311	modified to reflect change from TCLP data base to handwritten logbooks. Changed metals spiking instructions	LAD	030805	030805
03	Added expiration dates for TCLP fluids (19R) Added DOC requirement Revised TCLP logbook to include SPLP and spaces for pH and exp. dates.	LAD	01/07	01/07
04	Sect. 4: Added use of fluorinated extraction vessels for organics. updated TCLP/SPLP Logbook example.	LAD	03/08	03/08
05	Updated Figure 8 - TCLP extraction logbook page.	LAD	03/09	03/09

**TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND
NON-VOLATILE ORGANIC ANALYTES**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-510-05**, titled **Toxicity Characteristic Leaching Procedure (TCLP) for Inorganic and Non-Volatile Organic Analytes**.

Recipient: _____ Date: _____

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STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-510-05**, titled **Toxicity Characteristic Leaching Procedure (TCLP) for Inorganic and Non-Volatile Organic Analytes**.

Recipient: _____ Date: _____

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to define the procedures used by Katahdin Analytical Services, Inc., personnel for TCLP extraction of samples for inorganic and non-volatile organic components using USEPA Method 1311 (Test Methods for Evaluating Solid Waste, Physical / Chemical Methods, US EPA SW846), with the modifications discussed in Table 2.

The TCLP (Toxicity Characteristic Leaching Procedure) is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.

If an analysis of the liquid fractions of the TCLP extract indicates that a regulated compound is present at a concentration that, after accounting for dilution from the other fractions of the extract, would be equal to or above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract. The regulated toxicity characteristic analytes are listed in Table 3.

1.1 Definitions - None.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in TCLP extractions. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, Personnel Training and Demonstration of Capability.

It is the responsibility of all Katahdin technical personnel involved in TCLP extractions to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be aware of inherent laboratory hazards, proper disposal procedures for contaminated materials, and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this

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method may not be precisely known; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets (MSDS) is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the 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 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 Waste Management Plan for further details on pollution prevention techniques.

Wastes from TCLP extraction may contain acids, heavy metals, toxic organics, and other toxic components and should be disposed of in a manner appropriate to the hazards they present. Further information regarding waste classification and disposal may be obtained by consulting the Katahdin Hazardous Waste Management Plan and the Department Manager.

2.0 SUMMARY OF METHOD

- 2.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP extract.
- 2.2 For wastes containing greater than or equal to 0.5% solids, the liquid phase is first separated from the solid phase and stored for later analysis. The particle size of the solid phase is reduced, if necessary, and the solid phase is extracted with an amount of extraction fluid equal to 20 times its weight. The composition of the extraction fluid employed depends on the alkalinity of the solid phase of the waste. After extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter.
- 2.3 If they are compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract and these are analyzed

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together. If they are incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

Because the dissolved solids contents of TCLP extracts are typically high, analyses of these extracts are often troubled by matrix interferences. Methods to detect and overcome matrix interferences are integral to the TCLP procedure and are discussed in detail in Section 8.0, Quality Control.

Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 APPARATUS AND MATERIALS

- 4.1 Agitation apparatus (rotary extractor) - The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 revolutions per minute (rpm) – see Figure 1. Each of the laboratory's rotary extractors is equipped with a device that displays the actual rotation rate in rpm. The rotation rate of each extractor is monitored before each use, and the measured rotation rates are recorded in a logbook maintained for that purpose (see Figure 7). If the measured rotation rate of an extractor is outside the range 30 ± 2 rpm, it must be taken out of service until it can be repaired.
- 4.2 Extraction vessels - must fit the rotary extractor and have sufficient capacity to hold the sample and the extraction fluid (jars with capacities of 2.2 L are normally used). The vessel must be made of borosilicate glass or fluorinated polyethylene if the extract is to be analyzed for organics. If the extract is to be analyzed only for inorganics, polyethylene or polypropylene containers may be used.
- 4.3 Filter Holder - Filter holders for pressure filtration are used. They are constructed of type 316 stainless steel (with or without PTFE linings) and are capable of sustaining internal pressures exceeding 50 psi. These devices have an internal capacity of 1.5 L and accommodate glass fiber filters 142 mm in diameter.
- 4.4 Filters - Borosilicate glass fiber filters containing no binder materials and having an effective pore size of 0.6 to 0.8 μm , 142 mm diameter or equivalent. Prefilters must not be used. Glass fiber filters are fragile and should be handled with care. Filters should be acid-washed with 1N HNO_3 and triple rinsed with laboratory reagent grade water (minimum 500 mL/ rinse) prior to use.
- 4.5 pH meter accurate to ± 0.05 units at 25°C. The pH meter must be calibrated on each day of use.

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- 4.6 pH indicator strips covering the pH range 0 - 14 in increments of 1 pH unit.
 - 4.7 Laboratory balance accurate to within ± 0.01 grams (all weight measurements are to be within ± 0.1 grams).
 - 4.8 Beakers flasks, glass, 500 mL..
 - 4.9 Watch glasses, appropriate diameter to cover beakers.
 - 4.10 Magnetic stirrer.
-

5.0 REAGENTS

Reagent grade chemicals shall be used in all tests. Other grades may be used only if it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

- 5.1 Laboratory reagent grade water – Water free of any analyte of interest. Laboratory reagent grade water should be monitored periodically for impurities.
- 5.2 Hydrochloric acid, concentrated (HCl) – reagent grade.
- 5.3 Nitric acid, concentrated (HNO₃) – reagent grade.
- 5.4 Hydrochloric acid, 1N. Dilute 83 mL reagent grade HCl to 1000 mL with laboratory reagent grade water.
- 5.5 Nitric acid, 1N, for acid-washing filters. Dilute 63 mL reagent grade HCl to 1000 mL with laboratory reagent grade water.
- 5.6 Sodium hydroxide (NaOH) – reagent grade, pellets.
- 5.7 Glacial acetic acid (CH₃COOH) – reagent grade.
- 5.8 Extraction Fluid #1 - Add 114 mL glacial acetic acid and 51.4 g sodium hydroxide to approximately 1500 mL of laboratory reagent grade water in a clean borosilicate glass extraction vessel reserved for this purpose. Shake until the sodium hydroxide is completely dissolved. Pour this solution into a clean, graduated 20 L carboy reserved for Extraction Fluid #1 and rinse the extraction vessel three times with approximate liter volumes of laboratory reagent grade water, adding the rinsates to the carboy. Add laboratory reagent grade water to the carboy to bring the volume to the 20 L graduation. Cap the carboy and agitate until the fluid is well mixed. When correctly prepared, the pH of this fluid will be 4.93 ± 0.05 . The fluid may be used for up to one year from the preparation date.

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- 5.9 Extraction Fluid #2 - Add approximately 10 L of laboratory reagent grade water to a graduated 20 L carboy reserved for Extraction Fluid #2. Add 114 mL glacial acetic acid to the carboy, and then add laboratory reagent grade water to bring the volume to the 20 L graduation. Cap the carboy and agitate until the fluid is well mixed. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05 . The fluid may be used for up to one year from the preparation date.

NOTE: The pH of each extraction fluid must be checked prior to each use to ensure that it has been prepared accurately, and the measured pH is recorded in the Non-Volatile TCLP Extraction Logbook (Figure 8) for each sample extracted. Details of the preparation of these fluids (reagent lot numbers, volumes, and masses; measured pH; etc.) are recorded in the TCLP Fluid Preparation and Use Logbook (Figure 6). Upon preparation, each new batch of extraction fluid is assigned a 3-digit batch number by the analyst (batches are numbered consecutively), and the Katahdin Sample Number of each client sample extracted with a particular fluid batch is recorded in the TCLP Fluid Preparation and Use Logbook. Extraction fluids are monitored for impurities as described in Section 8.0 of this SOP.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All samples shall be collected in a soil jar using an appropriate sampling plan.

- 6.1 Sufficient sample must be collected to support the preliminary determinations and to provide an extract volume adequate for all analytical and quality control purposes. The necessary sample size will depend on the solids content of the waste, but in no instance should less than 250 g of waste be provided to the laboratory.
- 6.2 Preservatives shall not be added to samples before extraction. Samples should be stored at 4°C and opened immediately prior to TCLP extraction.
- 6.3 TCLP extracts should be prepared for analyses and analyzed as soon as possible following TCLP extraction. Extracts for metals analysis must be acidified to a pH < 2 with nitric acid. Extracts for other analyses should be preserved according to the guidance given in the individual analytical methods. Extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses.
- 6.4 Sample holding times for non-volatile TCLP extraction and analysis summarized in the following table:

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TCLP PARAMETER	FROM COLLECTION TO TCLP EXTRACTION	FROM TCLP EXTRACTION TO PREPARATIVE EXT'N	FROM PREP EXT'N TO ANALYSIS
PEST/HERBS	14	7	40
SEMIVOLATILES	14	7	40
MERCURY	28	N/A	28
METALS EXCEPT MERCURY	180	N/A	180

7.0 PROCEDURES

The procedure consists of a series of preliminary evaluations of the waste, followed by the actual extraction. Flow charts summarizing the procedure appear as Figures 2 and 3. Preliminary evaluations are to be performed on a minimum 100 g aliquot of the waste. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include: (1) determination of the percent solids, Section 7.1; (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration, Section 7.2; (3) particle size evaluation, Section 7.3; and (4) determination of the appropriate extraction fluid to be used for the TCLP extraction, Section 7.4.

All information and measurements pertaining to TCLP extractions are recorded in the Non-Volatile TCLP Extraction Logbook (Figure 8). In the following procedure, the section or line of the Non-Volatile TCLP Extraction Logbook page in which the pertinent information should be recorded is indicated in bold, e.g. **Section II** or **Line C**.

PRELIMINARY EVALUATIONS

7.1 Determination of Percent Solids (**Section I**) - Percent solids is defined for TCLP as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids) the percent solids determination may be omitted. Proceed to Section 7.3, Particle Size Evaluation.

If the sample is liquid or multiphasic, liquid/solid separation by filtration is required to make a preliminary determination of percent solids. This involves the filtration device. The procedure is as follows, Sections 7.1.1 through 7.1.9:

7.1.1 Pre-weigh the filter (**Line A**) and the container that will receive the filtrate (filtrate vessel) (**Line B**).

7.1.2 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

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- 7.1.3 Weigh out a subsample of the waste (100 gram minimum) and record the combined weight of the weigh boat and waste (**Line C**).
- 7.1.4 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged, prior to filtration. Centrifugation is to be used only as an aid to filtration. If centrifugation is used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.
- 7.1.5 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder, spreading the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.
- 7.1.6 Weigh the weigh boat and any residue clinging to it (**Line D**). Determine the total weight of waste to be filtered by subtracting the weight of the weigh boat and residue from the weight of the weigh boat and waste (**Line E**).
- 7.1.7 Gradually apply vacuum or gentle pressure of 1-10 psi until air or pressurizing gas moves through the filter, collecting any filtrate in the pre-weighed filtrate vessel. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

- 7.1.8 Weigh the filtrate vessel and its contents (**Line F**). Determine the weight of the liquid phase by subtracting the weight of the filtrate vessel from the total weight of the filtrate-filled container (**Line G**).
- 7.1.9 Calculate the percent wet solids as follows (**Line H**):

$$\text{Percent wet solids} = \frac{(\text{Total weight of waste}) - (\text{Weight of liquid phase})}{\text{Total weight of waste}}$$

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7.2 If the percent solids determined in Section 7.1.9 above is equal to or greater than 0.5% and the weight of water entrained in the filter is small in comparison with the weight of the solid phase, then proceed to Section 7.3 to determine whether the solid material requires particle size reduction. Continue with Section 7.2 if it is noticed that the amount of the filtrate entrained in wetting the filter is significant in proportion to the weight of the solid phase. If the percent solids determined in Section 7.1.9 is less than 0.5%, then proceed to Section 7.5.4 using a fresh portion of the waste.

7.2.1 Remove the solid phase and filter from the filtration apparatus.

7.2.2 Dry the filter and solid phase at 100± 20°C until two successive weighings yield the same value within ±1%. Record the weight of the filter and dry solids (**Line I**).

NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

7.2.3 Calculate the weight of dry solids by subtracting the weight of the filter from the weight of the filter and dry solids (**Line J**).

7.2.4 Calculate the percent dry solids as follows (**Line L**):

$$\text{Percent dry solids} = \frac{\text{Weight of dry solids}}{\text{Total weight of waste}} \times 100$$

Note: Non-aqueous liquid samples (e.g. oils) may be entrained in the filter, and may remain in the filter after drying, contributing weight to the dried filter. If this is the case, the surface of the filter should be examined for apparent solids or particulate material. If none are found, a comment to that effect should be made in the Comments section of the Non-Volatile TCLP Extraction Logbook (e.g. "No apparent solids present – dry solid weight is due to entrained non-volatile liquid"), and the sample should be treated as if it contains less than 0.5% dry solids.

7.2.5 If the percent dry solids is less than 0.5%, then proceed to Section 7.5.4. If the percent dry solids is greater than or equal to 0.5%, proceed to Section 7.3.

7.3 Particle Size Evaluation - Visually evaluate the particle size of the solid phase of the waste. Filamentous material (cloth, paper, etc.) will require particle size reduction if it has a surface area per gram of less than 3.1 cm³. Other solid materials require particle size reduction if the particles are greater than 1 cm in their narrowest dimension (i.e. if they will not pass through a 9.5 mm standard sieve). Particle size reduction may be accomplished by cutting, crushing, or grinding the waste to a surface area or particle size as described above. Perform particle size reduction on

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the solid material that will actually undergo extraction, not on that used for the preliminary determinations.

- 7.4 Determination of Appropriate Extraction Fluid - If the solid content of the waste is greater than or equal to 0.5%, determine the appropriate fluid for the non-volatiles extraction as follows:

7.4.1 Weigh out a small subsample of the solid phase of the waste, reduce the particle size (if necessary) to approximately 1 mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 500 mL beaker or Erlenmeyer flask.

7.4.2 Add 96.5 mL of laboratory reagent grade water to the beaker, cover with a watch glass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH (**Section II**). If the pH is <5.0, use Extraction Fluid #1 and proceed with the TCLP extraction, Section 7.5.

NOTE: pH measurements may initially be performed using a pH indicator strip. If the measured pH is less than 3 or greater than 7, record the pH obtained from the indicator strip to the nearest whole pH unit. If the measured pH is between 3 and 7, use the pH meter to obtain a more accurate reading and record the pH to at least one decimal place.

7.4.3 If the pH from Section 7.4.2 is >5.0, add 3.5 mL 1N HCl, stir briefly, cover with a watch glass, heat to 50°C, and hold at 50°C for 10 minutes.

7.4.4 Let the solution cool to room temperature and record the pH (**Section II**). If the pH is <5.0, use Extraction Fluid #1. If the pH is still >5.0, use Extraction Fluid #2. Proceed to the TCLP extraction, Section 7.5.

TCLP EXTRACTION FOR NON-VOLATILES

- 7.5 A minimum sample size of 100 grams (solid and liquid phases) is recommended. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample, whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatiles organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to perform all of the required analyses. If necessary, multiple extractions may be performed and the extracts combined and aliquoted for analysis.

7.5.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solid), weigh out a subsample of the waste (100 g minimum), record the weight (**Section III**), and proceed to Section 7.5.11. If the sample

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is liquid or multiphasic, liquid/solid separation is required - proceed to Section 7.5.2.

- 7.5.2 Pre-weigh the container that will receive the filtrate (filtrate vessel) (**Line M**).
- 7.5.3 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid-wash the filter if extracting for metals components. Acid-washed filters may be used for non-volatile extractions even when metals are not of concern.
- 7.5.4 Weigh out a subsample of the waste (100 gram minimum) and record the combined weight of the waste and weigh boat (**Line N**). If the waste contains <0.5% dry solids, the liquid portion of the waste, after filtration, is defined as the TCLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the required analyses. For wastes containing >0.5% dry solids, information is obtained in Section 7.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the TCLP extract.
- 7.5.5 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If centrifugation is used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the sample filtration system.
- 7.5.6 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder. Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.
- 7.5.7 Weigh the weigh boat and any residue clinging to it (**Line O**). Determine the total weight of waste to be filtered by subtracting the weight of the weigh boat and residue from the weight of the weigh boat and waste (**Line P**).
- 7.5.8 Gradually apply vacuum or gentle pressure of 1-10 psi until air or pressurizing gas moves through the filter, collecting any filtrate in the pre-weighed filtrate vessel. If this point is reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase in pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psi (i.e., filtration

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does not result in any additional filtrate within a 2 minute period), stop the filtration.

The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

- 7.5.9 Weigh the filtrate vessel and its contents (**Line Q**). Determine the weight of the liquid phase by subtracting the weight of the filtrate vessel from the total weight of the filtrate-filled container (**Line R**). Decant the liquid phase into a graduated cylinder and measure and record its volume (**Line S**). Pour the liquid phase back into the filtrate vessel for storage. The liquid phase may now either be analyzed or stored at 4°C until time of analysis.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, as outlined in Section 7.5.8, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

- 7.5.10 Calculate the weight of wet solids by subtracting the weight of the liquid phase from the total weight of waste (**Line T**).
- 7.5.11 If necessary, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 7.3. Describe the particle size reduction process in **Section IV** of the logbook. When the surface area or particle size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.
- 7.5.12 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{(20) (\text{Weight of wet solids})}{100}$$

Slowly add this amount of appropriate extraction fluid to the extractor vessel. Record the fluid batch ID, the amount used, and the pH (measured on day of use) in **Section III** of the logbook. Close the extractor bottle tightly (Teflon tape may be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30± 2 RPM during the extraction period of 18 ± 2 hours. Record the extraction start and end times and the room temperatures in **Section IV** of the logbook.

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NOTE: As agitation continues, pressure may build within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

- 7.5.13 Following the extraction, separate the contents of the vessel into its component liquid and solid phases by filtering through a new acid-washed glass fiber filter, as outlined in Section 7.5.6. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration.

NOTE: If the waste contained no initial liquid phase, it is only necessary to filter enough extract to support the required analyses. However, if the waste contained an initial liquid phase, the entire contents of the extraction vessel must be filtered.

- 7.5.14 Prepare the TCLP extract as follows:

- 7.5.14.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Section 7.5.13 is defined as the TCLP extract. Proceed to Section 7.5.15.
- 7.5.14.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Section 7.5.13 with the initial liquid phase of the waste obtained in Section 7.5.8. This combined liquid is defined as the TCLP extract. Proceed to Section 7.5.15.
- 7.5.14.3 If the initial liquid phase of the waste, as obtained from Section 7.5.8, is not or may not be compatible with the filtered liquid resulting from Section 7.5.13, do not combine these liquids. Measure the volume of filtrate obtained in Section 7.5.13 and record in **Section IV** of the logbook. Individually analyze these two liquids, collectively defined as the TCLP extract, and combine the results mathematically, as described in Section 7.6.

- 7.5.15 Following collection of the TCLP extract, the pH of the extract should be measured and recorded (**Section IV**). Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH <2. All other aliquots must be stored under refrigeration (4°C) until analyzed.

- 7.6 The TCLP extract shall be prepared and analyzed according to appropriate analytical methods. TCLP extracts to be analyzed for metals shall be acid digested except in

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those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to $\pm 0.5\%$), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where: V_1 = The volume of the first phase (L).

C_1 = The concentration of the analyte of concern in the first phase (mg/L).

V_2 = The volume of the second phase (L).

C_2 = The concentration of the analyte of concern in the second phase (mg/L).

- 7.6 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality control requirements.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 1311 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are listed in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

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

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the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed

8.1 A minimum of one method blank for every 20 extractions performed using a particular batch of extraction fluid and per 20 extractions performed in a particular extraction vessel must be extracted and analyzed for the same contaminants as all associated samples. The method blanks are analyzed to check for laboratory contamination. A count of extractions performed in each extraction vessel is maintained in order to monitor the frequency of method blanks (1 per 20 extractions per vessel) required for each extraction vessel. .

8.1.1 After TCLP extraction, TCLP method blanks must undergo preparative extraction and analysis within method holding times (refer to Section 6.4). For this reason it may be necessary to extract more than one method blank using a particular batch of extraction fluid. For example, suppose that a sample requiring analysis for TCLP metals and semivolatiles is extracted using freshly prepared fluid from Batch 300. Because the fluid is new, a method blank is extracted with the sample and analyzed for the same components as the sample. Eight days later, a different sample requiring full TCLP analysis (metals, semivolatiles, pesticides, and herbicides) is extracted using fluid from Batch 300. Because the holding time for the previous TCLP method blank for pesticides and herbicides has expired, a new TCLP method blank must be extracted and analyzed for pesticides and herbicides. The new method blank need not be analyzed for metals and semivolatiles, because the first method blank that was prepared with fluid from Batch 300 has already been analyzed for these constituents.

8.1.2 Each TCLP method blank is identified in the TCLP extraction logbooks by a seven-character code. The first three characters are "PBT", which stands for "Preparation Blank - TCLP". Characters 4 through 6 consist of the three-digit preparation number of the extraction fluid. The seventh character is a letter, starting with "A" and proceeding alphabetically, which is unique to the extraction date for a particular batch of fluid. For example, "PBT316A" refers to the first TCLP method blank extracted using fluid from Batch 316; "PBT316B" refers to the second TCLP method blank extracted using the same fluid. The extraction date of each TCLP method blank is recorded in the TCLP Fluid Preparation and Use Logbook.

8.2 The laboratory recommends that a matrix spike be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level. Because the laboratory charges for the preparation and analysis of TCLP matrix spikes, selection of samples for TCLP matrix spiking is left to the discretion of the client. A minimum of one TCLP matrix spike must be analyzed for each batch of 20 TCLP extractions. As a minimum, follow the

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matrix spike addition guidance provided in each analytical method. Additional matrix spiking directions and guidance are provided in Table 4 and Figures 4 and 5.

8.2.1 Matrix spikes are to be added after filtration of the TCLP extract and before any preservation. Matrix spikes should not be added prior to TCLP extraction of the sample.

8.2.2 Instructions for preparing TCLP matrix spikes for metals analysis are contained in Table 4. Instructions for preparing TCLP matrix spikes for organics analyses are contained in Figures 4 and 5. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of TCLP extract as that which was analyzed for the unspiked sample.

8.2.3 Matrix spike recoveries are calculated by the following formula:

$$\text{Recovery (\%)} = 100 (X_s - X_u) / K$$

where: X_s = measured value for the spiked sample,
 X_u = measured value for the unspiked sample, and
 K = known value of the spike in the sample

8.2.4 The purpose of the matrix spike is to monitor the performance of the sample preparation and analytical methods used and to determine whether matrix interferences exist. Use of internal calibration methods (e.g. the method of standard additions [MSA]), modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration of the TCLP extract when the recovery of the matrix spike is below the expected analytical method performance. Metallic analytes must be quantitated by the method of standard additions if the TCLP matrix spike recovery for the analyte is less than 50% and the measured concentration of the analyte in the unspiked aliquot is within 20% of the regulatory level.

8.3 Each new analyst must demonstrate her/his ability to perform the method acceptably by while being witnessed by an analyst who is experience in performing the method. To successfully demonstrate the method, the analyst must perform the method in conformance with all the requirements of the SOP, referring to the SOP for guidance as necessary. In addition, each analyst must demonstrate the ability to produce TCLP Extraction Blanks that are free of contamination. This demonstration will require the analyst to collect and file the analytical results from four Extraction Blanks that he/she has generated.

8.4 All quality control measures described in the appropriate analytical methods shall be followed.

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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 Department Manager and with the QAO.

Refer to the current revisions of USEPA Method 1311 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, US EPA SW846, Third Edition, Final Update I (7/92), Method 1311

Federal Register, Volume 55, Number 126, Friday, June 29, 1990, PP 26986-26998

Federal Register, Volume 57, Number 227, Tuesday, November 24, 1992, PP 55114-55117

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TABLE 1
QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Toxicity Characteristic Leaching Procedure (TCLP)/ EPA 1311	Method Blanks	One per 20 samples extracted using a particular batch of extraction fluid.	Refer to individual analytical methods.	Prepare fresh extraction fluid and repeat TCLP extraction of all associated samples.
		One per 20 samples extracted in a particular extraction vessel.	Refer to individual analytical methods.	Remove extraction vessel from service.
	Matrix Spike	One per 20 TCLP extractions performed (required). One per waste type (suggested, left to discretion of client).	For metallic analytes, >50% if native analyte concentration is within $\pm 20\%$ of regulatory level. For other analytes, refer to appropriate analytical methods.	For metallic analytes, quantitate by method of standard additions. For other analytes, refer to appropriate analytical methods.
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	New analyst's performance of the method is witnessed by an experienced analyst. New analyst must produce method blanks that meet all method and laboratory acceptance criteria.	Repeat analysis until able to demonstrate acceptable performance of the method to witnessing analyst and by producing acceptable method blanks; document successful performance in personal training file.

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TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-510-05	EPA METHOD 1311
Reagents	Extraction Fluid #1 prepared using sodium hydroxide pellets.	Extraction Fluid #1 prepared using 1N sodium hydroxide solution.
QC - Method Blanks	Frequency of one method blank per 20 extractions performed using a particular batch of extraction fluid <u>and</u> per 20 extractions performed in a particular extraction vessel.	Frequency of one method blank per 20 extractions performed in a particular extraction vessel.
QC - Spikes	Matrix spike recommended for each waste type.	Matrix spike required for each waste type.

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

TABLE 3

TOXICITY CHARACTERISTIC CONSTITUENTS AND REGULATORY LEVELS

Constituent	Regulatory Level (mg/L)
Arsenic	5.0
Barium	100.0
Benzene	0.5
Cadmium	1.0
Carbon tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
Chromium	5.0
o-Cresol	200.0
m-Cresol	200.0
p-Cresol	200.0
Cresol	200.0
2,4-D	10.0
1,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
1,1-Dichloroethylene	0.7
2,4-Dinitrotoluene	0.13
Endrin	0.02
Heptachlor (and its hydroxide)	0.008
Hexachlorobenzene	0.13
Hexachloro-1,3-butadiene	0.5
Hexachloroethane	3.0
Lead	5.0
Lindane	0.4
Mercury	0.2
Methoxychlor	10.0
Methyl ethyl ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
Selenium	1.0
Silver	5.0
Tetrachloroethene	0.7
Toxaphene	0.5
Trichloroethylene	0.5
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl Chloride	0.2

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TABLE 4

TCLP MATRIX SPIKING FOR METALLIC ANALYTES

SPIKING INSTRUCTIONS			
Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 50 mL Final Volume (mL)
TCLP Matrix Spike (ICP)	CLPP-SPK-1	Inorganic Ventures	0.050
	CLPP-SPK-INT1	Lab Prepared (see below)	0.50
TCLP Matrix Spike (Mercury)	1000 ug/L Hg Standard	Prepared from 1000 mg/L stock standard	0.10

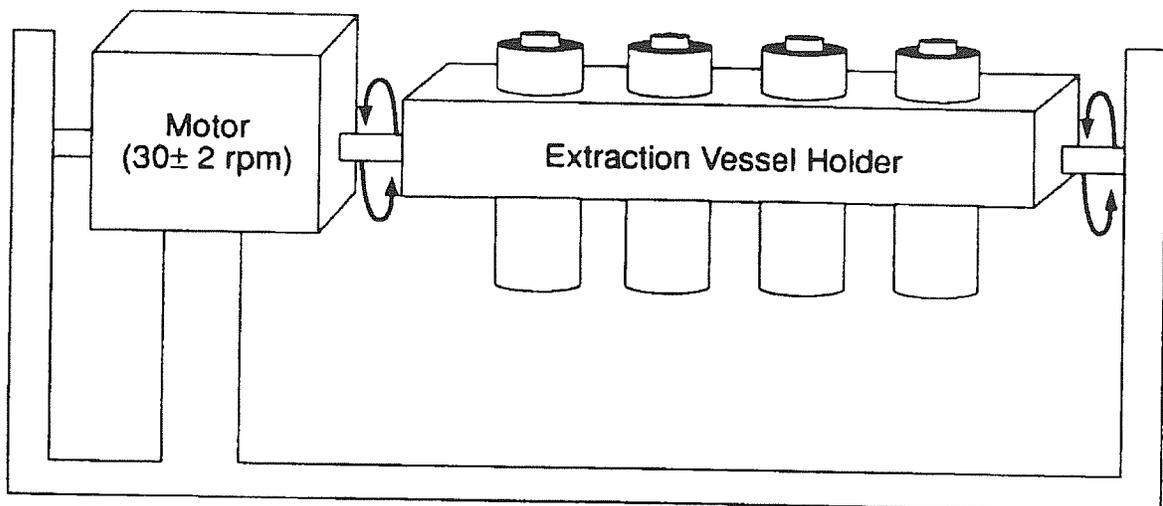
Note: Spiking must be performed after TCLP extraction and before preservation.

PREPARATION OF INTERMEDIATE SPIKING SOLUTIONS			
Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	QCP-CICV-3	Inorganic Ventures	10.0
	1000 mg/L Sb	High Purity Standards	5.0
	10000 mg/L K	High Purity Standards	10.0
	10000 mg/L Na	High Purity Standards	7.5
	10000 mg/L Mg	High Purity Standards	5.0
	10000 mg/L Ca	High Purity Standards	2.5
1000 ug/L Hg Standard	1000 mg/L Hg	Inorganic Ventures	0.10

ELEMENT CONCENTRATIONS IN MATRIX SPIKES AND SPIKING SOLUTIONS				
Element	CONCENTRATION IN SOLUTION, mg/L			
	TCLP Matrix Spike	CLPP-SPK-1	CLPP-SPK-INT1	1000 ug/L Hg Std.
Arsenic	2.000		200	
Barium	2.000	2000		
Cadmium	0.050		5	
Chromium	0.200	200		
Lead	0.500		50	
Selenium	2.000		200	
Silver	0.050	50		
Mercury	0.0020			1000

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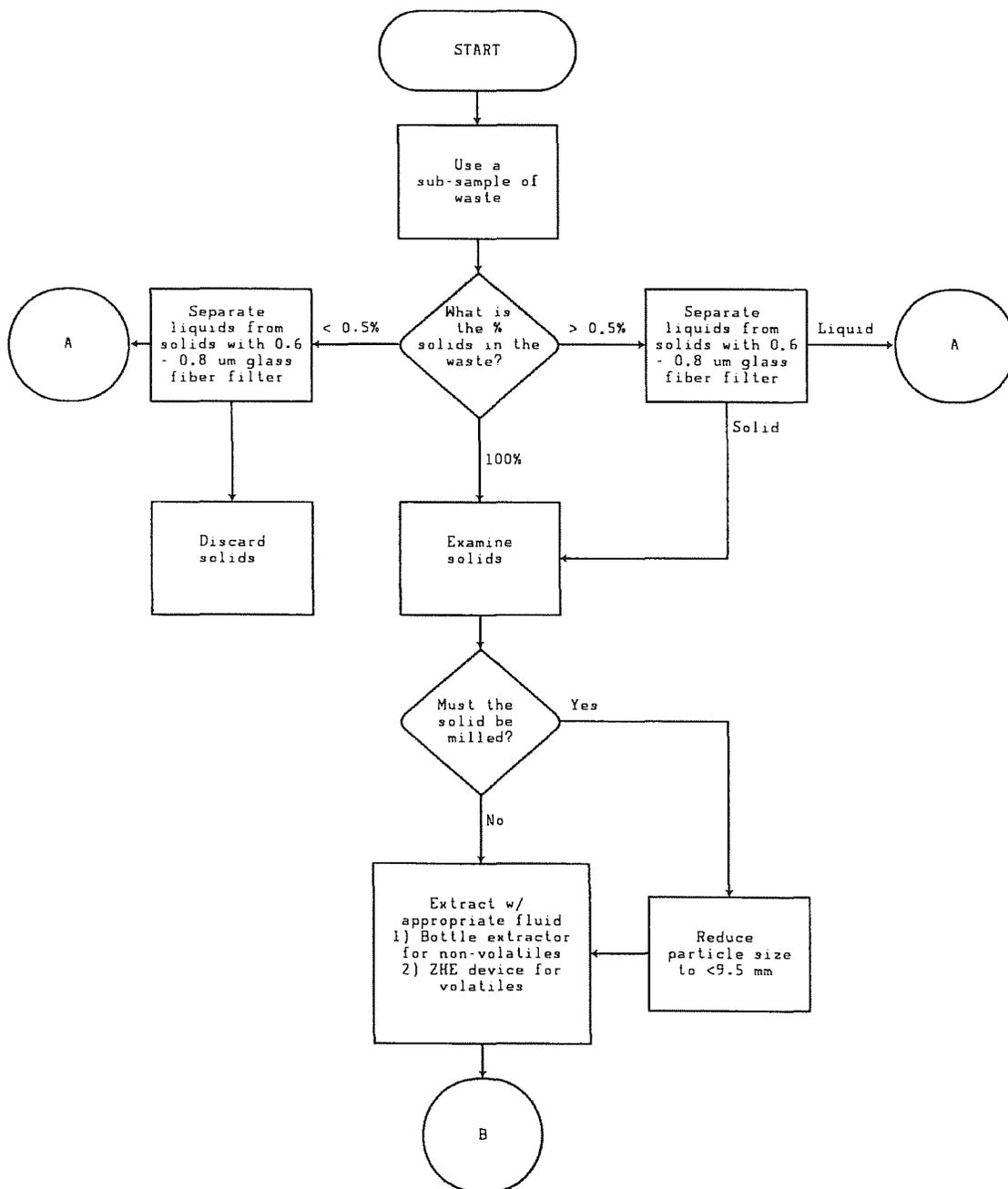
FIGURE 1
ROTARY AGITATION APPARATUS



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NON-VOLATILE ORGANIC ANALYTES

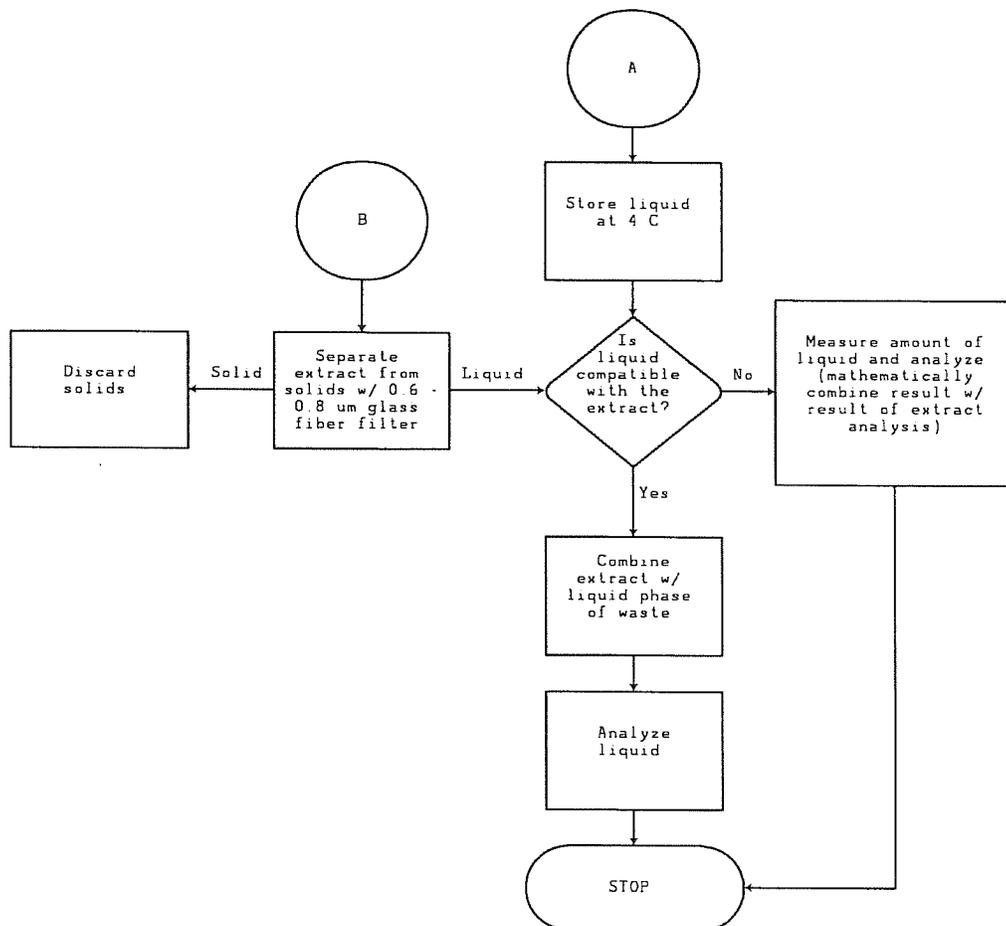
FIGURE 2

TCLP FLOW CHARTS



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FIGURE 3
TCLP FLOW CHARTS



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FIGURE 4

SVOA TCLP MATRIX SPIKE AND SURROGATE GUIDELINES

MATRIX SPIKE

The following compounds are reported for TCLP matrix spikes, although a full list spike solution is utilized (refer to SOP CA-502, current revision). Acid extractable compounds are at 100 ug/mL and base/neutral extractable compounds are at 50 ug/mL. 1.0 mL of this mix is added to the sample designated for the TCLP matrix spike.

Pyridine
1,4-Dichlorobenzene
2-Methylphenol
3-,4-Methylphenol*
Hexachloroethane
Nitrobenzene
Hexachlorobutadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol
2,4-Dinitrotoluene
Hexachlorobenzene
Pentachlorophenol

* Due to coelution on the GC/MS, 3-methylphenol and 4-methylphenol are reported as the combined concentration for the two isomers; the matrix spike solution contains 4-methylphenol at 100 ug/mL.

SURROGATE

The following surrogate compounds are reported for TCLP samples, although the surrogate mix also includes one additional surrogate (refer to SOP CA-502, current revision). Acid extractable surrogates are at 100 ug/mL and base/neutral extractable surrogates are at 50 ug/mL. 1.0 mL of this mix is added to all samples.

2-Fluorophenol	100 ug/mL
Phenol-d5	100 ug/mL
Nitrobenzene-d5	50 ug/mL
2-Fluorobiphenyl	50 ug/mL
2,4,6-Tribromophenol	100 ug/mL
Terphenyl-d14	50 ug/mL

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FIGURE 5

PESTICIDE TCLP MATRIX SPIKE AND SURROGATE GUIDELINES

MATRIX SPIKE

The following compounds are reported for TCLP matrix spikes, although a full list spike solution is utilized (refer to SOP CA-515, current revision). All compounds are at 0.5 ug/mL. 1.0 mL of this mix is added to the sample designated for the TCLP matrix spike.

Endrin
Heptachlor
Methoxychlor
Lindane
Heptachlor Epoxide

SURROGATE

Surrogates are at 1.0 ug/mL. 1.0 mL of this mix is added to all samples.

Decachlorobiphenyl (DCB)
Tetrachloro-m-xylene (TCMX)

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FIGURE 6

EXAMPLE PAGE FROM TCLP FLUID USE LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.					
Non-Volatile TCLP Extraction Fluid Preparation and Use Log					
FLUID PREPARATION					
TCLP Fluid # <u>1</u>			Fluid Batch # <u>662</u>		
Reagent	Manufacturer's Lot Number	Reagent Volume (mL)	Reagent Mass (g)	Fluid Final Volume (L)	
Glacial Acetic Acid	A13806	14.1	N.A.	20L	
Sodium Hydroxide		N.A.	51.4 g		
Preparation Date: <u>2/28/05</u> Prepared by: <u>JWM</u> Measured pH: <u>4.95</u>					
FLUID USE LOG					
KATAHDIN Sample Number	TCLP Extraction Start Date	Extract To Be Analyzed For:			
		Metals	SVOA	Pest	Herb
1) PBT662A					
2)					
3)					
4)					
5)					
6)					
7)					
8)					
9)					
10)					
11)					
12)					
13)					
14)					
15)					
16)					
17)					
18)					
19)					
20)					

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FIGURE 7

EXAMPLE PAGE FROM ROTARY EXTRACTOR RPM VERIFICATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

ROTARY EXTRACTOR RPM VERIFICATION LOGBOOK

EXTRACTOR #1: TOP SHELF SERIAL NUMBER: NONE
EXTRACTOR #2: MIDDLE SHELF SERIAL NUMBER: 1173
EXTRACTOR #3: BOTTOM SHELF SERIAL NUMBER: 1169

Please record the number of RPMs for each extractor each time they are used.

Date	Initials	Extractor #1	Extractor #2	Extractor #3	Comments
10/26/06	ARTB	out of service	50	30	Replaced fuse in Ext. #2
10/	ALL	out of service	29	Not Use	
11/01/06	DMF	↓	30	↓	
11/30/06	DMF	↓	not in use	30	
12/27/06	DMF	↓	↓	30	
01/09/07	DMF	↓	↓	30	
01/11/07	DMF	↓	↓	30	
01/22/07	DMF	↓	↓	29	
01/25/07	DMF	↓	↓	30	
01/29/07	DMF	↓	↓	30	
01/31/07	DMF	↓	↓	30	
02/06/07	DMF	↓	↓	30	
02/12/07	DMF	↓	↓	30	

Acceptance Range is 28-32 RPMS.
Meters Should Be Verified Against A Wrist Watch Annually And Recorded In The Comments Section.

TITLE: TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) FOR INORGANIC AND NON-VOLATILE ORGANIC ANALYTES

FIGURE 8

EXAMPLE PAGE FROM NON-VOLATILE TCLP EXTRACTION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
Non-Volatile TCLP / SPLP Extraction Log

Katahdin Sample No.: SC0805-70 Extraction Type (Check one): TCLP SPLP
Client: _____ Matrix: SC

I. SOLIDS DETERMINATION (Check one box below): Performed by: ASB on 2/23/09

100% wet solids - waste will obviously yield no liquid upon pressure filtration (proceed to Section II).
 <100% solids (perform solids determination below):

A. Weight of filter _____ g	G. Weight of liquid phase (F-B) _____ g
B. Weight of filtrate vessel _____ g	H. Percent wet solids [(E-G)/E x 100] _____ %
C. Weight of weigh boat + waste _____ g	Note: Steps I - L are optional. Refer to SOP.
D. Weight of weigh boat + residue _____ g	I. Weight of filter + dry solids _____ g
E. Total weight of waste (C-D) _____ g	J. Weight of dry solids (I - A) _____ g
F. Weight of filtrate vessel + filtrate _____ g	L. Percent dry solids (J/E x 100) _____ %

II. pH DETERMINATION AND FLUID SELECTION Performed by: ASB on 2/23/09

Initial pH of solid phase: 7.25 (For TCLP, if <5, proceed to Section III using TCLP Fluid #1. If >5, proceed to next step.)
TCLP only: pH after addition of 3.5 mL of 1 N HCl: 1.30 (If <5, use TCLP Fluid #1. If >5, use TCLP Fluid #2)
SPLP only: Sample from east (use SPLP Fluid #1) west (use SPLP Fluid #2) of Mississippi River.

III. EXTRACTION SETUP (Check one box below): Performed by: ASB on 2/23/09

100% wet solids: 2000 mL of Fluid # 1 (Batch 859) added to 1000 g unfiltered waste.
 <0.5% dry solids: _____ mL of waste filtered (filter sufficient volume to support all required analyses).
 >0.5% dry solids and <100% wet solids (perform phase separation below):
 _____ mL of Fluid # _____ (Batch _____) added to _____ g solid phase of waste.

M. Weight of filtrate vessel _____ g	Q. Weight of filtrate vessel + filtrate _____ g
N. Weight of weigh boat + waste _____ g	R. Weight of liquid phase (Q - M) _____ g
O. Weight of weigh boat + residue _____ g	S. Volume of liquid phase _____ mL
P. Total weight of waste (N - O) _____ g	T. Weight of wet solids (P - R) _____ g

Balance ID: OHUS Calvey 102
 Associated Extraction Blank ID: PB884L Fluid pH on day of use: 4.93
 Extraction Bottle ID: #21 Fluid expiration date: 2/19/10

IV. ROTARY EXTRACTION CONDITIONS (Rotary extraction not required if waste <0.5% dry solids)

Rotary extractor ID: #3
 Rotary extraction started: Date 2/23/09 Time 1515 Analyst ASB Room Temp. (degrees C) 17.7
 Rotary extraction completed: Date _____ Time _____ Analyst _____ Room Temp. (degrees C) _____
 Elapsed extraction time (HH:MM): _____ pH of extract after extraction: _____
 Rotary extraction filtered: Date _____ Analyst _____ Filter Lot Number _____
 Was pre-extn. filtrate from Section III combined with rotary extract (check one)? Yes No N.A.
 If no, enter the volume of filtrate obtained from the rotary extraction: _____ mL

This TCLP/SPLP extract to be analyzed for (check all that apply):
 Metals Semivolatiles Pesticides Herbicides Cyanide

Comments: _____

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: JCBanta Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10.24.00

General Manager: Debra 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	10.24.00	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**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-512-07**, titled **PREPARATION OF
SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT
EXTRACTABLE SEMI-VOLATILES ANALYSIS**.

Recipient: _____ Date: _____

I acknowledge receipt of copy ___ of document **SOP CA-512-07**, titled **PREPARATION OF
SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT
EXTRACTABLE SEMI-VOLATILES ANALYSIS**.

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for analysis of extractable semivolatile organic compounds. This SOP is specifically applicable to EPA Method 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

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

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

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

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 interferences, 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.

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- 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 3/4 ".
- 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

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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.

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- 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

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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.

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- 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).

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- 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

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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 to 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 to 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.

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- 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 $< 30^\circ\text{C}$. 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 $4 \pm 2^\circ\text{C}$ 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

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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 extraction 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 ₂ SO 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 ₂ SO 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		

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FIGURE 1
EXAMPLE OF LOGBOOK PAGE

SU-SON

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL SEMIVOLATILE

Extraction Method: (check one)	SWB45 3550 (SONIC) ✓	SWB45 3540 (SOX)	SWB45 3535 (ASE)
Analytical Method: (check one)	SWB45 8270 ✓	SWB45 3580 (OILS/WIPES)	OTHER
Standards	Surrogate ID (1): <i>SV2239</i>	Spike ID (1): <i>SV2231</i>	
	Surrogate ID (2):	Spike ID (2):	
Solvents	Solvent Lot # (Mec2): <i>G22E23</i>	Solvent Lot # (Acetone): <i>E46E42</i>	
Consumables	Filter Paper Lot # (SON) <i>1119164</i>	Filter Paper Lot # (KD) <i>J137505</i>	
Misc.	Nitrogen Bath Temperature: <i>34°C</i>	Sonicator Horns Tuned:	

Date Extracted	Ext. Inlet	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Final Vol. (mL)	Date Conc.	Tray Location	Initials	Comments
7-17-08	GN	W653548-1	30.01	1 mL	NR	1 mL	7-17-08	SV324 24	KF	R53707
		-2	30.03		1 mL			25		
		-3	29.97					26		
<hr/>										
									KF	7-17-08

QAEX147 0000067

Date Extracted	Ext. Inlet	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Final Vol. (mL)	Date Conc.	Tray Location	Initials	Comments
7-17-08	GN	SB3793-2	30.00	1 mL	NR	1 mL	7-17-08	SV324 27	KF	
		SB3801-1	29.99			2 mL		PP710 E3		soil chips
		-2	29.96					E4		soil chips
		-3	30.03					E5		
		SB3845-1	30.01			1 mL		SV324 28		Recy - SB3217-6
<hr/>										
									KF	7-17-08

Reviewed By: _____ Date: _____

QAEX147 0000068

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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
Benidine	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	

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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-Nitrosopiperidine
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	Pentachloronitrobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

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Prepared By: Mike Thomas Date: 8/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: J. Burton Date: 10/25/00

QA Officer: Dorothy J. Nadeau Date: 10-23-00

General Manager: Dennis F. Kufan 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	
02	Addition of SPE Procedure. Minor changes throughout. 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

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Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy _____ of document **SOP CA-515-05**, titled **PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS**.

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TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

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

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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

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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 interferences, further cleanup of the sample extract may be needed to minimize interferences.

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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
-

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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 (MeCL₂) - 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 decachlorobiphenyl (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

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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 applicable
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Any sample cleanup performed
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

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- 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 interference, 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.

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- 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 H₂SO₄ 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.

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- 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 H₂SO₄ 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

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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.

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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 assembly 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 H₂SO₄ 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 $\frac{3}{4}$ 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

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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

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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 \approx 1 mL hexane.

- 4.6 Reduce the extracts to \approx 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 \approx 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 N₂ 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

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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 each and every 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 each and every 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.

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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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TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-515-06	METHOD 3510, current revision
Apparatus/Materials	<ol style="list-style-type: none"> 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 	<ol style="list-style-type: none"> 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	<ol style="list-style-type: none"> 1. entire contents of 1 L sample bottle transferred to separatory funnel 	<ol style="list-style-type: none"> 2. one liter graduated cylinders used to transfer initial sample volume to separatory funnel
Procedures	<ol style="list-style-type: none"> 3. extract collection in amber bottle or Erlenmeyer flask 4. extract dried using Na₂SO₄ 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 	<ol style="list-style-type: none"> 2. extract collection in Erlenmeyer flask 3. extract dried using Na₂SO₄ 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		

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TABLE 2

SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)

TOPIC	KATAHDIN SOP CA-515-06	METHOD 3520, current revision
Apparatus/Materials	<ol style="list-style-type: none"> short-stem funnels 12 or 16 mL vials used for final extract 	<ol style="list-style-type: none"> drying columns 2 mL vials used for final extract
Reagents		
Sample preservation/ handling	<ol style="list-style-type: none"> entire contents of 1 L sample bottle transferred to CLLE 	<ol style="list-style-type: none"> one liter graduated cylinders used to transfer initial sample volume to CLLE
Procedures	<ol style="list-style-type: none"> CLLE for 18 + 2 hours extract dried using Na₂SO₄ in short stem funnels no apparatus height specification for concentration on water bath sample removed from water bath when volume reaches ~10 mL hexane added directly to K-D body at start of concentration process 	<ol style="list-style-type: none"> CLLE for 18-24 hours extract dried using Na₂SO₄ in drying columns partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min sample removed from water bath when volume reaches 1-2 mL 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	<ol style="list-style-type: none"> 1. no methanol addition 2. extraction using Horizon SPE-DEX 4790 Automated Extractor System. 3. extract dried using Na₂SO₄ 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 	<ol style="list-style-type: none"> 1. 5mL methanol added to all samples and blanks 2. extraction using Empore solid-phase extraction system 3. extract dried using Na₂SO₄ 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

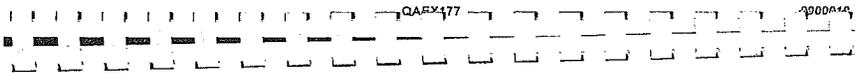
KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - AQUEOUS PESTICIDE/PCB

PIP sep

Extraction Method: (check one)	SW846 3520 (CLLE)	SW846 3510 (SEP) <input checked="" type="checkbox"/>	SW846 3535 (SPE)
Analytical Method: (check one)	SW846 8081 <input checked="" type="checkbox"/>	SW846 8082 <input checked="" type="checkbox"/>	EPA 808 <input type="checkbox"/> CLP OLM04.2 <input type="checkbox"/> CLP OLC2.1 <input type="checkbox"/> Other: <input type="checkbox"/>
Standards	Surrogate ID: 406654	Spike ID: 406665	Spike ID: 406663
Solvents	Solvent Lot # (Mez2): H4043	Solvent Lot # (Hexane): H22415	Solvent Lot # (Acetone):
Consumables	Filter Paper Lot # K11672365	Acid Lot # 435026	NaSO ₄ Lot # 27969003
Nitrogen Bath Temperature: 37°C	Val Lot #: 000978037 / 00098146		
Prep Start Time: 1400	Prep End Time: 1500	CLLE Start Time:	CLLE End Time:

Date Extracted	Ext. Inlt.	Sample ID	Initial Vol. ml	Sur. Vol.	Spike Vol.	Fraction		Final Vol. ml	Date Conc.	Trey Location	Initials	Clean-Up				Comments
						Pass	PCB					GPC	Flu.	Add Wash	Dist	
10/22/09	OB	W470360-1 W470360-1	1000	1ml	NR	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	1000	10-23-09	F5	GN	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	Rep. → R 112301
		W470360-2								F3						Rep. → R 112305
		-3								F6						
		-1	200		NR					F7						Prep 1123A - 11/10/09 by B. Visual in DI
		W470360-2	1000		1ml	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>			F4		<input type="checkbox"/>				
		-3				<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>			F9		<input checked="" type="checkbox"/>				
10-23-09 GN																

EX-002 - Revision 1 - 10/15/09



Date Extracted	Ext. Inlt.	Sample ID	Initial Vol. ml	Sur. Vol.	Spike Vol.	Fraction		Final Vol. ml	Date Conc.	Trey Location	Initials	Clean-Up				Comments
						Pass	PCB					GPC	Flu.	Add Wash	Dist	
10/22/09	OB	SC6328-1	200	1ml	NR	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	1000	10-23-09	F10	GN	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Prep 1123A - 11/10/09 by B. Visual in DI
		SC6340-2								F11						
		-6								F12						
		-8								F13						
		SC6345-11								A1						
		SC6413-1								A2						
		SC6433-15a	1060							A3						
		-16j	1040							A4		<input checked="" type="checkbox"/>				
		SC6457-2k	1060							A5						
		-3k	970							A6						
		-4L	1040							A7						
10-23-09 GN																

EX-002 - Revision 1 - 10/15/09

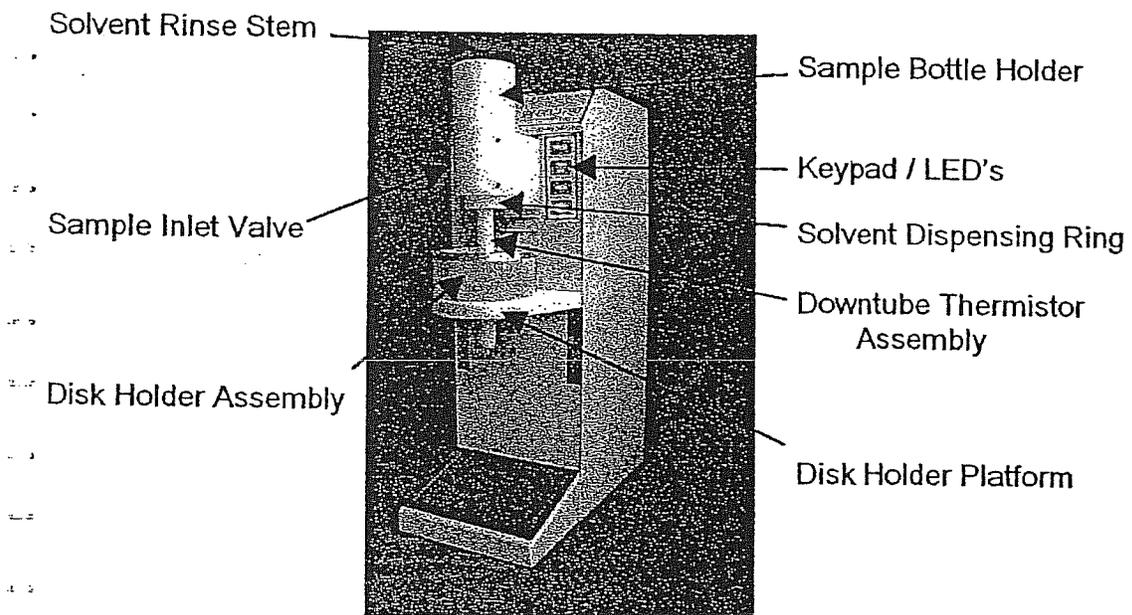
QAEX177

000011

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

FIGURE 2

HORIZON AUTOEXTRACTOR SYSTEM DIAGRAM



TITLE: PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS BY METHOD 8151

Prepared By: Keith Tanguay Date: 7/98

Approved By:

Group Supervisor: Michael J. Skoman Date: 1/26/01

Operations Manager: John C. Burton Date: 1/26/01

QA Officer: Dorothy J. Kadeau Date: 1.26.01

General Manager: Deanna F. Keenan Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8151A	Format changes, added pollution prevention, other minor changes to sections 7+8.	DK	1.26.01	1/26/01
02 8151A	Wording was added or changed to clarify sections 6, 7, 8 & 9. Minor changes throughout.	MRC	11.08.04	11.08.04
03 8151A	Sect. 7.0 - added information regarding TCLP samples. Small changes to reflect current practices. Updated Logbook page.	LAD	04/06	04/06
04 8151A	Sect. 7.4 & 7.10: changed glassware rinse solvent from ether to MeCl ₂ . Sect. 7.5: Sample volume determined by comparison to reference bottle & added 800ml of DI water is added to TCLP samples. Sect. 7.10 & 7.11: time shaken from 4 to 3 min. 7.17: removed old way of determining sample vol. 7.18: Added it may be necessary to add more Me ₂ SO. 7.22: changed Vol. Sample K _d to. 7.31: Added additional info to be recorded in logbook. Typos and formatting fixed.	LAD	03/08	03/08
05	Sect. 5.2 - Added wording to clarify acidification procedure. Sect. 7.13 - Removed necessity to reduce acid amt. for TCLP samples. Sect. 7.18 - added fume hood. Sect. 7.32 - added Me ₂ SO and NaCl ₂ lot #'s. Updated f.g. 1 - logbook page.	LAD	05/09	05/09

**TITLE: PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS - METHOD
8151**

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-516-05**, titled **PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS BY METHOD 8151**.

Recipient: _____ Date: _____

**KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE**

I acknowledge receipt of copy _____ of document **SOP CA-516-05**, titled **PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS BY METHOD 8151**.

Recipient: _____ Date: _____

**TITLE: PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS - METHOD
8151**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures, based on EPA SW 846 method 8151, used by Katahdin Analytical Services, Inc. technical personnel for the extraction of chlorinated phenoxy acid herbicides from aqueous samples such as surface, well and discharge waters. Detection limits are at the ug/L level or greater

1.1 Definitions

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.

DCAA: Dichlorophenylacetic acid

2,4-D: 2,4-Dichlorophenoxy acetic acid

ETHER: Diethyl ether- unpreserved

2,4,5-TP (Silvex): 2,4,5-Trichlorophenoxypropionic acid

2,4,5-T : 2,4,5-Trichlorophenoxyacetic acid

DIAZALD (a.k.a. Diazogen®): 99% (N-methyl-N-nitroso-p-toluenesulfonamide) See cautions in 1.3 Safety

CARBITOL: 2-(2-Ethoxyethoxy)ethanol

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of herbicides from aqueous samples. 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 herbicides from aqueous samples 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.

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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

This procedure requires the use of materials that, if handled improperly, pose a potential health risk to everyone in the laboratory. Follow instructions that describe the use of commercially available peroxide test strips for Diethylether. Special care must be taken when working with diazomethane.

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. (See cautions prior to 7.24.)

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 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 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 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.

To minimize ether exposure in the laboratory, allow glassware to air dry in a fume hood before bringing to dish washing area

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2.0 SUMMARY OF METHOD

Chlorinated phenoxy acids and their esters are initially exposed to an alkaline hydrolysis at pH>12 and serially extracted three times with methylene chloride to remove chlorinated hydrocarbons and phthalate esters. The hydrolyzed sample then undergoes a pH adjustment to pH<2 and is extracted with diethyl ether. The diethyl ether extract is collected in a 500 mL screw cap bottle that contains approximately 20 grams of acidified anhydrous sodium sulfate. After drying for a minimum of two hours, the extract is concentrated to 1 ml. The 1 ml extract is brought up to 4 mls with the addition of 1 ml of isooctane, 0.5 ml of methanol and 1.5 mls of diethyl ether. The 4 ml extract then undergoes diazomethane esterification (methylation) and is subsequently analyzed by GC-ECD. Compounds of interest are detected as methyl esters.

3.0 INTERFERENCES

Organic acids, especially chlorinated acids, cause the most direct interference. Phenols, including chlorophenols, also may interfere. Alkaline hydrolysis and subsequent extraction eliminate many of the predominant chlorinated insecticides. Because the herbicides react readily with alkaline substances, loss may occur if there is alkaline contact at any time except in the controlled alkaline hydrolysis step. Glassware and glass wool should be acid-rinsed and sodium sulfate (Na_2SO_4) should be acidified to minimize any alkaline contact.

4.0 APPARATUS AND MATERIALS

- 4.1 2 L Separatory Funnel, Teflon FEP with screw closures
- 4.2 Glass rod for crushing Na_2SO_4
- 4.3 pH paper (0-14)
- 4.4 gas tight volumetric syringes, 1.0 mL, 0.5 mL
- 4.5 mechanical separatory funnel shaker
- 4.6 Water/Steam bath (for K-D solvent evaporation) Organomation S-Evap Model 120
- 4.7 Kuderna-Danish apparatus:
 - Concentrator tube (or collector), 10 mL graduated
 - Evaporator flask, 500 mL
 - Three ball macro Snyder column

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- 4.8 Nitrogen blow-down apparatus (for concentrating extracts in 10 mL concentrator tubes)
- 4.9 Pasteur pipets, Pasteur pipettes, 5 ¾"
- 4.10 12 mL vials
- 4.11 500 mL sample bottles
- 4.12 Scoopula(s)
- 4.13 Graduated cylinders, 25 mL, 100 mL, 1000 mL
- 4.14 Diazomethane Generator (See figure 2)
- 4.15 Boiling chips, teflon, or silicon carbide, (carborundum, 2 mesh)
- 4.16 Clean sodium sulfate jars (~2Liters) for collecting the aqueous phase.

5.0 REAGENTS

- 5.1 Potassium hydroxide (37%): Prepare by dissolving 37 g of potassium hydroxide pellets in DI water and diluting to 100 mL.
- 5.2 Acidified Anhydrous Sodium Sulfate: Prepare by adding hexane to a 2.5 Kg jar of sodium sulfate crystals until the crystals are completely submerged. Measure 25mL of concentrated hydrochloric acid or sulfuric acid with a graduated cylinder and add it to the hexane saturated salt crystals. Quickly stir the mixture with a glass rod until the sodium sulfate is loose. Then decant the solvent layer and transfer the sodium sulfate on to a sheet of aluminum foil under a hood. Let dry overnight and then transfer back to the original jar. Label jar as acidified sodium sulfate. Record date and initials on jar. Cover and store at room temperature. Acidified anhydrous sodium sulfate will be referred to as sodium sulfate further in this SOP.
- 5.3 Sulfuric acid (1+3): Prepare by slowly adding 25 mL of sulfuric acid to 75 mL of DI water. This dilution should be done within an ice water jacket; store the diluted acid in a laboratory refrigerator.
- 5.4 Herbicide surrogate solution containing 5 µg/mL DCAA acid in acetone.
- 5.5 Underivitized Chlorinated Herbicide Stock Solutions: Contains 18 compounds at various concentrations - 100 ug/ml for all except for MCPA and MCPP which are at 10,000 ug/ml. Dilute to 5.0 ug/ml and 500 ug/ml with acetone.

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- 5.6 Diazald solution: weigh out ~6.8 grams of Diazald into a 50 mL volumetric flask and dissolve in a mixture of 25 mL carbitol and 25 mL of ether (50 mL of 1:1 v/v carbitol/ether). Bring to the volume mark only after all of the Diazald is in solution. Use the sonicator bath briefly if necessary. This solution is stable if kept at -10 - 20°C for one month.
- 5.7 Acetone: pesticides residue grade or equivalent.
- 5.8 Ether: pesticide residue grade or equivalent (unpreserved).
- 5.9 Methanol: pesticide residue grade or equivalent.
- 5.10 Carbitol: 2-(2-Ethoxyethoxy) ethanol
- 5.11 Hexane: pesticide residue grade or equivalent
- 5.12 Isooctane: pesticide residue grade or equivalent
- 5.13 Organic-free reagent water.
- 5.14 1+9 Hydrochloric Acid: For rinsing glassware (1 volume of HCL and 9 volumes of reagent water).
- 5.15 Silicic Acid: (H₂SiO₅) - 100 mesh powder.
- 5.16 10N Sodium Hydroxide
- 5.17 Sodium Chloride: (NaCl) - Pre-baked at 400°C for at least 4 hours.
- 5.18 Laboratory Reagent Grade Water

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Aqueous samples are collected in a 1L amber glass bottle. Samples are stored at 4 (±) °C until extraction.

Holding time for extraction of aqueous samples for Method 8151 is 7 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

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8151**

Store all extracts at 4°C (±2°C) 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

INITIAL EXTRACTION

- 7.1 Thoroughly rinse all glassware and separatory funnels to be used in this procedure with 1+9 HCL, then Laboratory reagent grade water, acetone and methylene chloride. (Note: All glassware used between this step and the Diazomethane esterification must be pre-rinsed in this fashion.) Use only glassware that has been acid rinsed.
- 7.2 Assign a separate quality control number to each blank and associated spike and record this information in the appropriate log book. One blank and at least one LCS should be extracted per batch.
- 7.3 Sample specific matrix spikes and matrix spike duplicates are extracted per client request or per project requirement. When the client does not specify sample QC, then the extractions lab will chose a sample for quality control (one set per 20 samples designated as MS/MSD) to extract and analyze. If sufficient volume of sample(s) is not available for an MS, MSD, the lab will extract an LSC, LCSD instead.
- 7.4 Assemble, label, and methylene chloride rinse a 2 liter separatory funnel with stopcock and closure, a 500 mL glass sample bottle, and a 2 Liter Na₂SO₄ jar for each sample including blank, lab control sample and lab control sample duplicate. Make sure no residual MeCl₂ is present on glassware.
- 7.5 Determine sample bottle volumes by comparing to reference bottle. Record sample volumes in logbook. Transfer the 1-L sample aliquot to a 2-L separatory funnel. For TCLP samples, transfer 200ml of sample to funnel using a pre-rinsed 1000 mL graduated cylinder. 800mL DI water is added to all TCLP samples.
- 7.6 Laboratory reagent grade water will serve as the method blank and lab control sample (LCS). For each blank and LCS, add 1000 mL of DI water to the separatory funnel using a clean 1000 mL graduated cylinder. 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 group supervisor.
- 7.7 Add 250g of NaCl to the samples and the QC. Seal and shake to dissolve the salt.

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- 7.8 Using a gas-tight volumetric syringe, add 1mL of herbicide surrogate solution to all of the samples, including blank and LCS's. Record surrogate number and amount added. Rinse syringes with acetone before and after use. Extreme accuracy should be used when measuring and adding spike and surrogate solutions! Double check the solution number and amount used.
- 7.9 Add 1mL herbicide spiking solution to LCS and MS/MSD, as required. Record spike number and amount added in appropriate logbook. Rinse syringes with methanol before and after use.

Hydrolysis

- 7.10 Add 17 mL of 10N NaOH to all 1-L samples (3.4ml to TCLP samples), seal, and shake. Using pH paper, adjust the pH to 12 or greater by adding more 10N NaOH if necessary. Shake for 3 minutes on mechanical shaker. Let samples sit for 20 minutes, shake for 3 minutes and repeat three times. This will complete the hydrolysis step.

Solvent Washes

- 7.11 Add 60mL of methylene chloride to the sample bottles, rinse the bottles and add the rinses to the separatory funnel. Extract the sample by vigorously shaking the funnel for 3 minutes, with periodic venting to release excess pressure. Allow the organic layer and the water layer to separate for a minimum of 10 minutes. Discard the methylene chloride layer.
- 7.12 Repeat step 7.11 two more times, discarding the methylene chloride layer each time.

Extraction

- 7.13 Add 17 mL of cold (4°C) 12N sulfuric acid to all samples, seal, and shake to mix. Using pH paper, adjust the pH of the sample to 2 or less than 2 by adding more 12N sulfuric acid if necessary.
- 7.14 Add 120 mL ether to each separatory funnel. Shake and vent until there is no more pressure build-up. Shake on mechanical shaker for 3 minutes. Allow the phases to separate for 10 minutes.
- 7.15 Collect the aqueous (bottom) layer in a 2 liter Na₂SO₄ jar, and the ether (top) layer in a 500 mL sample bottle containing about 20g of acidified Na₂SO₄. Cap and shake the ether layer and drying agent. Return the aqueous layer to the separatory funnel.

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- 7.16 Extract the aqueous layer two more times with 60 mL aliquots of ether as in steps 7.14-7.15. Combine ether (top layer) in the sample bottle. Prior to last extraction, rinse the 2-L Na₂SO₄ jar with ether and transfer to funnel to remove any remaining analytes.
- 7.17 Dispose the aqueous layer in the "N low" waste (sep-funnel waste) container. To minimize ether exposure in the laboratory, allow glassware to air dry in a fume hood overnight or rinse them with tap water before bringing to dish washing area.

Drying Step

- 7.17 Additional acidified Na₂SO₄ is added to the extract if it is not free flowing crystals or it is in a cake form. Shake the extract (ether phase) and the drying agent for one minute.
- 7.18 Allow the extract to remain in contact with the Na₂SO₄ for at least two hours, but, preferably stored overnight in the fume hood.

Note: The drying step is very critical to ensuring complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling. Neutralize the aqueous layer in a hood, then dispose in the Sep. Funnel waste container. To minimize ether exposure in the laboratory, allow glassware to air dry in a fume hood before bringing to dish washing area.

EXTRACT CONCENTRATION

- 7.21 Assemble a Kuderna-Danish apparatus with concentrator tube for each sample. Rinse the KD glassware with methylene chloride making sure no residual MeCl₂ is present on glassware before transferring samples.
- 7.22 Carefully, decant the extract into the K-D apparatus. Avoid allowing any acidified sodium sulfate crystals to fall in the concentration tube. Use a glass rod to crush any caked sodium sulfate in the glass jar. Rinse the glass jar 3 times with 10 mL of ether. Let drain between rinses. Thoroughly rinse funnel with ether and let drain.
- 7.23 Add 2 clean boiling chips to the K-D collector and attach a Macro-snyder column. Pre-wet the column with ether and place the K-D apparatus on the steam bath (which is heated no higher than 60°C). When the volume of liquid reaches approximately 2-4 mL, remove from the steam bath and allow to drain and cool for several minutes. Use caution, the ether will evaporate rapidly!

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- 7.24 Remove the column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of ethyl ether.
- 7.25 Concentrate the sample to 0.5 mL using the nitrogen blow-down apparatus.
- 7.26 Add 1.0 mL of isooctane and 0.5 mL of methanol. Rinse pipette and sides of tube to dilute to final volume of 4.0 mL with ether.

DIAZOMETHANE ESTERIFICATION

VERY IMPORTANT: Diazomethane is a toxic carcinogen and can explode under certain conditions! The following precautions MUST be followed:

- Use only a well ventilated hood. Do not breathe the vapors.
 - Do not heat above 90°C. Explosion may result.
 - Diazomethane must NEVER come into contact with ground glass surfaces as rough surfaces are proven initiators of detonations.
 - Avoid exposure of the solution to bright light - explosion may result.
 - Always use EXTREME caution when handling either diazomethane or Diazald.
- 7.27 Assemble the diazomethane bubbler (see Figure 2).
- 7.28 Add 5.0 mL of ether to tube 1. Add about 2.0-3.0 mLs of the Diazald solution (from 5.6) and 1.5 mL of 37% KOH to tube 2. Add the 37% KOH solution last to begin the reaction, and quickly cap both tubes.
- 7.29 Apply a nitrogen flow of 5-10 mL/min so bubbles emerge slowly. Bubble directly into the sample extract's concentrator tube. Allow the diazomethane to bubble through the sample for 1-2 minutes or until the yellow color persists. There is sufficient Diazald solution for esterification of two, maybe three, samples. Rinse exit tube with ether between samples.
- 7.30 Remove the exit tube from the KD concentrator tube, cover and store at room temperature for 20-30 minutes.
- 7.31 Destroy any unreacted diazomethane by adding 0.1 to 0.2 g silicic acid to the collectors. Allow to stand until the evolution of nitrogen is complete. Pipette the extract into a hexane rinsed 12 mL vial and rinse the collector twice with hexane (1-

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2 mL each time). Be careful to leave the silicic acid in the concentrator tubes. Bring to a final volume of 10mL using a reference vial, mix well, and let sit for a few minutes.

- 7.32 The extract is now ready for analysis. Make sure that initial volumes, intermediate volumes, aliquot volumes, and final volumes have been recorded in the logbook. Also verify that the surrogate and spike identification numbers and amounts used have been recorded as well as the Diazald identification number. The temperature of the water in the nitrogen evaporation water bath is recorded in the extraction logbook. The lot numbers of all of the solvents, acids and bases, sodium sulfate, sodium chloride, as well as all of the filter papers that are used in the extraction process are recorded in the logbook. Any deviations from the SOP or any abnormal sample observation should be noted as comments.
- 7.33 The data entered here are later used in calculations of the final result; see SOP CA-305, Analysis of Herbicides in Extracts of Water & Soil.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

- 8.1 The purity of the solvents used is checked on a lot number basis and is kept on file in the Extractions Laboratory.
- 8.2 A method blank and a laboratory control sample (LCS) must be extracted for each and every item listed below:
- Each sample matrix (water)
 - Each extraction method or level
 - Every batch of 20 samples, or fewer, extracted in a 24-hour period
- 8.3 A matrix spike (MS), and matrix spike duplicate (MSD) should be prepared every 20 samples.
- 8.4 Sample specific matrix spikes and matrix spike duplicates are extracted per client request or per project requirements. When the client does not specify sample QC, the extractions lab will choose one (per 20) samples for quality control to extract and analyze.
- 8.5 Surrogate and Spike acceptability criteria can be found in SOP CA-305, Analysis Of Chlorinated Herbicides by GC Using Methylation Derivatization: SW-846 Method 8151.

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- 8.6 If all quality control criteria are not met, appropriate steps must be taken to determine the cause. Problems indicate either matrix effect or an out of control event in the procedure.
- 8.7 Batch QC Requirements: If surrogates or spike compounds fail their criteria in the blank or lab control samples, the entire extraction batch is in question. A Corrective Action Report is initiated in the GC lab, and completed using information obtained from the Organics Prep Lab. If possible, the entire batch of samples is re-extracted with new QC samples. If no more sample can be obtained for re-extraction, any results reported must be flagged in the report and a narrative is included qualifying the data. Refer to SOP CA-305, Analysis of Chlorinated Herbicides by GC Using Methylation Derivatization: SW-846 Method 8151, for further details.

Each extraction 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 the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update III, December 1996, Method 8151A.

Katahdin SOP CA-305, Analysis of Chlorinated Herbicides by GC Using Methylation Derivatization: SW-846 Method 8151, current revision.

LIST OF TABLES AND FIGURES

Table 1	Summary of Method Modifications
Figure 1	Example of extraction logbook
Figure 2	Diazomethane Solution Generator

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TABLE 1

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-516-05	Method 8151, current revision
Apparatus/Materials	Use nitrogen blowdown technique.	Use two ball micro Snyder column.
Reagents	Unpreserved diethyl ether. 10N NaOH. Acidify sulfate in Hexane.	Ethyl ether preserved with BHT. 6N NaOH. Acidify sulfate in Ether.
Sample preservation/ handling		
Procedures	Shake for 3 minutes. Samples poured through acidified sulfate before KD.	Shake for 2 minutes. Samples poured through acidified glass wool before KD.
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

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FIGURE 1

EXAMPLE OF AQUEOUS HERBICIDE LOGBOOK PAGE

Herb sep

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - HERBICIDES

Extraction Method: (✓ one)	SEP. FUNNEL: ✓	SONICATION: <i>u, 1ml¹</i>	Analytical Method: SW846 8151 ✓
Standards	Surrogate ID (1): <i>60592</i>	Surrogate ID (2): <i>60592</i>	Spike ID (1): <i>60601</i> Spike ID (2):
Solvents	Meq2 Lot #: <i>H09277</i>	Acetone Lot #: <i>—</i>	Hexane Lot #: <i>E51619</i>
	Ether Lot #: <i>C1449</i>	Methanol Lot #: <i>610807</i>	Isooctane Lot #: <i>08064</i>
Consumables	Filter Paper Lot # (SON) <i>—</i>	Filter Paper Lot # (KD) <i>315884</i>	Powdered NaSO ₄ Lot # <i>—</i>
	Crystal NaSO ₄ Lot #: <i>2796707</i>	HCl Lot #: <i>074727</i>	H ₂ SO ₄ Lot #: <i>085688</i>
	NaOH Lot #: <i>624507</i>	Silicic Acid Lot #: <i>0992444</i>	Diazald ID: <i>620600</i>
	Potassium Hydroxide Lot #: <i>063405</i>	NaCl ₂ Lot #: <i>084362</i>	
Misc.	Nitrogen Bath Temperature:	Sonicator Home Tuned: <i>—</i>	Balance ID: <i>—</i>

Ext. Date	Ext. Vol.	Sample ID	Final Vol. / Weight (g)	Sum. Vol. (mL)	Spk. Vol. (mL)	Date Extracted	Date Conc.	Conc. Int.	Final Vol. (mL)	Tray Loc.	Comments
<i>5/12/09</i>	<i>CB</i>	<i>W663636-1</i>	<i>1000</i>	<i>1mL</i>	<i>NR</i>	<i>5/13/09</i>	<i>5/13/09</i>	<i>RF</i>	<i>10mL</i>	<i>RF 875</i>	<i>R 4985</i>
		<i>-2</i>			<i>1mL</i>					<i>C10</i>	
		<i>-3</i>								<i>C11</i>	
		<i>-4</i>	<i>200</i>		<i>NR</i>					<i>C12</i>	<i>RF 900A</i>
		<i>-5</i>								<i>D1</i>	<i>RF 900B</i>

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - HERBICIDES

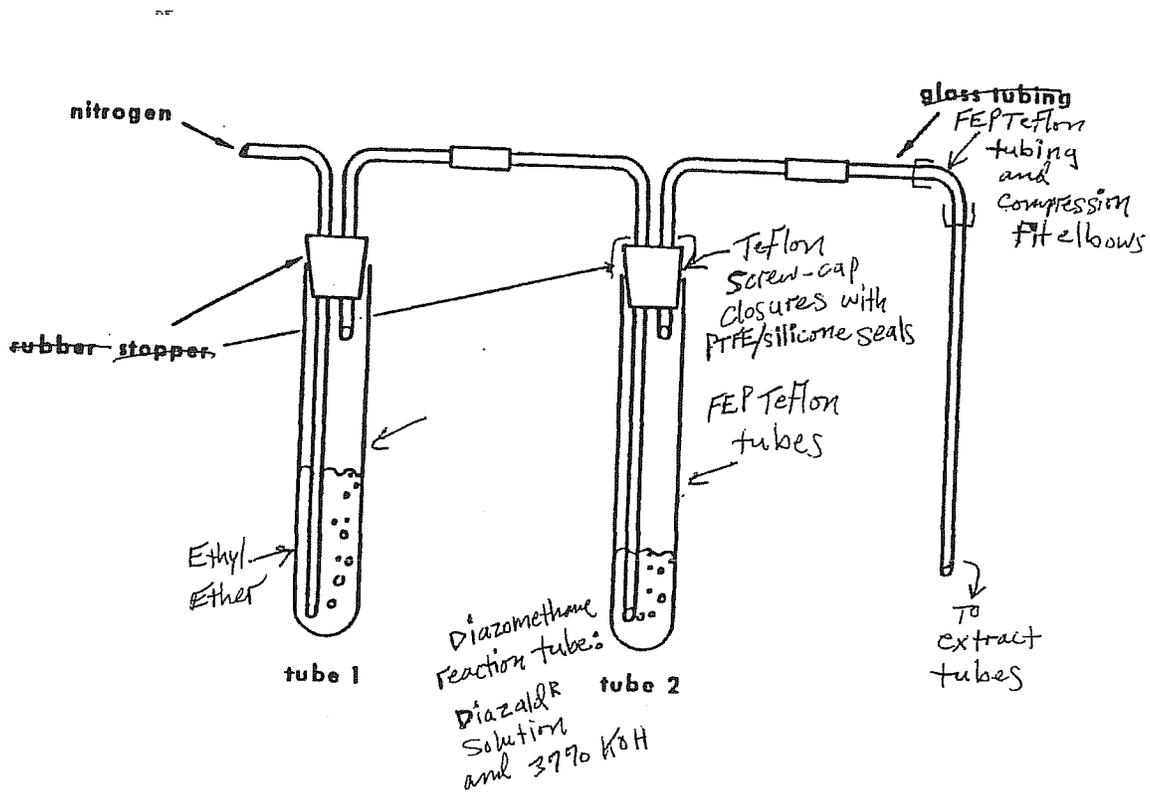
Ext. Date	Ext. Vol.	Sample ID	Final Vol. / Weight (g)	Sum. Vol. (mL)	Spk. Vol. (mL)	Date Extracted	Date Conc.	Conc. Int.	Final Vol. (mL)	Tray Loc.	Comments
<i>5/12/09</i>	<i>CB</i>	<i>SC2142-1b</i>	<i>200</i>	<i>1mL</i>	<i>NR</i>	<i>5/13/09</i>	<i>5/13/09</i>	<i>RF</i>	<i>10mL</i>	<i>RF 875</i>	<i>RF 900A to 1000mL in D1</i>
		<i>-2 f</i>								<i>D2</i>	<i>RF 900B</i>
		<i>SC2215-1b</i>	<i>1050</i>							<i>D4</i>	
		<i>SC2299-3a</i>	<i>200</i>							<i>D5</i>	<i>RF 900A to 1000mL in D1</i>

Reviewed By: _____ Date: _____

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FIGURE 2

DIAZOMETHANE SOLUTION GENERATOR



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: J. Senter Date: 11/15/00

QA Officer: Deborah J. Madreau Date: 11/16/00

General Manager: Denise F. Neffan Date: 11/20/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	EN	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 LND 6-26-06	Updated Sect. 7.0 to include SIM. Updated figures 2 and 3 to include current SVOC ^{& compounds} ANALYTES 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 LOD and MSD 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 consumable's lot #'s and recording the Nitrogen water bath temp. in logbook	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS

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

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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,

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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.

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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 ($\pm 5^{\circ}\text{C}$). 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 $\frac{3}{4}$ " 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.

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- 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-d ₆	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-d ₅	50 ug/mL
p-terphenyl-d ₁₄	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.

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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.

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Store all extracts at 4°C ($\pm 2^\circ\text{C}$) 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

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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

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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

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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.

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- 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 \approx 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 \approx 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 \approx 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 \approx 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 N₂ 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.
-

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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 semivolatiles for quality control acceptance criteria.

Each extraction 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.

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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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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	<ol style="list-style-type: none"> 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 Na₂SO₄ 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 	<ol style="list-style-type: none"> 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 Na₂SO₄ in drying columns 6. Wash the extractor flask and sodium sulfate column with 100 to 125 mL of extraction solvent to complete the quantitative transfer 7. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20min 8. Water bath at 15-20 deg C above solvent boiling point 9. Sample removed from water bath when volume reaches 1-2 mL
QC - Spikes	1. Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1. Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1. Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1. Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC – MDL		

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FIGURE 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-Nitrotropiperidine
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: 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. Burton Date: 1/22/01

QA Officer: Dorothy J. Madreau Date: 1-22-01

General Manager: Deanna F. Keegan Date: 1/22/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3010A	Format changes, added pollution prevention, block digester; revised database references; revised and added tables.	DN	1-22-01	1/22/01
02 3010A	Added wording allowing use of digesters for ICP-MS analysis. Added use of block digester as primary heating source & adjusted volumes. Revised standard solution names & concs. in Figures 3 & 4.	DN	8-29-02	8-29-02
03	Added Uranium to spiking solutions for LCS & MS/D. Removed the Internal Custody Record for Metals Digestates figure and reference.	LAD	04/06	04/06
04	Minor changes to Section 7 to reflect current practices. Updated Figure 1 - Sample Prep Logbook. Updated Figure 2 and 3 - Spike amounts.	LAD	05/09	05/09

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**.

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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.

TITLE: ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS

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

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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^oC. 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% HNO₃.

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- 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% HNO₃, 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 Repipettors (adjustable repeating pipettors with reservoirs) for dispensing concentrated nitric acid and 1:1 HCl.

5.0 REAGENTS

- 5.1 Concentrated nitric acid, HNO₃ – 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 HNO₃ 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.

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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 HNO₃ (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 HNO₃. 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.

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- 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.

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- 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.

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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 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

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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.

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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 HNO₃ 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 HNO₃.
9. Resume heating until gentle reflux action occurs.
10. Continue heating, adding additional HNO₃ 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 polyethylene 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 10.05 ml N/A : N/A
 CLPP-SPK-INT1 (ID/Vol): MW9925 10.5 ml Hot Plate No.: A
 CLPP-SPK-INT2 (ID/Vol): MW9930 10.5 ml Temp.: 95 °C
 (N-Spike (ID/Vol): MS1332 10.05 ml

REVIEWED
6/27/06
KATAHDIN ANALYTICAL
METALS SECTION

Spiking Witnessed by: N/A

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Wt/Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LC2WWF271CW1	WF271CW1	<u>0.05</u>	L	<u>0.05</u>	L	AQ	IC	DJJ	06/27/2006	N/A	N/A	N/A	N/A	N/A	N/A
LCSWWF271CW1	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006	N/A	N/A	N/A	N/A		
PBWVWF271CW1	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006	N/A	N/A	N/A	N/A		
WW3165-001	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-002	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-003	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-004	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-005	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-006	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-007	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-008	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-009	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-010	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-011	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-012	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-013	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-014	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-015	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-016	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-017	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-018	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-019	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-020	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						

DJJ 6/27/06

Digestion performed by: DJJ On: 6/27/06

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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

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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					

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. Burtis Date: 1/23/07

QA Officer: Doroah J. Nadeau Date: 1.23.01

General Manager: Deanna F. Wujcik 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 SO (cal. blank) followed by SI (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, ICRB, 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 MAU4 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

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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**.

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TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

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".

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

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

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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.

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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.

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5.0 REAGENTS

- 5.1 Hydrochloric acid, concentrated (HCl) – spectroscopic grade.
- 5.2 Nitric acid, concentrated (HNO₃) – spectroscopic grade.
- 5.3 Reagent water, trace metals free.
- 5.4 Calibration blank – reagent water containing HCl (5% v/v) and HNO₃ (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.

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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)

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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.

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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.

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- 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 successively 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

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calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for a CCB or ICB is greater than the PQL, sample results that are less than the PQL or greater than or equal to ten times the measured CCB concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

If DoD QSM acceptance criteria are being used, the absolute values of results of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed.

8.11 Interference check solutions ICSA and ICSAB (refer to Section 1.1) are analyzed at the beginning of each run to verify interelement correction factors and background correction. ICSA contains interferent elements (Al, Ca, Fe, and Mg) only, at concentrations of 200 mg/L to 500 mg/L. Results for interfering elements in the ICSA must fall within 80% to 120% of the expected values. Results for unspiked elements in ICSA must fall within \pm PQL if the PQL is greater than 0.01 mg/L, within \pm 2xPQL if the PQL is less than or equal to 0.01 mg/L. If DoD QSM acceptance criteria are being used, the absolute value of unspiked elements must be less than the LOD. ICSAB contains interferent elements at concentrations of 200 mg/L to 500 mg/L, and analytes at concentrations of 20 mg/L or less. Results for all elements (interferents and analytes) in ICSAB must fall within 80% to 120% of the expected values. If the ICSA or ICSAB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICSA or ICSAB has been analyzed.

8.12 A Practical Quantitation Limit (PQL) Check Standard or low level continuing calibration verification (LLCCV) is analyzed at the beginning (after the ICV and ICB samples) and at the end of each run. Element concentrations in this solution are at the laboratories practical quantitation limit. Element recoveries for the PQL check Standard must fall between 70-130% of the expected values. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run, unless the PQL Check Standard recovery is greater than 130% and the samples results are less than the PQL.

If DoD QSM acceptance criteria are being used, recoveries must fall between 80-120%. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run.

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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 $\frac{1}{2}$ 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 $\frac{1}{2}$ PQL for DoD), associated sample results that are less than the PQL (less than $\frac{1}{2}$ 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.

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The relative percent difference between sample duplicate, matrix spiked duplicate or LCS duplicate, is calculated as follows:

$$\text{RPD (\%)} = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100$$

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:

$$\text{Difference (\%)} = \frac{|L-S|}{S} * 100\%$$

where:

L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

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

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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 Maintenance

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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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 $\pm 10\%$ 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 \geq PQL and $10\times <$ 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 $\pm 10\%$ 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 \geq PQL and $< 10\times$ 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 $\pm 30\%$ 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 $\pm 20\%$ of true value. For analytes not spiked, \pm PQL, or, if $PQL \leq 0.01$ mg/L, $\pm 2\times$ 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.

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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 $\pm 20\%$ 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 \geq 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 $1/2$ 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 $\pm 20\%$ of true value, unless vendor-supplied or statistical limits have been established. DoD: Recovery within $\pm 20\%$ 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 $\pm 25\%$ 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 $\pm 25\%$ of true value, if sample $< 4x$ spike added. DoD: Use QC acceptance criteria specified by DoD for LCS. RPD $\leq 20\%$ for duplicate spikes and sample duplicates.	1) Flag results. 2) DOD: Perform additional project specific quality control tests.

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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 $\pm 10\%$ 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 $\pm 25\%$.	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 succeeding higher stds until recovery <u>not</u> within $\pm 10\%$. 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: \pm 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

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

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

Element	CONCENTRATION IN SOLUTION, mg/L								
	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

Element	CONCENTRATION IN SOLUTION, mg/L			
	ICP Intermed STD		PQL-INT	ICSAB-INT
Aluminum	240		30	
Antimony			0.8	
Arsenic			0.8	
Barium			0.5	
Beryllium			0.5	
Boron			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	

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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	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

ATTACHMENT 1

HARDNESS BY CALCULATION

As referenced in "Standard Methods for the Examination of 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:

$$\text{Total Hardness, mg equivalent CaCO}_3/\text{L} = 2.497 (\text{Ca, mg/L}) + 4.118 (\text{Mg, mg/L})$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent CaCO}_3/\text{L} = 2.497 (\text{Ca, mg/L})$$

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Prepared By: George Brewer Date: 01/01

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: John C. Benton Date: 1/29/01

QA Officer: Dorothy J. Kadeau Date: 1-29-01

General Manager: Deanna F. Keenan Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
00 7470A	NA	EN	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	LAN	02-16-05	02-16-05
02	Updated Fig. 1 - new prep logbook page	LAN	04/08	04/08
03	Updated Figure 1 - Example of a mercury preparation logbook page.	LAN	03/09	03/09
04	Added LOD definition. Updated sections 8, 9, 10 and Table 1 for DOD QSM version 4.1 compliance.	EN	08/09	08/09

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Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-615-04**, titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**.

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TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY
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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.

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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

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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.

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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 Repipettors (adjustable repeating pipettors 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

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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

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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.

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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.

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- 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 preparation 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

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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.

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7.15.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 3. A linear regression program may be used to obtain the intercept concentration.

7.15.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

- The apparent concentrations from the calibration curve must be linear over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised.
- The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.
- The determination must be free of spectral interference and corrected for nonspecific background interference.

DATA REDUCTION AND REPORTING

7.16 Results are obtained in concentration units (ug/L) from the instrument. Electronic instrument data files are imported into the Metals ACCESS database for data reduction. Sample preparation information (initial sample volumes and final digestate volumes) are entered directly into the Metals ACCESS database to allow calculation of final results for reporting. Results are calculated as follows:

$$\text{Mercury concentration (ug/L)} = \frac{\text{MC} \times \text{DF} \times \text{IV}}{\text{FV}}$$

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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.

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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.

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- 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 $\frac{1}{2}$ 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 $\frac{1}{2}$ PQL for DoD), associated sample results that are less than the PQL (less than $\frac{1}{2}$ 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

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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:

D₁ = Spike sample result
D₂ = 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

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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
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TABLE 1
QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA 7470	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ≥ 0.995 .	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within $\pm 10\%$ of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL. DoD: Less than LOD	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within $\pm 30\%$ 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 $\pm 20\%$ 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 $\geq PQL$ 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 $1/2$ 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 $\pm 20\%$ of true value.	Redigest all affected samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery $\pm 25\%$ of true value, if sample $> 4x$ spike value.	Flag results.
Matrix Spike Duplicate Sample (P)	One per digestion batch of 20 or fewer samples.	1) Recovery $\pm 25\%$ of true value, if sample $< 4x$ spike added. 2) RPD $\leq 20\%$ for duplicate spikes.	Flag results	

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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.		

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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

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FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK

Katahdin Analytical Services, Inc. Metals Preparation Benchsheet

Reagent Information:
 JT Baker HNO3: 6241034 JT Baker HCL: N/A JT Baker H2SO4: 620022 Method: 7470
 JT Baker KMNO4: MCEI JT Baker K2S2O8: MCEI JT Baker NH2OH-HCl: MCEI

Standards/Spike Information:
 1 ppm A: 11566 1000 uL of 1 ppm A to 100 mL
 1 ppm B: 11566 200 uL of 1 ppm A to 100 mL
 LCSW = 125 uL of 1 ppm A to 25 mL S0.5 = 50 uL of 1 ppm A to 100 mL
 Spike(S/P) = 25 uL of 1 ppm A to 25 mL S1.0 = 100 uL of 1 ppm A to 100 mL

Balance ID: N/A Water Bath ID: B Thermometer ID: ACC-5
 Digestion Start Time (@ 96 °C): 13:50 Digestion End Time (@ 95 °C): 15:30

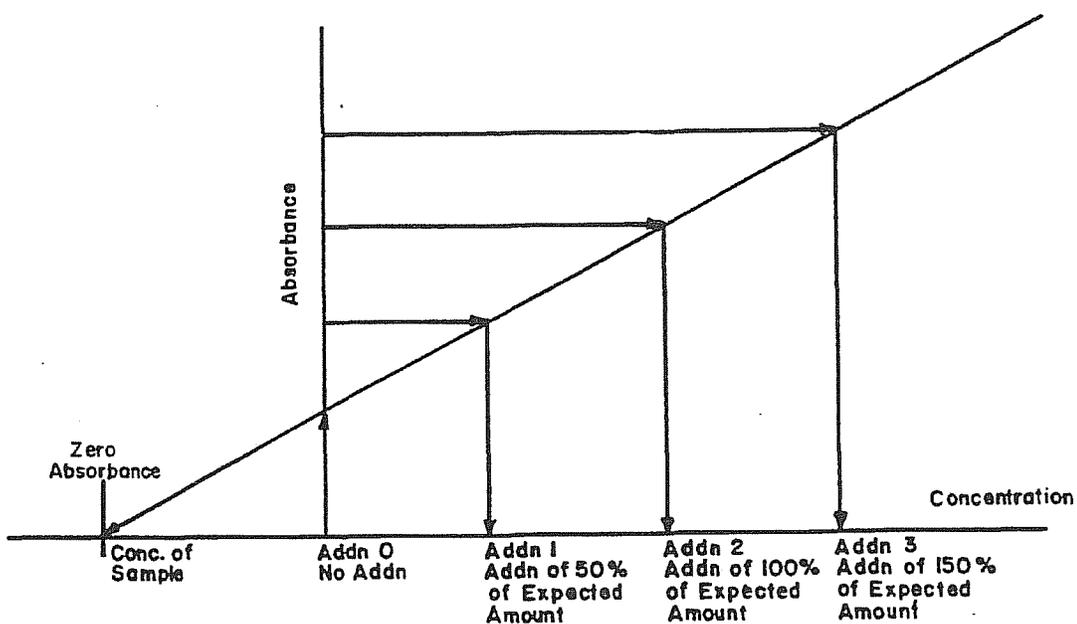
Sample ID	Batch ID	Initial Wt/Vol	Initial Vol	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LCSWZB24HGWO	ZB24HGWO	<u>0.225</u>	<u>L</u>	<u>0.225</u>	<u>L</u>	AQ	HG	DWM	02/24/2009	N/A	N/A	N/A	N/A		
PBWZB24HGWO	ZB24HGWO		<u>L</u>		<u>L</u>	AQ	HG	DWM	02/24/2009	N/A	N/A	N/A	N/A		
SC0797-001T	ZB24HGWO		<u>L</u>		<u>L</u>	AQ	HG	DWM	02/24/2009						
SC0805-007T	ZB24HGWO		<u>L</u>		<u>L</u>	AQ	HG	DWM	02/24/2009						
SC0834-013T	ZB24HGWO		<u>L</u>		<u>L</u>	AQ	HG	DWM	02/24/2009						
SC0835-002T	ZB24HGWO		<u>L</u>		<u>L</u>	AQ	HG	DWM	02/24/2009						<u>E</u>
SC0835-001T	ZB24HGWO		<u>L</u>		<u>L</u>	AQ	HG	DWM	02/24/2009						<u>E</u>
SC0846-013	ZB24HGWO		<u>L</u>		<u>L</u>	AQ	HG	DWM	02/24/2009						<u>E</u>
SC0858-001T	ZB24HGWO		<u>L</u>		<u>L</u>	AQ	HG	DWM	02/24/2009						
SC0858-002T	ZB24HGWO		<u>L</u>		<u>L</u>	AQ	HG	DWM	02/24/2009						
SC0868-013	ZB24HGWO		<u>L</u>		<u>L</u>	AQ	HG	DWM	02/24/2009						<u>E</u>
SC0868-015	ZB24HGWO		<u>L</u>		<u>L</u>	AQ	HG	DWM	02/24/2009						<u>E</u>
SC0834-013T			<u>L</u>		<u>L</u>										

Dum 2-25-09

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY
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FIGURE 2

STANDARD ADDITIONS PLOT



TITLE: pH CONCENTRATION MEASUREMENTS IN AQUEOUS SAMPLES

Prepared By: Wet Chemistry Date: 2/97

Approved By:

Group Supervisor: Keith Farquhar Date: 02/30/01

Operations Manager: John C. Burton Date: 2/13/01

QA Officer: Dorothy J. Nadeau Date: 2/13/01

General Manager: Debra F. Huffman Date: 2/13/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 9040B	Format changes, added pollution prevention database and operation of Accumet pH meter.	EN	2/13/01	2/13/01
02 9040B	Revised section 6.0.	EN	5/23/01	
03 9040B	Replaced applicable database w/ KIMS replaced figures 1 and 2 added wording to section 4 minor changes throughout.	UAN	11/29/04	11/29/04
04	Updated logbook and batch sheet examples.	UAN	05/09	05/09

TITLE: pH CONCENTRATION MEASUREMENTS IN AQUEOUS SAMPLES

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-708-04**, titled **pH CONCENTRATION MEASUREMENT IN AQUEOUS SAMPLES**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **SOP CA-708-04**, titled **pH CONCENTRATION MEASUREMENT IN AQUEOUS SAMPLES**.

Recipient: _____ Date: _____

TITLE: pH CONCENTRATION MEASUREMENTS IN AQUEOUS SAMPLES

1.0 SCOPE AND APPLICATION

This SOP describes the procedure used by Katahdin Analytical Services technical personnel for performing pH calibrations and measurements for buffer solutions and aqueous sample determinations as described in EPA Method 150.1, SM 4500-H⁺ B, and SW 846 Method 9040.

This procedure is applicable to all sample pH measurements within the Katahdin laboratory, as well as any buffer solution preparations or sample pH adjustments necessary for preservation, distillation or titration.

1.1 Definitions

pH - A measure of the acidity or alkalinity of a solution, defined as $-\log [H^+]$.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of aqueous pH by Methods 150.1, SM 4500-H⁺ B, and SW 846 Method 9040. Each analyst must demonstrate and document his/her ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of pH by Methods 150.1, SM 4500-H⁺ B, and SW 846 Method 9040 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

TITLE: pH CONCENTRATION MEASUREMENTS IN AQUEOUS SAMPLES

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 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 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 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 Environmental Health and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

The pH of a sample is determined electrometrically using a glass electrode in combination with a reference potential or a combination electrode. The procedure described in this SOP uses a combination electrode.

3.0 INTERFERENCES (taken from EPA Method 150.1 and SW 846 9040B)

The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants or high salinity.

Sodium error at pH levels greater than 10 can be reduced or eliminated by using a "low sodium error" electrode.

Temperature effects on the electrometric measurement of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference can be controlled with instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second source is the change of pH inherent in the sample at various temperatures. This error is sample

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dependent and cannot be controlled. It may therefore be noted by reporting both the pH and temperature at the time of analysis.

Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by laboratory reagent grade water rinsing. An additional treatment with hydrochloric acid (1:10) may be necessary to remove any remaining film.

4.0 APPARATUS AND MATERIALS

- 4.1 pH Meter (Accumet® pH/Conductivity Meter, Model 20 or equivalent) with automatic temperature compensation (ATC)
 - 4.2 Plastic dose cups
 - 4.3 Teflon-coated stir bars
 - 4.4 Stir bar retriever
 - 4.5 Magnetic stir plate
 - 4.6 Thermometer
-

5.0 REAGENTS

- 5.1 Buffer solutions (pH 2.0, 4.0, 7.0, 10.0, 12.0), traceable to NIST reference material
 - 5.2 Laboratory reagent grade water
-

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected in plastic or glass jars and stored at 4°C until analysis.

pH samples require immediate analysis upon receipt by the laboratory. From EPA Method 150.1: High-purity waters and waters not at equilibrium with the atmosphere are subject to changes when exposed to the atmosphere, therefore the sample containers should be filled completely and kept sealed prior to analysis.

Katahdin project managers will remind clients doing compliance monitoring that, in order to meet the regulatory requirements for holding times, a field pH is required. An example is compliance work originating in South Carolina and requesting SW846 method 9040B. If

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requested to perform a laboratory pH, the analysis must be performed as soon as possible and the data must be notated as being performed out of hold time.

7.0 PROCEDURES

7.1 NORMAL RANGE CALIBRATION (pH range 3.5 - 10.5)

- 7.1.1. Meter should be calibrated daily. As described in the following steps, conduct a two-point calibration with pH buffers 4 and 10. Perform a calibration check using pH 7 buffer. The source/lot number of each solution at the time of analysis must be recorded in the logbook (Figure 1).
- 7.1.2. Dispose of buffer solutions used the previous day (see Waste Disposal, section 1.4). Put about 20 mL of the appropriate buffer solution into new dose cups. Place a tiny stir bar in each new cup.
- 7.1.3. Rinse probe (i.e., electrode) with laboratory reagent grade water. Gently blot dry with kimwipe.
- 7.1.4. Place pH 4 buffer on stir plate. Turn stir plate on so that the stir bar spins without creating a vortex. Push 1 key (meaning add a standard). Then push 4 (meaning calibrate). Record the value in the pH logbook.
- 7.1.5. Remove pH 4 buffer. Rinse probe. Blot dry.
- 7.1.6. Repeat step 7.1.4 with the pH 10 buffer. Record the value in the pH logbook. Remove pH 10 buffer. Rinse and dry probe.
- 7.1.7. With the pH 7 buffer, repeat step 7.1.4, but DO NOT press any keys as this reading is a calibration check. Record the reading in pH logbook. Results must be within 0.05 pH units of the true value for analysis to proceed.

NOTE: If buffer readings are not within 0.05 pH units of expected values (4.00, 7.00 and 10.00) the electrode may need cleaning. Rerun and enter in the pH lab notebook that the meter was recalibrated with the pH 4 and 10 buffers, with the pH 7 buffer used as a calibration check. Also note any maintenance performed.

7.2. LOW RANGE CALIBRATION

- 7.2.1. If pH of a sample as determined in Section 7.4 is less than 3.5, the instrument must be recalibrated using buffers that bracket the expected pH of the sample, as described below. The source/lot number and temperature of each solution at the time of analysis must be recorded in the logbook (Figure 1).

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- 7.2.2 Rinse probe (i.e., electrode) with laboratory reagent grade water. Gently blot dry with kimwipe.
- 7.2.3 Place pH 2 buffer on stir plate. Turn stir plate on so that the stir bar spins without creating a vortex. Push *1* key (meaning add a standard). Then push *4* (meaning calibrate). Record the value in the pH logbook.
- 7.2.4 Remove pH 2 buffer. Rinse probe. Blot dry.
- 7.2.5 Repeat step 7.2.3 with the pH 7 buffer. Record the value in the pH logbook. Remove pH 7 buffer. Rinse and dry probe.
- 7.2.6 With the pH 4 buffer, repeat step 7.2.3, but DO NOT press any keys as this reading is a calibration check. Record reading in pH logbook. Results must be within 0.05 pH units of the true value for analysis to proceed.

NOTE: If buffer readings are not within 0.05 pH units of expected values (2.00, 4.00 and 7.00) the electrode may need cleaning. Rerun and enter in pH lab notebook that meter was recalibrated with the pH 2 and 7 buffers, with the pH 4 buffer used as a calibration check. Also note any maintenance performed.

7.3 HIGH RANGE CALIBRATION

- 7.3.1 If pH of a sample as determined in Section 7.4 is greater than 10.5, the instrument must be recalibrated using buffers that bracket the expected pH of the sample, as described below. The source/lot number and temperature of each solution at the time of analysis must be recorded in the logbook (Figure 1).
- 7.3.2 Rinse probe (i.e., electrode) with laboratory reagent grade water. Gently blot dry with kimwipe.
- 7.3.3 Place pH 7 buffer on a stirplate. Turn stir plate on so that the stir bar spins without creating a vortex. Push *1* key (meaning add a standard). Then push *4* (calibrate). Record the value in the pH logbook.
- 7.3.4 Remove pH 7 buffer. Rinse probe. Blot dry.
- 7.3.5 Repeat step 7.3.3 with the pH 12 buffer. Record the value in the pH logbook. Remove pH 12 buffer. Rinse and dry probe.
- 7.3.6 With the pH 10 buffer, repeat step 7.3.3, but DO NOT press any keys as this reading is a calibration check. Record reading in pH logbook. Results must be within 0.05 pH units of the true value for analysis to proceed.

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NOTE: If buffer readings are not within 0.05 pH units of expected values (7.00, 10.00 and 12.00) the electrode may need cleaning. Rerun and enter in pH lab notebook that meter was recalibrated with the pH 7 and 12 buffers, with the pH 10 buffer used as a calibration check. Also note any maintenance performed.

7.4 ANALYSIS OF SAMPLES

- 7.4.1 Sample analysis may proceed once the meter has been calibrated for the day with two buffers that bracket the expected pH of the sample, and after checking the calibration with a mid-range solution between the two buffers used for calibration of the meter (refer to sections 7.1, 7.2, and/or 7.3 as applicable).
- 7.4.2 For the range being used, rerun the appropriate calibration check point as the laboratory control sample (LCS) for the analytical batch. An LCS is required at the beginning of every batch of twenty or fewer samples.
- 7.4.3 Record date, time and initials for this analytical session.
- 7.4.4 Samples should be equilibrated to room temperature prior to analysis (i.e., at the same temperature as the calibration buffers, ± 2 °C). A more accurate pH reading will be achieved when the buffers and the samples are at the same temperature. However, the Accumet® pH meter is equipped with automatic temperature compensation (ATC) for when samples and buffers are not at the same temperature. Refer to the Accumet® Model 20 pH/Conductivity Meter operating Instructions, #300143.3 (Revision C) for information on the ATC probe.
- 7.4.5 Shake sample well. Pour about 25 ml into a clean dose cup. Place a tiny stir bar in cup. Place on stir plate, turn on stir plate and immerse probes.
- 7.4.6 When meter locks, record value displayed.
- 7.4.7 Rinse probe with laboratory reagent grade water and blot dry between samples.
- 7.4.8 Place probe in pH 4 buffer solution to store until next analysis.

7.5 CLEANING PROCEDURE

If an electrode becomes coated with an oily material that will not rinse free, the electrode can either (refer to instrument manual):

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- be cleaned with an ultrasonic bath, or
- be washed with detergent, rinsed several times with laboratory reagent grade water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with laboratory reagent grade water.

7.6 REPORTING AND CALCULATIONS

- 7.6.1 Final pH values are reported to one decimal place (i.e. report three significant figures for pH readings over 10.0 and two significant figures for pH readings less than 10.0).
- 7.6.2 When a sample duplicate is analyzed, both the original result and duplicate result are recorded in the pH logbook; however, the original sample result is to be reported to the client.
- 7.6.3 After completion of each test, the logbook must be signed and dated by the person performing the test. All unused lines are to be "z-ed" out and initialed and dated.
- 7.6.4 The sample data results, with any appropriate notations, are entered into KIMS by the analyst. A batch sheet is generated (Figure 2). Raw data and batch sheets are reviewed for completeness and accuracy by the Inorganic Department Manager or other qualified designee.
- 7.6.5 All batch sheets and copies of the raw logbook data are filed with the Inorganic Department Manager for approximately 3 months, for reference by analysts. Prior data are archived.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges

listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, remaining sample volume and client and project specific Data Quality Objectives. The Department Manager, Operations Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

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- 8.1 One sample duplicate is to be analyzed per batch or every 10 sample analyses.
- 8.1.1 Acceptance criteria for duplicates is a difference of less than or equal to 20% relative percent difference between sample and duplicate results (For SM 4500H B, the RPD \leq 10%).
- 8.1.2. If criterion is not met, check calibration and reanalyze sample in duplicate.
- 8.2 One Laboratory Control Sample (LCS) is to be analyzed per batch or every 20 samples.
- 8.2.1 The LCS must be within 90-110% recovery for analysis to proceed.
- 8.2.2 If criteria are not met, recalibrate.

9.0 METHOD PERFORMANCE

Refer to Method 9040.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, Method 150.1, Revised March 1983.

Standard Methods For the Examination of Water and Wastewater, 18th Edition, 1992, Method 4500-H⁺.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update III, December 1996, Method 9040B.

Accumet® Model 20 pH/Conductivity Meter operating Instructions, #300143.3 (Revision C)

List of Figures and Tables

Table 1	QC Requirements
Table 2	Summary of Method Modifications
Figure 1	Example of pH Logbook Page
Figure 2	Batch Sheet for pH

TITLE: pH CONCENTRATION MEASUREMENTS IN AQUEOUS SAMPLES

TABLE 1
QC REQUIREMENTS

Analytical Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
EPA 150.1 SW 9040 and SM4500-H ⁺	PH (water)	2-point calibration with pH buffers with a midrange cal. check	Once per day	± 0.05 pH units for every buffer	If calibration is not achieved, check meter, buffer solutions, and probe; replace if necessary; repeat calibration
		LCS	One per batch of twenty or fewer samples	90-110%R	Correct problem, recalibrate
		Sample duplicate	One sample duplicate per every ten field samples	RPD ≤20 (For 4500H B, RPD ≤10)	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD is still unacceptable, report original result with notation or narration.

TITLE: pH CONCENTRATION MEASUREMENTS IN AQUEOUS SAMPLES

TABLE 2
 SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-708-04	METHOD 150.1, current revision
Apparatus/Materials		
Reagents		
Sample preservation/handling		
Procedures	<ol style="list-style-type: none"> 1) All buffers and samples are analyzed at room temperature. PH meter is equipped with automatic temperature compensation. 2) One aliquot of sample used for pH analysis. 	<ol style="list-style-type: none"> 1) Report both pH and temperature at the time of analysis. 2) Repeat pH measurement on successive volumes of sample until values differ by less than 0.1 pH units.
QC – Spikes		
QC – LCS		
QC - Accuracy/Precision		

TITLE: pH CONCENTRATION MEASUREMENTS IN AQUEOUS SAMPLES

TABLE 2, cont'd.

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-708-04	METHOD 9040, current revision
Apparatus/Materials		
Reagents		
Sample preservation/handling		
Procedures	<p>3) All buffers and samples are analyzed at room temperature. pH meter is equipped with automatic temperature compensation.</p> <p>4) One aliquot of sample used for pH analysis.</p>	<p>3) Report both pH and temperature at the time of analysis.</p> <p>4) Repeat pH measurement on successive volumes of sample until values differ by less than 0.1 pH units.</p>
QC – Spikes		
QC – LCS		
QC - Accuracy/Precision		

TITLE: pH CONCENTRATION MEASUREMENTS IN AQUEOUS SAMPLES

TABLE 2, cont'd.

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-708-04	METHOD SM 4500-H ⁺ B, current revision
Apparatus/Materials		
Reagents		
Sample preservation/handling		
Procedures	<ol style="list-style-type: none"> 1) All buffers and samples are analyzed at room temperature. PH meter is equipped with automatic temperature compensation. 2) Results for the calibration check must be within 0.05 pH units of the true value for analysis to proceed. 3) One aliquot of sample used for pH analysis. 	<ol style="list-style-type: none"> 1) Report temperature at which pH measurement is made. 2) The reading should be within 0.1 unit. 3) For buffered samples, condition electrodes after cleaning by dipping them into sample for 1 min. Blot dry, immerse in a fresh portion of the same sample, and read pH. For dilute, poorly buffered solutions, equilibrate electrodes by immersing in three of four successive portions of sample. Take a fresh sample to measure pH.
QC – Spikes		
QC - LCS		
QC - Accuracy/Precision		

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FIGURE 2

KIMS BATCH SHEET FOR pH

WST CHEMISTRY BATCH REPORT
 Apr 06 2009, 08:07 am
 Batch: WG62266

Parameter: pH(Laboratory)

Prep Date: N/A

Date Analyzed: 02-APR-09

Prep Method: N/A

Analyst Initials: JF

Prep Chemist: N/A

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SC1496-1	SAMP	SM 4500H-B	20.000mL	20.000mL	1	7.81	7.8 pH	NA	.1	.1	.1		
SC1496-2	SAMP	SM 4500H-B	20.000mL	20.000mL	1	7.08	7.1 pH	NA	.1	.1	.1		
WG62266-1	LCS	SM 4500H-B	20.000mL	20.000mL	1	7	7.0 pH	NA	.1	.1	.1		100
WG62266-2	DUP	SM 4500H-B	20.000mL	20.000mL	1	7.12	7.1 pH	NA	.1	.1	.1	0	

Comments:

WG62266-1 SC1496-2
 WG62266-2 SC1496-2

Entered by:

Date: 4-6-09

Accepted by:

CP

Date: 4/6/09

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

Prepared By: Wet Chemistry Date: 8/96

Approved By:

Group Supervisor: Keith Tangway Date: 2/21/01

Operations Manager: John C. Burton Date: 2/13/01

QA Officer: Dorothy J. Nadeau Date: 2/13/01

General Manager: Derran F. Huffan Date: 2/12/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03 9045C	Format changes, added pollution prevention, database and operation of Accumet pH meter and calibration.	DN	2/13/01	2/13/01
04 9045C	Addition to scope and Application to include reference for 9040B use when aqueous phase is >20%	DN	8/27/02	8/27/02
05 9045C	added KIMS minor changes throughout added wording to sect. 6 New fig. 1 and 2	LAD	12/01/04	12/01/04
06 9045C	Added SW-846 reference. Minor formatting changes throughout.	LAD	03/07	03/07
07 9045D	Section 7.18 - Renamed "Equipment Maintenance" and revised for current practices. Add Wet Chem. Data Entry SOP reference. updated references in Section 10. Updated log book example.	LAD	08/09	08/09

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD
9045**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **CA-709-07**, titled **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **CA-709-07**, titled **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**.

Recipient: _____ Date: _____

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD
9045**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and techniques followed by Katahdin Analytical Services, Inc. personnel to determine the pH of soils and waste samples in accordance with EPA method 9045 (current promulgated revision). Method 9045 is an electrometric procedure for measuring pH in soils and waste samples. Wastes may be solids, sludges, or non-aqueous liquids. If water is present, it must constitute less than 20% of the total volume of the sample. If the aqueous phase is greater than 20%, pH determination should be performed in accordance with EPA method 9040 (current promulgated revision). Refer to the current revision of Katahdin SOP CA-708, pH Concentration Measurements in Aqueous Samples.

The procedures in this SOP are applicable to all non-CLP pH measurements performed for all soil matrices analyzed in the laboratory.

1.1 Definitions

pH - A measure of the acidity or alkalinity of a solution, defined as $-\log [H^+]$.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of pH in solids by EPA Method 9045. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the determination of pH concentration measurements in solid matrices to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for pH data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

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 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.

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. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

A representative aliquot of sample, measured in grams, is mixed with an equivalent volume of laboratory reagent grade water, measured in mL. The solution is allowed to settle, and the pH of the standing water (decanted) is determined electrometrically.

3.0 INTERFERENCES

3.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode. Strong acid solutions, with a true pH of <1, may give incorrectly high pH measurements.

3.2 Temperature fluctuations will cause measurement errors.

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- 3.3 Errors will occur when the electrodes become coated with an oily material. See section 7.18 for special cleaning instructions.
-

4.0 APPARATUS AND MATERIALS

- 4.1 pH meter, Accumet Model 20 or equivalent with Automatic Temperature Compensation (ATC)
- 4.2 Glass beakers, 25 mL and 400 mL
- 4.3 25 mL dose cups
- 4.4 Teflon coated stir-bars
- 4.5 Stir-bar retriever
- 4.6 Magnetic stirplate
- 4.7 Shaker, 12 place
- 4.8 Analytical balance, capable of weighing to 0.1 g
-

5.0 REAGENTS

- 5.1 Buffer solutions (pH 4.0, 6.0, 7.0, 8.0, 10.0, 12.0)
- 5.2 Laboratory reagent grade water (Lab Water)
-

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected in soil jars and stored at 4°C until analysis. Since there are no published holding times for this method, sample analysis should be performed as soon as possible.

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7.0 PROCEDURES

SAMPLE PREPARATION

- 7.1 Mix samples thoroughly. Discard any foreign objects such as sticks, leaves and rocks. Decant any standing liquid. Using the balance, weigh out 20.0 g of sample into a 400 mL glass beaker. Record weight in pH logbook (Figure 1).
- 7.2 Add 20 mL of laboratory reagent grade water to the sample. Cover the top of the beaker with parafilm.
- 7.3 Place the sample on the shaker and allow it to shake, at medium speed, for one hour.
- 7.4 After one hour, remove the sample from the shaker and allow it to settle for one hour.
- 7.5 After one hour, decant the standing liquid into a 25 mL beaker. If no standing liquid is present, add sufficient laboratory reagent grade water to result in standing water, cover with parafilm, and repeat steps 7.3 through 7.5.
- 7.6 Record total volume of laboratory reagent grade water added to sample in pH logbook. If volume of laboratory reagent grade water (in mL) added to sample exceeds the initial gram weight of the sample, flag sample data in pH logbook with reason for addition of excess laboratory reagent grade water (eg. minimum volume of water required in order to cover pH probe).

CALIBRATION OF PH METER

- 7.7 NORMAL RANGE CALIBRATION (pH range 3.5 - 10.5)
 - 7.7.1 Meter should be calibrated daily. As described in the following steps, conduct a two-point calibration with pH buffers 4 and 10. Perform a calibration check using pH 7 buffer. The source/lot number of each solution at the time of analysis must be recorded in the logbook (Figure 1).
 - 7.7.2 Dispose of buffer solutions used the previous day (see Waste Disposal, section 1.4). Put about 20 mL of the appropriate buffer solution into new dose cups. Place a tiny stir bar in each new cup.
 - 7.7.3 Rinse probe (i.e., electrode) with laboratory DI laboratory reagent grade water. Gently blot dry with kimwipe.
 - 7.7.4 Place pH 4 buffer on stir plate. Turn stir plate on so that the stir bar spins without creating a vortex. Push 1 key (meaning add a standard). Then push 4 (calibrate). Record the value in the pH logbook.

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- 7.7.5 Remove pH 4 buffer. Rinse probe. Blot dry.
- 7.7.6 Repeat step 7.7.4 with the pH 10 buffer. Record the value in the pH logbook. Remove pH 10 buffer. Rinse and dry probe.
- 7.7.7 With the pH 7 buffer, repeat step 7.7.4, but DO NOT press any keys as this reading is a calibration check. Record reading in pH logbook. Results must be within 0.05 pH units of the true value for analysis to proceed.

NOTE: If buffer readings are not within 0.05 pH units of expected values (4.00, 7.00 and 10.00) the electrode may need cleaning. Rerun and enter in pH lab notebook that meter was recalibrated with the pH 4 and 10 buffers, with the pH 7 buffer used as a calibration check. Also record any maintenance performed.

7.8 LOW RANGE CALIBRATION

- 7.8.1 If pH of a sample as is less than 3.5, the instrument must be recalibrated using buffers that bracket the expected pH of the sample, as described below. The source/lot number and temperature of each solution at the time of analysis must be recorded in the logbook (Figure 1).
- 7.8.2 Rinse probe (i.e., electrode) with laboratory reagent grade water.
- 7.8.3 . Gently blot dry with kimwipe.
- 7.8.4 Place pH 2 buffer on stir plate. Turn stir plate on so that the stir bar spins without creating a vortex. Push 1 key (meaning add a standard). Then push 4 (calibrate). Record the value in the pH logbook.
- 7.8.5 Remove pH 2 buffer. Rinse probe. Blot dry.
- 7.8.6 Repeat step 7.8.3 with the pH 7 buffer. Record the value in the pH logbook. Remove pH 7 buffer. Rinse and dry probe.
- 7.8.7 With the pH 4 buffer, repeat step 7.8.3, but DO NOT press any keys as this reading is a calibration check. Record reading in pH logbook. Results must be within 0.05 pH units of the true value for analysis to proceed.

NOTE: If buffer readings are not within 0.05 pH units of expected values (2.00, 4.00 and 7.00) the electrode may need cleaning. Rerun and enter in pH lab notebook that meter was recalibrated with the pH 2 and 7 buffers, with the

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pH 4 buffer used as a calibration check. Also record any maintenance performed.

7.9 HIGH RANGE CALIBRATION

- 7.9.1 If pH of a sample as determined in Sections 7.10 through 7.17 is greater than 10.5, the instrument must be recalibrated using buffers that bracket the expected pH of the sample, as described below. The source/lot number and temperature of each solution at the time of analysis must be recorded in the logbook (Figure 1).
- 7.9.2 Rinse probe (i.e., electrode) with laboratory reagent grade water. Gently blot dry with kimwipe.
- 7.9.3 Place pH 7 buffer on a stirplate. Turn stir plate on so that the stir bar spins without creating a vortex. Push *1* key (meaning add a standard). Then push *4* (calibrate). Record the value in the pH logbook.
- 7.9.4 Remove pH 7 buffer. Rinse probe. Blot dry.
- 7.9.5 Repeat step 7.9.3 with the pH 12 buffer. Record the value in the pH logbook. Remove pH 12 buffer. Rinse and dry probe.
- 7.9.6 With the pH 10 buffer, repeat step 7.9.3, but DO NOT press any keys as this reading is a calibration check. Record reading in pH logbook. Results must be within 0.05 pH units of the true value for analysis to proceed.

NOTE: If buffer readings are not within 0.05 pH units of expected values (7.00, 10.00 and 12.00) the electrode may need cleaning. Rerun and enter in pH lab notebook that meter was recalibrated with the pH 7 and 12 buffers, with the pH 10 buffer used as a calibration check. Also record any maintenance performed.

ANALYSIS OF SAMPLES

- 7.10 Sample analysis may proceed once the meter has been calibrated for the day with two buffers that bracket the expected pH of the sample, and after checking the calibration with a mid-range solution between the two buffers used for calibration of the meter (refer to sections 7.7, 7.8, and/or 7.9 as applicable).
- 7.11 For the range being used, rerun the appropriate calibration check point as the laboratory control sample (LCS) for the analytical batch. An LCS is required at the beginning of every batch of twenty or fewer samples.

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD
9045**

- 7.12 Record date, time and initials for this analytical session.
- 7.13 The decanted samples should be equilibrated to room temperature prior to analysis (i.e., at the same temperature as the calibration buffers, ± 2 °C). A more accurate pH reading will be achieved when the buffers and the samples are at the same temperature. However, the Accumet® pH meter is equipped with automatic temperature compensation (ATC) for when samples and buffers are not at the same temperature. Refer to the Accumet® Model 20 pH/Conductivity Meter operating Instructions, #300143.3 (Revision C) for information on the ATC probe.
- 7.14 Pour about 25 ml of the supernatant into a clean dose cup. Place a tiny stir bar in cup. Place on stir plate, turn on stir plate and immerse probes.
- 7.15 When meter locks, record value displayed.
- 7.16 Rinse probe with laboratory reagent grade water and blot dry between samples.
- 7.17 Place probe in pH 4 buffer solution to store until next analysis.

EQUIPMENT MAINTENANCE

- 7.18 If an electrode becomes coated with an oily material that will not rinse free, the electrode can either (refer to instrument manual):
- be cleaned with an ultrasonic bath, or
 - be washed with detergent, rinsed several times with laboratory reagent grade water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with laboratory reagent grade water.

An electrode that will not calibrate properly must be replaced.

REPORTING OF RESULTS

- 7.19 All pH measurements less than 10.0 are to be reported using two significant figures.

Examples: 2.46 = 2.5
 6.32 = 6.3
 9.94 = 9.9

- 7.20 All pH measurements which are at or greater than or round up to 10.0 are to be reported to three significant figures.

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Examples: 9.95 = 10.0
 12.25 = 12.3
 13.76 = 13.8
 11.95 = 12.0

- 7.21 When a sample duplicate is analyzed, both the original result and duplicate result are recorded in the pH logbook; however, the original sample result is to be reported to the client.
- 7.22 After completion of each test, the logbook must be signed and dated by the person performing the test. All unused lines are to be "z-ed" out and initialed and dated.
- 7.23 The sample data results, with any appropriate notations, are entered into KIMS by the analyst. Refer to the current revision of SOP CA-762 for instructions on data entry. A batch sheet is generated (Figure 2). Raw data and batch sheets are reviewed for completeness and accuracy by the Inorganic Department Manager or other qualified designee.
- 7.24 All batch sheets and copies of the raw logbook data are filed with the Inorganic Department Manager for approximately 3 months, for reference by analysts. Prior data are archived.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, remaining sample volume and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

- 8.1 One sample duplicate is to be analyzed per batch or every 10 sample analyses.
 - 8.1.1 Acceptance criteria for duplicates is a difference of less than or equal to 20% relative percent difference between sample and duplicate results.
 - 8.1.2. If criterion is not met, check calibration and reanalyze sample in duplicate.
 - 8.2 One Laboratory Control Sample (LCS) is to be analyzed per batch or every 20 samples.
 - 8.2.1 The LCS must be within 90-110% recovery for analysis to proceed.
 - 8.2.2 If criteria are not met, recalibrate.
-

9.0 METHOD PERFORMANCE

Refer to method 9045.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB, revised November 2004, Method 9045D.

Katahdin SOP CA-101, Equipment Maintenance

Katahdin SOP CA-762, Wet Chemistry Data Entry and Review Using Katahdin Information Management System (KIMS)

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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9045**

LIST OF TABLES AND FIGURES

Table 1	QC Requirements
Table 2	Summary of Method Modifications
Figure 1	Example of pH - Soils Logbook Page
Figure 2	Example of Batch Sheet for pH

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

TABLE 1
 QC REQUIREMENTS

Analytical Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW9045	PH (soil)	2-point calibration with pH buffers with a midrange cal. check	Once per day, prior to use	± 0.05 pH units for each buffer	If calibration is not achieved, check meter, buffer solutions, and probe; replace if necessary; repeat calibration
		LCS	One per batch of twenty or fewer samples	90-110% recovery	Correct problem, recalibrate
		Sample duplicate	One sample duplicate per every ten field samples	RPD ≤20%	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD is still unacceptable, report original result with notation or narration.

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

TABLE 2
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-709-07	METHOD SW846 9045, current revision
Apparatus/Materials		
Reagents		
Sample preservation/handling		
Procedures	<ol style="list-style-type: none"> 1) Shake, at medium speed, for one hour. 2) Add more liquid after shaking and settling if there is no standing liquid left. 3) All buffers and samples are analyzed at room temperature. pH meter is equipped with automatic temperature compensation. 	<ol style="list-style-type: none"> 1) Continuously stir the suspension for five minutes. 2) No guidance for samples with no standing liquid left. 3) Report both pH and temperature at the time of analysis.
QC – Spikes		
QC – LCS		
QC - Accuracy/Precision		

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045**

FIGURE 1

EXAMPLE OF pH LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES, INC.					
CORROSIVITY pH / pH Soil					
Accumet 20 pH Meter - SN - C0024321			pH Probe SN - 81271408		
SW 846 9045D ✓			CLP SOW		
CALIBRATION STDS:		CALIBRATED TO:	LOT NO:		
pH 7.00		7.00	SWL 2685		
pH 4.00		4.00	SWL 2740		
pH 5.00					
pH 10.00		10.00	SWL 2655		
pH 8.00					
pH 12.00					
LAB SAMPLE ID	ANALYSIS TIME	SAMPLE VOL (mL)	SAMPLE WEIGHT(g)	pH	REPORTED pH
LCS WG 12345	12:20	20.0	20.0000	7.01	7.0
SC 1234-5	12:22	20.0	20.008	6.80	6.8
DUP WG 1234-6	12:24	20.0	20.006	6.81	6.8
SC 1234-6	12:26	20.0	20.001	7.79	7.8
↓ -7	12:28	20.0	19.998	6.31	6.3
↓ -8	12:30	20.0	19.997	5.19	5.2
<div style="position: absolute; top: 50%; left: 50%; transform: translate(-50%, -50%); opacity: 0.5;"> JAD 08.04.09 </div>					
ANALYST: (JAD)				DATE: 08.04.09	
CHECKED BY:				DATE:	

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FIGURE 2
EXAMPLE OF BATCH SHEET FOR pH

XXXXXXXXXXXX

WET CHEMISTRY BATCH REPORT
Apr 06 2009, 08:12 am
Batch: WG62267

Parameter: pH(Soil)
Date Analyzed: 02-APR-09
Analyst Initials: JF

Prep Date: 02-APR-09
Prep Method: SW846 9045C
Prep Chemist: JF

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS (t)	PQL	MDL	Adj PQL	RPD	Rec
SC1497-1	SAMP	SW846 9045C	20.176g	20mL	1	8.7	8.7 pH	96.	.1	.1	.1		
SC1497-2	SAMP	SW846 9045C	20.051g	20mL	1	7.62	7.6 pH	89.	.1	.1	.1		
SC1497-3	SAMP	SW846 9045C	20.193g	20mL	1	7.55	7.6 pH	88.	.1	.1	.1		
WG62267-1	LCS	SW846 9045C	20g	20mL	1	7	7.0 pH	NA	.1	.1	.1		100
WG62267-2	DUP	SW846 9045C	20.173g	20mL	1	8.69	8.7 pH	NA	.1	.1	.1	0	

Comments:

WG62267-1 SC1497-1
WG62267-2 SC1497-1

Entered by:

Date: 4-6-09

Accepted by:

Date:

4/6/09

TITLE: TOTAL DISSOLVED SOLIDS (FILTERABLE RESIDUE) BY EPA METHOD 160.1
AND STANDARD METHODS 2540 C

Prepared By: Betsy Carbone Date: 12/97

Approved By:

Group Supervisor: Keith Torgerson Date: _____

Operations Manager: John C. Burton Date: 1/24/01

QA Officer: Dorothy J. Nadeau Date: 1-22-01

General Manager: Dennis F. Kufan Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, added pollution prevention, added SM 2540 C. Minor changes to sections 7+8 and Tables.	DN	1-22-01	1-22-01
02	minor changes to section 8	DN	3-28-02	3-28-02
03	Changed name to include referenced methods. added Kims entry to text and figures. Revised balance calibration weights, desiccant drying temperature and blank requirement. Added wording to sect 8.	LAD	03/6/05	03/6/05
04	added definitions to section 1	LAD	02/08	02/08
05	Added requirement to record the oven and thermometer IDs in logbook. Added references to section 10.	DN	08/09	08/09

**TITLE: TOTAL DISSOLVED SOLIDS (FILTERABLE RESIDUE) BY EPA METHOD 160.1
AND STANDARD METHODS 2540 C**

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-719-05, titled TOTAL DISSOLVED SOLIDS (FILTERABLE RESIDUE) BY EPA METHOD 160.1 AND STANDARD METHODS 2540 C.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-719-05, titled TOTAL DISSOLVED SOLIDS (FILTERABLE RESIDUE) BY EPA METHOD 160.1 AND STANDARD METHODS 2540 C.

Recipient: _____ Date: _____

TITLE: TOTAL DISSOLVED SOLIDS (FILTERABLE RESIDUE) BY EPA METHOD 160.1 AND STANDARD METHODS 2540 C

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services, Inc. technical personnel to measure the concentration of total dissolved solids (TDS) found in drinking and surface waters, domestic waters and groundwaters, industrial effluent and leachates as described in EPA Method 160.1 and SM 2540 C.

This procedure applies to all determinations of dissolved solids as performed by Katahdin Analytical. This procedure is applicable to measurement of TDS to a Practical Quantitation Level of 10 mg/L when 100 mL of sample is used for analysis.

1.1 Definitions

Total Dissolved Solids – the portion of total solids that passes through a standard glass fiber filter.

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.

LCS - Laboratory Control Sample - A standard or solid reference material of known value that has been brought through the sample preparation and analysis process. The LCS is used to assess the accuracy of the method.

MB – Method Blank - Reagent water that has been brought through the sample preparation and analysis process. The MB is used to assess contamination.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the MDL.

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 total dissolved solids by Method 160.1 and 2540 C. 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 TDS by EPA 160.1 and SM 2540 C to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab

TITLE: TOTAL DISSOLVED SOLIDS (FILTERABLE RESIDUE) BY EPA METHOD 160.1 AND STANDARD METHODS 2540 C

notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous waste 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.

2.0 SUMMARY OF METHOD

A well-mixed sample is filtered through a standard glass fiber. The filtrate is evaporated and dried to constant weight at 180°C.

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AND STANDARD METHODS 2540 C**

3.0 INTERFERENCES

Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation, and rapid weighing.

Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180°C to insure that all the bicarbonate is converted to carbonate.

Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to 200 mg.

4.0 APPARATUS AND MATERIALS

- 4.1 Vacuum pump - capable of generating vacuum at 20 to 25 psi
 - 4.2 Glass fiber filter discs without organic binders, 47 mm diameter, nominal pore size 2.0 um or less. Gelman type A/E, Millipore type AP40, Whatman type 934-AH, or equivalent are acceptable.
 - 4.3 Oven - while oven temperature during evaporation must not exceed 98°C, the oven must have the capability of reaching 180° ± 2° C.
 - 4.4 Plastic filter funnel with filter support grid and magnetic closure, 300 mL capacity
 - 4.5 Filter flask with side arm, 500 mL capacity
 - 4.6 Evaporation dishes - porcelain dishes (crucibles) capable of containing a 100 mL maximum volume.
 - 4.7 Analytical balance - capable of weighing to 0.2 mg; balance must be calibrated in accordance with Katahdin SOP, CA-102, Balance Calibration, before each measurement. Weights used are 100.0 g, 10.0 g, 2.0 g and readings are recorded in the TDS Logbook (Figure 1).
 - 4.8 Desiccator - with conditioned indicating desiccant (desiccant is conditioned by drying at 210°C for one hour)
 - 4.9 Graduated Cylinders – 10, 25, 50, 100, 250 mLs.
-

TITLE: TOTAL DISSOLVED SOLIDS (FILTERABLE RESIDUE) BY EPA METHOD 160.1 AND STANDARD METHODS 2540 C

5.0 REAGENTS

- 5.1 Laboratory reagent grade water,
 - 5.2 Laboratory Control Sample (LCS) solution - Carefully weigh out 0.7450 g of reagent grade potassium chloride. Add it to a one liter volumetric flask and bring it up to volume with laboratory reagent grade water. Mix well until dissolved. The true value is 745.0 mg/L. Alternatively, a purchased solution with a certified TDS value may be used.
-

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected in plastic or glass bottles.

The analytical holding time for this test is 7 days from the time of sampling. To obtain a PQL of 10 mg/L, 100 mL of sample is required. To minimize microbiological decomposition of solids, samples are required to be refrigerated (4°C) from the time of collection until analysis.

The filtrate from the total suspended determination (EPA 160.2 or SM 2540 D) may be used for the determination of total dissolved solids.

7.0 PROCEDURES

- 7.1 Prepare evaporation dishes - Heat evaporating dishes (crucibles) to 180°C for 1 hour. Cool in desiccator for at least one hour and store until needed. Weigh each dish before use and record initial weight in TDS Logbook (figure 1).
- 7.2 Prepare glass fiber filter - Assemble filter apparatus. Insert filter disk with wrinkled side up. Note: handle filter disks carefully with forceps, grasping each disk at its outer edge to avoid damaging the exposed portion of the filter. Apply vacuum and wash filter disk with three successive 20-mL aliquots of laboratory reagent grade water. Continue suction until no traces of water remain. Discard the rinsates. Rinsed filters may be stored in the desiccator until use.
- 7.3 Assemble filter apparatus with clean filter and start the vacuum pump. Shake sample vigorously and quantitatively transfer 100 mL of sample, or another measured aliquot less than 100 mL, to the filter funnel using a graduated cylinder. Record the sample volume in the TDS Logbook.

Alternatively, 100 mL, or another measured aliquot, of the filtrate generated during TSS analysis may be used (refer to Katahdin SOP CA-720, current revision).

TITLE: TOTAL DISSOLVED SOLIDS (FILTERABLE RESIDUE) BY EPA METHOD 160.1 AND STANDARD METHODS 2540 C

- 7.4 Filter sample through the glass fiber filter. Rinse with three successive 10-mL aliquots of laboratory reagent grade water while applying suction to remove all water.

Note: If the measured volume does not filter completely, the sample must be prepared again. It is impossible to know how much of the dissolved matter has gone through the filter paper and therefore not possible to determine the actual sample aliquot. For these cases, it is advisable to prepare several extra filter discs and store them in the desiccator.

Note: Since excessive residue may form a water trapping crust, samples in excess of 200 mg of residue must be rerun at a dilution.

- 7.5 Quantitatively transfer filtrate to porcelain dishes (crucibles) and evaporate to dryness in oven at 98°C (to prevent boiling and splattering of the sample). Record initial time and temperature of the oven in the TDS Logbook. Record the oven ID and the thermaometer ID in the TDS logbook.
- 7.6 Dry evaporated sample for at least 1 hour at 180°C ± 2.0°C. Record initial and final time and temperature (at 180°C) in the TDS Logbook. Cool in the desiccator for at least one hour and weigh. Repeat cycle of heating, cooling, and weighing until a constant weight is obtained or until the difference between successive weighings is 0.5 mg or 4%, whichever is less. For each sample, method blank, and LCS, record the final two weights in the TDS logbook once a constant reading is obtained.
- 7.7 The initial sample volume and initial and final sample weights are entered manually into the Katahdin Information Management System (KIMS) for calculation and reporting. After the data are entered, a batch sheet (Figure 2) is automatically printed by KIMS. Refer to the current revision of Katahdin SOP CA-732 ("Wet Chemistry Data Entry and Review Using Katahdin Information Management System") for further information.

The final concentration of TDS is calculated by KIMS using the following formula:

$$\text{TDS (mg/L)} = \frac{[\text{Wt of crucible + residue (g)}] - [\text{Wt of crucible (g)}] * 1000 \text{ mg} * 1000 \text{ mL}}{\text{Sample Volume (mL)} \quad \text{g} \quad \text{L}}$$

- 7.8 After completion of each test, the TDS Logbook must be signed and dated by the person performing the test. All unused lines are to be "z-ed" out and initialed and dated.
-

**TITLE: TOTAL DISSOLVED SOLIDS (FILTERABLE RESIDUE) BY EPA METHOD 160.1
AND STANDARD METHODS 2540 C**

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 The analytical balance must be calibrated in accordance with Katahdin SOP, CA-102, Balance Calibration, before each measurement. Weights used are 100 g, 10 g and 2.0 g and readings should be recorded in the TDS logbook.

ASTM Class 1 Weight	Acceptance Range
100g	99.0000g - 101.0000g
10g	9.9000g - 10.1000g
2.0g	1.9800g - 2.0200g

- 8.2 One blank sample must be run with every batch of twenty or fewer samples. Fill a preweighed evaporating dish with laboratory reagent grade water, evaporate, and reweigh.

Acceptance Criteria: The measured TDS for the blank must be below the PQL of 10 mg/L.

TITLE: TOTAL DISSOLVED SOLIDS (FILTERABLE RESIDUE) BY EPA METHOD 160.1 AND STANDARD METHODS 2540 C

Corrective Action for Non-Conformance: Reanalyze all associated samples. If samples can not be reanalyzed, a Corrective Action Report (CAR) must be initiated and data appropriately qualified.

- 8.3 A duplicate sample must be run for every 10 samples. Calculate the mean (X) of the sample and sample duplicate and relative percent difference (RPD) of the duplicates as follows:

$$X = \frac{X_1 + X_2}{2}$$

$$RPD = \frac{X_1 - X_2}{X} \times 100$$

where X_1 = filterable residue (TDS) measurement for sample
 X_2 = filterable residue (TDS) measurement for sample duplicate

Acceptance Criteria: Relative Percent Difference (RPD) \leq 20%.

Corrective Action for Non-Conformance: Reanalysis of sample in duplicate; if RPD is again >20%, report original result with the appropriate flag or narration.

- 8.4 One Laboratory Control Sample (LCS) must be prepared and analyzed with every sample batch of twenty or fewer samples.

Acceptance Criteria: Recovery within 90%-110% of the true value.

Corrective Action for Non-Compliance: Reanalyze all associated samples. If samples can not be reanalyzed, a Corrective Action Report (CAR) must be initiated and data appropriately qualified.

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 and filed with the 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 Methods 160.1 and 2540 C for other method performance parameters and requirements.

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AND STANDARD METHODS 2540 C**

10.0 APPLICABLE DOCUMENTS/REFERENCES

Method 160.1, Total Filterable Residue, "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, Revised March, 1983.

Method 2540 C. Total Dissolved Solids Dried at 180°C, "Standard Methods for the Examination of Water and Wastewater", 18th edition, 1992, APHA-AWWA-WPCF.

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.

Katahdin SOP CA-101, Equipment Maintenance, current revision.

LIST OF FIGURES & TABLES

TABLE 1 QC Requirements

TABLE 2 Summary Of Method Modifications

FIGURE 1 Example of TSS Logbook Page

FIGURE 2 Example of TSS Batch Sheet

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TABLE 1

QC REQUIREMENTS

Applicable Parameter or Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Total Dissolved Solids EPA Method 160.1 & SM Method 2540 C	Method blank	One per prep batch of 20 or fewer samples)	TDS < PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the associated samples.
	LCS	One per prep batch of 20 or fewer samples)	90%-110% recovery	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
	Sample Duplicate	One sample duplicate per ten samples	RPD ≤20	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD still >20, report original result with notation.
	Initial Demonstration of Performance: Precision and accuracy study using 4 replicate analyses LCS	Initially once per analyst, then yearly.	All recoveries within method QC acceptance limits	Investigate source of problem; rerun P & A study
	MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

**TITLE: TOTAL DISSOLVED SOLIDS (FILTERABLE RESIDUE) BY EPA METHOD 160.1
AND STANDARD METHODS 2540 C**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-719-05	EPA method 160.1and SM 2540C, current revisions
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision	Duplicate Frequency 1/10	Duplicate Frequency 1/20
QC - MDL		

TITLE: REACTIVE CYANIDE: SW-846, 7.3.3.2

Prepared By: Wet Chemistry Date: 8/96

Approved By:

Group Supervisor: Keith Tanguay Date: 2/13/01

Operations Manager: John C. Buxton Date: 2/13/01

QA Officer: Deborah J. Nadeau Date: 2/13/01

General Manager: Deanna F. Majumdar Date: 2/13/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, added pollution prevention database and new CN SOP reference. Other minor changes throughout.	DN	2/13/01	2/13/01
02	Major changes to apparatus and procedure to reflect current practice. Added reference for SOP CA-107 to sect. 1.4. Added program specific info to Sect. 8. Supervisor => dept. manager, ASTM II Water => lab, reagent grade H ₂ O. Updated figures	LAD	04/06	04/06
03	Section 7.1.3 and 7.1.4 changed spike amount from 0.1 mL to 0.05 mL. Added definitions to section 1.	LAD	02/08	02/08
04	Sec. 1.4 - Changed "G" Stream to "N-High Stream". Sect. 2.0 - Changed SOP reference. Removed Secs 4.7 and 4.10. Sect. 4.13 - changed to Appendix. Sect. 5.3 - Added purchased with certified reference value option. Added DOC and MDC criteria to Table 1. Added 2 method deviations to Table 2.	LAD	05/09	05/09
05	Added definitions to section 1.1. Revised Table 1. Added EHSU, subsampling, QA-86, DoD, NELAC and CA-101 references.	DN	08/09	08/09

TITLE: REACTIVE CYANIDE: SW-846, 7.3.3.2

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STANDARD OPERATING PROCEDURE**

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Recipient: _____ Date: _____

TITLE: REACTIVE CYANIDE: SW-846, 7.3.3.2

1.0 SCOPE AND APPLICATION

The intended application of this method is to determine the hydrogen cyanide released from wastes. This method is applicable to all waste except those that will form explosive mixtures when combined with acids. This test measures only the hydrocyanic acid evolved at the test conditions. It is not intended to measure forms of cyanide other than those that are evolved under the test conditions. The regulatory limit for *Total Releasable Cyanide* is 250 mg/Kg waste.

1.1 Definitions

Reactive Cyanide - Cyanide released under the test conditions defined under SW846 Chapter 7, 7.3.3.2 where the sample is exposed to mildly acidic conditions.

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.

LCS - Laboratory Control Sample - A standard or solid reference material of known value that has been brought through the sample preparation and analysis process. The LCS is used to assess the accuracy of the method.

LOD - Limit of Detection. The smallest amount or concentration of an analyte that must be present in a sample to be detected at a 99% confidence level. At the LOD, the false negative rate is 1%.

MB - Method Blank - Reagent water that has been brought through the sample preparation and analysis process. The MB is used to assess contamination.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the MDL.

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 reactive cyanide according to SW-846, 7.3.3.2. 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 reactive cyanide according to SW-846, 7.3.3.2, to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the

TITLE: REACTIVE CYANIDE: SW-846, 7.3.3.2

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. These materials include the following: Sodium Hydroxide, Potassium Cyanide, Sulfuric Acid, Hydrochloric Acid, Barbituric Acid, Silver Nitrate and Pyridine.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health & Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

All remaining basic waste from the distillation, receiver contents, is treated as though cyanide is present and disposed of in the pyridine ("N-High" stream) waste satellite located in the Wet Chemistry laboratory. When this container is full, it is then taken to the hazardous waste disposal area and the contents are transferred to the pyridine waste drum.

The acidic portion of the distillation, still contents, is placed in acid waste ("A" stream) via the satellite accumulation in the Wet Chemistry laboratory. Other wastes generated during the preparation of samples must be disposed of in

TITLE: REACTIVE CYANIDE: SW-846, 7.3.3.2

accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal" and CA-107, "The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents and Standards," current revisions. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with these SOPs.

2.0 SUMMARY OF METHOD

An aliquot of acid is added to a fixed weight of waste in a closed system. The gas that is generated is swept into a scrubber. The cyanide in the gas is absorbed in a NaOH scrubbing solution that is analyzed for cyanide by Katahdin SOP CA-773, "Colorimetric Analysis of Total and Amenable Cyanide Using the Automated Konelab Multiwavelength Photometric Analyzer".

3.0 INTERFERENCES

Interferences are undetermined.

4.0 APPARATUS AND MATERIALS

- 4.1 Magnetic stirrer; to achieve approximately 30 rpm.
- 4.2 Magnetic stirring bars and retriever.
- 4.3 Flexible tubing for connection from the nitrogen supply to the apparatus and from the flask to the absorber impinger unit (scrubber).
- 4.4 Nitrogen gas tank with regulator.
- 4.5 Gas valve capable of metering N₂ flow to 20 psi
- 4.6 Flowmeter capable of measuring flow at 60 mL/min at the distillation station.
- 4.7 Analytical balance weighing to 0.001g.
- 4.8 10-mL buret
- 4.9 12 gas washing bottles with 250ml graduated cylinders
- 4.10 Buret stand and holder

TITLE: REACTIVE CYANIDE: SW-846, 7.3.3.2

4.11 0.1mL and 5mL Eppendorf pipet and tips

5.0 REAGENTS

- 5.1 Sulfuric Acid (0.1 N), H₂SO₄: Add 5.6 ml of concentrated H₂SO₄ to laboratory reagent grade water and dilute to 2 liters.
- 5.2 Sulfuric Acid (0.01 N), H₂SO₄: Volumetrically transfer 200 ml of 0.1 N H₂SO₄ and dilute to 2 liters with laboratory reagent grade water to make the 0.01 N H₂SO₄.
- 5.3 Stock Cyanide Solution (1000 mg/L): **CAUTION SHOULD BE USED IN HANDLING KCN SALTS. ADD POTASSIUM HYDROXIDE TO VOLUMETRIC FLASK PRIOR TO INTRODUCTION OF THE KCN SALTS.** Place 0.5 g of KOH in 250-mL volumetric flask. Add 25 mL laboratory reagent grade water and swirl to dissolve. Add 0.6275 g of KCN to the flask, swirl to dissolve and bring to volume with laboratory reagent grade DI water. Standardize this solution with 0.0192 N AgNO₃, or purchase standard with certified reference value. The Stock Cyanide Solution should be made from a source that is different from that used to make the instrument calibration standard. See Section 7.1 for the standardization procedure. Note: This solution represents a 0.0192 N concentration when considered for reaction with AgNO₃. To satisfy the complexation with Ag, 2 equivalents are required for CN.
- 5.4 Sodium Hydroxide Solution (0.25 N) ,NaOH: Dilute 25.0 ml of 10 N NaOH to 1 liter of Laboratory reagent grade water. This solution could also be made by dissolving 10 g of NaOH in Laboratory reagent grade water and diluting to 1 liter.
- 5.5 Laboratory reagent grade water: Equivalent in protocol as reagent or DI water
-

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

- 6.1 Samples should be collected with a minimum of aeration. The filled sample bottle should contain no headspace and should be kept cool and in the dark until analysis. Samples can be held 14 days with no preservative. Perform analysis in a ventilated hood.
- 6.2 Samples can be preserved by adjusting the sample pH to 12 with strong base; however, this will cause dilution of the sample, increase the ionic strength, and possibly change other physical or chemical characteristics of the waste which may affect the rate of release of the hydrocyanic acid.
-

TITLE: REACTIVE CYANIDE: SW-846, 7.3.3.2

7.0 PROCEDURES

7.1 PREPARATION OF SAMPLE

7.1.1 Weigh approximately 10 g of sample in a 250 mL addition graduated cylinder. Record weight in the preparation logbook (Figure 1).

Note: Please refer to Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", current revision, for more information on subsampling.

7.1.2 To prepare Method Blank transfer 10 g of laboratory reagent grade water to a 250 mL addition graduated cylinder

7.1.3 To prepare LCS transfer 0.05 mL (100 µg CN) of the Stock Cyanide Solution (1000 mg/L, See Reagents 5.3) to a 250 mL addition graduated cylinder.

7.1.4 To prepare a Matrix Spike (MS), weigh approximately 10 g of sample in a 250 mL addition graduated cylinder. Spike the sample with 0.05 mL (100 µg CN) of the stock cyanide solution (1000 mg/L, see reagent 5.3).

7.1.5 To prepare sample Duplicate weigh out approximately 10 g of the sample selected/designated as the sample duplicate in a 250 addition graduated cylinder

7.1.6 Add 190 ml of 0.25 N NaOH to each of the absorber graduated cylinders.

7.1.7 Turn on the main valve on the Nitrogen tank. Make sure it is reading 300 psi or greater.

7.1.8 Adjust the local N₂ pressure valve in the hood ting knob and set the pressure to 20 psi on the low pressure gauge.

7.1.9 Turn the Outlet Valve on from the flowmeter on until the flow registers 60 mL/min.

7.1.10 Add 180 mL of 0.01N H₂SO₄ to the addition graduated cylinders and connect the apparatus as shown in Figure 3.

7.1.11 Use timer set for 30 minutes. After 30 minutes, disconnect all of the scrubbers on the apparatus.

7.1.12 Close off the main valve on the nitrogen tank followed by the pressure adjusting knob and then the outlet valve.

TITLE: REACTIVE CYANIDE: SW-846, 7.3.3.2

7.1.13 A portion of the scrubber is transferred to 40-mL VOA vial for CN analysis. The remainder is covered and titrated ASAP for reactive sulfide where requested.

7.2 ANALYSIS OF CN

7.2.1 Cyanide concentration in the scrubber is determined by automated colorimetry (e.g., Konelab) in accordance with the protocols delineated in the most current revision of Katahdin SOP CA-773, Total Cyanide, for the analysis procedure.

7.2.2 A portion of the scrubber solution may also be used for Reactive Sulfide analysis. See SOP CA-734, Reactive Sulfide: SW-846, 7.3.4.2.

$$R = \text{specific rate of release, mg / Kg / Sec} = \frac{A \times V}{W \times S}$$

7.2.3 The rate of release of HCN (mg/Kg/sec) is calculated as follows:

Where:

A = concentration of HCN in the scrubber as mg/L = 1.04 x CN mg/L
(1.04=MW HCN/MW CN= 27.03/26.0179)

V = volume in scrubber, Liters, i.e. 0.19

W = weight of waste, Kg

S = Time of measurement, Time N₂ stopped - Time N₂ started, sec

7.2.4 The releasable HCN as mg/Kg is calculated as follows:

$$\text{Total Releasable HCN, mg / Kg} = \frac{A \times V}{W}$$

Where:

A = concentration of HCN in the scrubber as mg/L
= 1.04 x CN mg/L (1.04=MW HCN/MW CN= 27.03/26.0179)

V = volume in scrubber, Liters, i.e. 0.19

W = weight of waste, Kg

7.3 REPORTING

7.3.1 Enter results, including sample preparation information, measured sample concentrations, and quality control data, into the Katahdin Information Management System for calculation and reporting. Refer to the current revision of SOP CA-762 ("Wet Chemistry Data Entry and Review Using Katahdin Information Management System") for further information. A batch sheet is generated (Figure 2). Raw data and batch sheets are reviewed

TITLE: REACTIVE CYANIDE: SW-846, 7.3.3.2

for completeness and accuracy by the Inorganic Department Manager or other qualified designee.

- 7.3.2 All batch sheets and copies of the raw logbook data are filed with the Inorganic Department Manager for approximately 3 months, for reference by analysts. Prior data are archived.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements.

These decisions are based on holding time considerations, remaining sample volume and client and project specific Data Quality Objectives. The 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.

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

Every instance of noncompliant method quality control requires the generation of a Corrective Action Report describing the problem, suspected cause and final resolution. Corrective action reports must be signed by the initiator, Department Manager, QA officer, and lab management.

- 8.1 One laboratory control sample (LCS) is distilled with every batch of 20 samples. The LCS spike solution (1000 mg/L cyanide standard) is an independently prepared standard from which 0.1 ml is distilled. Evaluate the % recovery based on historical laboratory data. The range of recovery for the LCS is 0 – 100 %.

TITLE: REACTIVE CYANIDE: SW-846, 7.3.3.2

- 8.2 A duplicate is run every ten samples. Sample duplicates are expected to agree within 20% relative difference. If duplicate samples are out of control, re-distill another replicate.
- 8.3 A method blank is analyzed with every batch or analytical session. The concentration of the blank must be less than the detection limit (1 mg/kg).
-

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 Inorganic 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 revisions of SW-846, 7.3.3.2 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste Physical/Chemical Methods, US EPA SW 846, 3rd edition, Volume 1C, Chapter Seven, Section 7.3.3.2, Rev. 2, September 1994.

Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, US EPA SW-846, 3rd Edition, Method 9012, Rev. 0, September 1986.

Katahdin SOP CA-773, "Colorimetric Analysis of Total and Amenable Cyanide Using the Automated Konelab Multiwavelength Photometric Analyzer".

Katahdin SOP CA-101, "Equipment Maintenance"

Katahdin SOP CA-762, "Wet Chemistry Data Entry and Review Using Katahdin Information Management System (KIMS)"

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

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.

TITLE: REACTIVE CYANIDE: SW-846, 7.3.3.2

List of Figures and Tables

Table 1	QC Requirements
Table 2	Summary of Method Modifications
Figure 1	Example of Logbook Page
Figure 2	Example of Batch Sheet
Figure 3	Reactive Cyanide Apparatus

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**TABLE 1
QC REQUIREMENTS**

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Reactive Cyanide SW-846 7.3.3.2	Method blank	One per prep batch of 20 or fewer samples	H ₂ CN not detected >PQL (For DoD QSM, no analyte detected > ½ PQL and > 1/10 the amount measured in any sample)	<ol style="list-style-type: none"> (1) Investigate source of contamination (2) Report all sample results <PQL. (3) Report sample results >10X the blank result and flag results with a "B". (4) Reanalyze all other samples associated with the failing blank.
	LCS	One per prep batch of 20 or fewer samples	0 – 100% nominal; statistically derived after sufficient historical	<ol style="list-style-type: none"> (1) If the LCS fails high, report samples that are <PQL. (2) Reanalyze /or recalibrate and reanalyze (3) Redistill, recalibrate and/or reanalyze other samples.
	Matrix Spike	One per prep batch of 20 or fewer samples	0-100 %R	<ol style="list-style-type: none"> (1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. (3) Analyze unspiked sample scrubber solution with post-scrub spike to confirm matrix interference present in the scrubber (4) It should be anticipated that 0% to very low recoveries may be evidenced in high metals content samples. (5) Notate sample result in raw data if matrix interference confirmed
	Sample Duplicate	One every ten samples	RPD ≤ 20%	<ol style="list-style-type: none"> (1) Investigate problem and reanalyze sample in duplicate (2) If RPD still >20, report original result with notation or narration.
	Demonstration of analyst proficiency	One time per analyst initially and annually thereafter	P&A meet method criteria	Repeat P&A study
	MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: REACTIVE CYANIDE: SW-846, 7.3.3.2

TABLE 2
SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-733-05	Method 7.3.3.2, current revision
Apparatus/Materials	(1) 250 mL scrubber (2) Gas washing bottles	(1) 50 mL scrubber (2) Ground glass glassware
Reagents	(1) Cyanide reference solution, 1000 mg/L prepared in 250 mL with 0.5 g KOH	(1) Cyanide reference solution, 1000 mg/L prepared in 250 mL with 0.625 g KOH
Sample preservation/handling		
Procedures	(1) 190 mL of 0.25 N NaOH are added to each scrubber (absorber bottle) prior to distillation. (2) 180 mL of 0.01 H ₂ SO ₄ added to reflux bottle.	(1) 50 mL of 0.25 N NaOH is added to each scrubber, then diluted with water to obtain an adequate depth of liquid in the scrubber. (2) Add enough sulfuric acid to fill flask half full.
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: REACTIVE CYANIDE: SW-846, 7.3.3.2

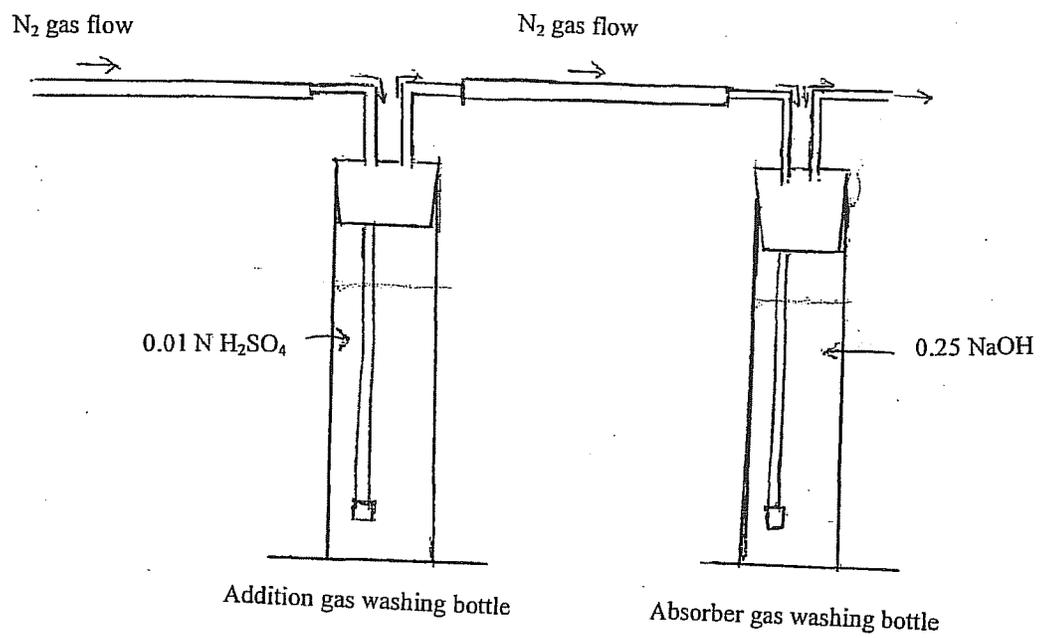
FIGURE 1

EXAMPLE OF LOGBOOK PAGE

KATAHDIN ANALYTICAL							
REACTIVE SULFIDE							
EPA: 7.3.4.2/9030			PQL: 27 mg/kg				
REAGENTS:							
IODINE STANDARD: W7828			STS SOLUTION-0.0375N: SWL 2671				
Na2S SOLUTION: W7757			NaOH-0.25N: W7915				
HCl-6N:			H2SO4-0.01N: W7827				
STANDARDIZATION OF I2							
VOL(ml) I2	VOL(ml) Na2S2O3		CALC OF I2 N				
10	6.70		0.02488				
10	6.65						
10	6.55						
	X: 6.63						
STANDARDIZATION OF H2S							
VOL(ml) I2	VOL(ml) Na2S2O3	VOL(ml) Na2S	CALC OF H2S mg/L				
10	5.30	2.0	408.10				
10	5.35	2.0					
10	5.40	2.0					
	X: 5.35						
Time of Analysis	Sample ID	Sample Wt. (g)	NaOH Trap Vol.(ml)	Analysis Vol.(ml)	ml I2 Soln Added	ml STS to Endpoint	Comments
09:00	Blank	10.0	190	150	10	6.65	09:50
	LCS	10.0			25	13.30	09:52
	SC139-1	10.313			10	6.80	09:55
	-1 dup	11.918			10	6.75	09:57
	-1 mb	10.272	∇	∇	25	14.15	09:59
NOTES:							
ANALYST: GJH			DATE: 03/25/09				
CHECKED BY:			DATE: 03/26/09				

TITLE: REACTIVE CYANIDE: SW-846, 7.3.3.2

FIGURE 3
REACTIVE CYANIDE APPARATUS



TITLE: REACTIVE SULFIDE: SW-846 7.3.4.2

Prepared By: Wet Chemistry Date: 8/96
 Approved By:
 Group Supervisor: J. Banta For K. Tanguay Date: 2/01
 Operations Manager: John C. Banta Date: 2/01
 QA Officer: Deborah J. Nadeau Date: 2.15.01
 General Manager: Debra J. Keefe Date: 2.15/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
02	Format changes, added pollution prevention and database. Deleted titration and added reference to analytical titrimetric SOP.	DN	2/15/01	2/15/01
03	Major changes to apparatus & procedure to reflect current practice. Added reference for CA-107 - sect. 1.4. Added program specific info to sect. 1.3 Supervisor => manager. Changes throughout to reflect current practice. Updated figures.	LAD	04/06	04/06
04	Sect. 1.1 - Added definitions. Minor changes throughout to reflect current practices. Updated method and Sop references. Updated Table 2 (method modifications)	LAD	05/09	05/09
05	Added definitions to section 1.1. Updated Table 1 for DoD QSM version 4.1 compliance. Added references to section 10.	DN	08/09	08/09

TITLE: **REACTIVE SULFIDE: SW-846 7.3.4.2**

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TITLE: REACTIVE SULFIDE: SW-846 7.3.4.2

1.0 SCOPE AND APPLICATION

The intended application of this method is to determine the hydrogen sulfide released from wastes. This method is applicable to all waste except those that will form explosive mixtures when combined with acids. This test measures only the hydrogen sulfide evolved at the test conditions. It is not intended to measure forms of sulfide other than those that are evolved under the test conditions. This method provides a means to determine the specific rate of release of hydrogen sulfide upon contact with an aqueous acid. The regulatory limit for *Total Releasable Sulfide* is 500 H₂S mg/Kg waste.

1.1 Definitions

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.

Laboratory Control Sample (LCS): A standard material of known concentration that has been brought through the sample preparation and analysis process. The LCS is used to assess the accuracy of the method. One LCS is required per batch.

LOD – Limit of Detection. The smallest amount or concentration of an analyte that must be present in a sample to be detected at a 99% confidence level. At the LOD, the false negative rate is 1%.

MB – Method Blank - Reagent water that has been brought through the sample preparation and analysis process. The MB is used to assess contamination.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the MDL.

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.

Reactive Sulfide: Hydrogen Sulfide released under the test conditions defined under SW846 Chapter 7, 7.3.4.2 where the sample is exposed to mildly acidic conditions

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of reactive sulfide according to SW-846, 7.3.4.2. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of all Katahdin technical personnel involved in the analysis of reactive sulfide according to SW-846, 7.3.4.2 and sulfide according to SW-846, 9034

TITLE: REACTIVE SULFIDE: SW-846 7.3.4.2

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. These materials include the following: Sodium Hydroxide, Sulfuric Acid, Hydrochloric Acid, Sodium Thiosulfate, Potassium Bi-iodate, Iodine and Sulfide.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and follow appropriate procedures such as: wearing safety glasses and gloves when working with chemicals or near an instrument; not taking food or drink into the laboratory; each analyst should know the location of all safety equipment and be trained on how to use it.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

The basic waste generated from this analysis is placed in satellite "G" or pyridine waste. The acidic waste is put in satellite "A" or acid waste.

Other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal" and CA-107, "The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents and Standards," current revisions. Expired standards are lab

TITLE: REACTIVE SULFIDE: SW-846 7.3.4.2

packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with these SOPs.

2.0 SUMMARY OF METHOD

An aliquot of acid is added to a fixed weight of waste in a closed system. The gas that is generated is swept into an alkaline scrubber. The specific rate of release of hydrogen sulfide is determined. The sulfide is quantified using method 9034, Katahdin SOP CA-722, "Titrimetric Determination of Sulfide using EPA Method 376.1, SM4500S2 F, SW846 9034 and SW 7.3.4".

3.0 INTERFERENCES

Interferences are undetermined.

4.0 APPARATUS AND MATERIALS

- 4.1 Magnetic stirrer; to achieve approximately 30 rpm.
- 4.2 Magnetic stirring bars and retriever.
- 4.3 Flexible tubing for connection from the nitrogen supply to the apparatus and from the flask to the absorber impinger unit.
- 4.4 Nitrogen gas tank with regulator.
- 4.5 Gas valve capable of metering N₂ flow to 20 psi
- 4.6 Flowmeter capable of measuring flow at 60 mL/min at the distillation station.
- 4.7 12 gas scrubbers, with 250mL graduated cylinders
- 4.8 Analytical balance weighing to 0.001g.
- 4.9 10-mL buret
- 4.10 Stir plate for titration
- 4.11 Buret stand and holder
- 4.12 Disposable pasteur pipets
- 4.13 1 mL and 5 mL calibrated Eppendorf pipets and tips

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- 4.14 250 mL graduated cylinder (Class A)
 - 4.15 400 mL beakers
-

5.0 REAGENTS

- 5.1 Laboratory reagent grade water: Equivalent in protocol as reagent or DI water
 - 5.2 Sulfuric acid (0.1 N), H₂SO₄: Add 5.6 ml of concentrated H₂SO₄ to laboratory reagent grade water and dilute to 2 liters.
 - 5.3 Sulfuric acid (0.01 N), H₂SO₄: Volumetrically transfer 200 ml of 0.1 N H₂SO₄ and dilute to 2 liters with laboratory reagent grade water to make the 0.01 N H₂SO₄.
 - 5.4 Sodium hydroxide solution (0.25 N), NaOH: Dilute 25.0 ml of 10 N NaOH to 1 liter of Laboratory reagent grade water. This solution may also be prepared by dissolving 10 g of NaOH in laboratory reagent grade water and diluting to 1 liter.
 - 5.5 6N hydrochloric acid - CAUTION: In a fume hood add 500 mls of concentrated HCl to 500 ml laboratory reagent grade water, slowly mix and allow to cool.
 - 5.6 Standard iodine solution 0.0250N: Dissolve 20 - 25 g KI in 1 L of laboratory reagent grade water and add 3.2 g iodine. After iodine has dissolved, standardize against 0.0375N sodium thiosulfate using the starch solution as an indicator. (See Katahdin SOP CA-722, Titrimetric Determination of Sulfide, for the standardization procedure).
 - 5.7 0.0375N sodium thiosulfate titrant, (Na₂S₂O₃): purchased
 - 5.8 Potassium iodide, KI, granular certified ACS grade
 - 5.9 Starch, 0.5%, preserved with chloroform: purchased
 - 5.10 Sodium sulfide (Na₂S) standard: Dissolve 3.75 g reagent grade sodium sulfide nonahydrate (Na₂S · 9H₂O; FW240.18) into 500 mL laboratory reagent grade water. This is equivalent to an estimated value of 1001 mg/L S or 1064 mg/L H₂S. This must be standardized in accordance with the procedure described in Section 7 below. The sodium sulfide standard is stable for 6 months from the date of preparation. Store in an amber glass container in the refrigerator.
-

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6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

- 6.1 Samples should be collected with a minimum of aeration. The filled sample bottle should contain no headspace and should be kept cool and in the dark until analysis. Begin analysis as soon as possible. Perform analysis in a ventilated hood.
- 6.2 Samples can be preserved by adjusting the pH to 12 with NaOH and adding zinc acetate. This may cause dilution of the sample, increase the ionic strength, and possibly change other physical or chemical properties of the waste that may affect the release of hydrogen sulfide. Samples are stored at 4°C in the dark.
-

7.0 PROCEDURES

7.1 STANDARDIZATION OF IODINE

- 7.1.1 Repeat the titration in triplicate. Base normality on the mean of the titrations unless an outlier is established by statistical rationale.
- 7.1.2 Using a class A volumetric pipet 10.0 mL of the Standard Iodine Solution (5.6) into a 400 mL beaker. Add 2 mL 6 N HCl and add 200 mL Laboratory reagent grade water.
- 7.1.3 Place beaker on a stir plate; stir gently so as not to excessively aerate the sample. Titrate with 0.0375N sodium thiosulfate from a 10- mL buret with the tip submerged. Titrate until a straw color (pale yellow) develops.
- 7.1.4 Add approximately ~1 mL of starch indicator with a disposable pasteur pipet. The color will turn blue. Titrate with sodium thiosulfate until blue color disappears and the solution is clear and colorless.
- 7.1.5 Record to two decimal places the total volume of sodium thiosulfate used for each of the three replicates. Using the average of the of the mLs of sodium thiosulfate used in the triplicate determinations calculate the normality of the I₂

$$\text{Normality } I_2 = \frac{mLs \text{ Na}_2\text{SO}_3 \times \text{Normality Na}_2\text{SO}_3}{mLs \text{ I}_2 \text{ Titrated}}$$

solution as follows:

7.2 STANDARDIZATION OF SODIUM SULFIDE

- 7.2.1 To standardize the Sodium Sulfide Standard (5.10) repeat the titration in triplicate. Base normality on the mean of the titrations unless an outlier is established by statistical rationale. The analysis is accomplished by placing

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an excess of iodine based upon the sulfide concentration in the flask and back titrating the excess iodine with sodium thiosulfate.

- 7.2.2 Using an adjustable pipet, add 10.0 mL of the Standard Iodine Solution (5.6) into a 400 mL beaker. Add 2 mL 6 N HCl and add 200 mL Laboratory reagent grade water.
- 7.2.3 Quantitatively aliquot 2.0 mL of standard sulfide solution (5.10) dispensing below the surface of the liquid in the Erlenmeyer.
- 7.2.4 Place Erlenmeyer flask on a stir plate; stir gently so as not to excessively aerate the sample. Titrate with 0.0375N sodium thiosulfate from a 25- mL buret with the tip submerged. Titrate until a straw color (pale yellow) develops.
- 7.2.5 Add approximately ~1 mL of starch indicator with a disposable pasteur pipet. The color will turn blue. Titrate with sodium thiosulfate until blue color disappears and the solution is clear and colorless.
- 7.2.6 Record to two decimal places the total volume of sodium thiosulfate used for each of the three replicates. Using the average of the of the mLs of sodium thiosulfate used in the triplicate determinations calculate the concentration of sulfide as S^{2-} mg/L as follows:

$$mg S^{2-} / L = \frac{(A \times B) - (C \times D) \times 16,000}{mLs Na_2S}$$

Where:

- A = mLs iodine solution
B = normality of iodine solution
C = mls $Na_2S_2O_3$ solution
D = normality of $Na_2S_2O_3$ solution, and
16,000 = mg equivalence S^{2-} , 32,066 mg / 2 equivalence

- 7.2.7 To convert the S^{2-} mg/L to H_2S multiply determined concentration of the sulfide solution times 1.06 where 1.06 = 34.08 g (MW H_2S)/32.07 g (MW S).
- 7.2.8 The concentration as H_2S is entered into the spreadsheet for further calculations.

7.3 SAMPLE PREPARATION FOR GENERATION OF RELEASABLE SULFIDE

- 7.3.1 Weigh approximately 10 g of sample in a 250 mL addition graduated cylinder. Record weight in the preparation logbook.

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- 7.3.2 To prepare Method Blank transfer 10 g of Laboratory reagent grade water to a 250mL addition graduated cylinder.
- 7.3.3 To prepare LCS transfer 10.0 mL of Sodium Sulfide Standard (5.10) to a 250mL addition graduated cylinder.
- 7.3.4 To prepare a Matrix Spike (MS), weigh approximately 10 g of sample in a 250mL addition graduated cylinder. Spike the sample with 10.0 mL of Sodium Sulfide Standard Solution (5.11).
- 7.3.5 To prepare sample Duplicate weigh out approximately 10 g of the sample selected/designated as the sample duplicate in a 250mL addition graduated cylinder
- 7.3.6 Add 190 ml of 0.25 N NaOH to each of the absorber graduated cylinders
- 7.3.7 Turn on the main valve on the Nitrogen tank. Make sure it is reading 300 psi or greater.
- 7.3.8 Adjust the local N₂ pressure valve in the hood and set the pressure to 20 psi on the low pressure gauge.
- 7.3.9 Turn the Outlet Valve of the flowmeter until the flow registers 60 mL/min.
- 7.3.10 Add 180mL of 0.01N H₂SO₄ to the addition graduated cylinders and connect the apparatus as shown in fig. 3.
- 7.3.11 Use a timer set for 30 minutes. After 30 minutes, disconnect all of the scrubbers on the apparatus.
- 7.3.12 Close off the main valve on the nitrogen tank followed by the pressure adjusting knob and then the outlet valve.
- 7.3.13 When requested a portion of the scrubber is transferred to 40-mL VOA vial for reactive CN analysis. The remainder is covered and titrated ASAP for reactive sulfide.

7.4 ANALYSIS OF SULFIDE

- 7.4.1 Releasable sulfide concentration is determined titrimetrically by analyzing a portion of the scrubber solution in accordance with the protocols delineated in the most current revision of Katahdin SOP CA-722, Titrimetric Determination of Sulfide Using EPA Method 376.1; SW846 9034. The scrubber aliquot is acidified to a pH of 2 using 15.0 mL 6N HCl prior to the start of the

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iodometric titration. In the case where only reactive sulfide is performed take the entire 190 mL scrubber solution for titration else take 150 mL.

7.4.2 A 40 mL portion of the scrubber solution may also be used for Reactive Cyanide analysis. See SOP CA-733, Reactive Cyanide: SW-846, 7.3.4.2.

7.4.3 The rate of release of H₂S (mg/Kg/sec) is calculated as follows:

$$R = \text{specific rate of release, mg / Kg / Sec} = \frac{A \times V}{W \times S}$$

Where:

A = concentration of H₂S in the scrubber as mg/L = 1.06 x S²⁻ mg/L
(1.06=MW H₂S/MW S=34.08 g /32.07 g)

V = volume in scrubber, Liters, i.e. 0.25

W= weight of waste, Kg

S = Time of measurement, Time N₂ stopped - Time N₂ started, sec

7.4.4 The releasable H₂S as mg/Kg is calculated as follows:

$$\text{Total Releasable H}_2\text{S, mg / Kg} = \frac{A \times V}{W}$$

Where:

A = concentration of H₂S in the scrubber as mg/L = 1.06 x S²⁻ mg/L
(1.06=MW H₂S/MW S=34.08 g /32.07 g)

V = volume in scrubber, Liters, i.e. 0.25

W= weight of waste, Kg

7.4.5 The above calculations are typically done using a spreadsheet template on which the analyst enters the sample number, date prepared, date analyzed, initial sample weight, trap volume, volume of sample distillate, volume of standard iodine solution used, volume of sodium thiosulfate used, and the normality of both the iodine and sodium thiosulfate solutions. Results are reported to 2 significant figures with the method reported as SW 846, 7.3.4.2.

7.4.6 Enter spreadsheet results, including sample preparation information, measured sample concentrations, and quality control data, into the Katahdin Information Management System for calculation and reporting. Refer to the current revision of SOP CA-762 ("Wet Chemistry Data Entry and Review Using Katahdin Information Management System") for further information. A batch sheet is generated (Figure 3). Raw data and batch sheets are reviewed for completeness and accuracy by the Inorganic Department Manager or other qualified designee.

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7.4.7 All batch sheets and copies of the raw logbook data are filed with the Inorganic Department Manager for approximately 3 months, for reference by analysts. Prior data are archived.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, remaining sample volume and client and project specific Data Quality Objectives. The 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.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality

Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

Every instance of noncompliant method quality control requires the generation of a Corrective Action Report describing the problem, suspected cause and final resolution. Corrective action reports must be signed by the initiator, Department Manager, QA officer, and lab management.

- 8.1 A method blank consisting of laboratory reagent grade water is analyzed with every batch or analytical session. The concentration of the blank must be less than the Practical Quantitation Limit (27 mg/kg)
- 8.2 A duplicate sample is also analyzed with every batch and duplicate samples are expected to agree within 20% relative difference.
- 8.3 A matrix spike sample is also analyzed with every batch of twenty samples. Acceptance criteria for spikes are 50 - 150% recovery.

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- 8.4 The efficiency of this method is measured by first standardizing the Sodium Sulfide Standard (5.10) using three 2.0 mL aliquots diluted to 200 mL as described in steps 7.2.3-7.2.7. After standardization, a 10.0 mL aliquot of the Sodium Sulfide Standard (5.10) is distilled and then analyzed as the LCS. A recovery of 50 - 150% is adequate to demonstrate proper system operation.
- 8.5 If any of the QC requirements are outside the recovery ranges listed above in Section 8.0, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Refer to table 1 for corrective actions. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Due to the short hold time associated with this method, samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.
-

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Inorganic 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 revisions of SW-846, 7.3.4.2 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, US EPA SW-846, 3rd Edition, Volume 1C, Chapter Seven, Section 7.3.4.2, "Test Method to Determine Hydrogen Sulfide Released from Wastes", Rev. 3, December, 1996.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods, US EPA SW 846, 3rd edition, Volume 1C, Chapter Seven, Section 7.3.3.2, Rev. 2, September 1994.

Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, US EPA SW-846, 3rd Edition, Method 9012, Rev. 0, September 1986.

Katahdin SOP CA-722, "Titrimetric Determination of Sulfide Using EPA Method 376.1, SM4500-S²⁻F, SW846 9034 and SW846 7.3.4".

Katahdin SOP CA-101, "Equipment Maintenance", current revision.

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Katahdin SOP CA-762, "Wet Chemistry Data Entry and Review Using Katahdin Information Management System (KIMS)"

Katahdin SOP QA-806, "Method Detection Limit and Instrument Detection Limit Studies"

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 and Tables

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TABLE 1
QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Reactive Sulfide SW-846 7.3.4.2	Method blank	One per prep batch of 20 or fewer samples	No analyte detected >PQL (For DoD QSM, no analyte detected > ½ PQL and > 1/10 the amount measured in any sample)	(1) Investigate source of contamination (2) Report all sample results <PQL. (3) Report sample results >10X the blank result and flag results with a "B". (4) If possible, reanalyze all other samples associated with the failing blank.
	LCS	One per prep batch of 20 or fewer samples	50%-150% rcvy, statistically derived from lab data	(1) If the LCS fails high, report samples that are <PQL. (2) Reanalyze /or recalibrate and reanalyze (3) Redistill, recalibrate and/or reanalyze other samples.
	Matrix Spike	One for every set of 10 samples	50%-150% rcvy.	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. (3) Analyze unspiked sample scrubber solution with post-scrubber spike to confirm matrix interference (4) Notate sample result in raw data if matrix interference confirmed
	Sample Duplicate	One sample duplicate per ten samples	RPD ≤ 20%	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD still >20, report original result with notation or narration.
	Demonstration of analyst proficiency – 4 replicates.	Once per analyst.	P&A meet method criteria	Repeat analysis until able to perform passing QC; document successful performance in personal training file
	MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

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TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-734-05	METHOD 7.3.4.2, current revision
Apparatus/Materials	Gas washing bottles are used	Glassware with ground glass connections is used
Reagents	1) 0.0375 N sodium thiosulfate purchased commercially. 2) Sodium Sulfide standard is prepared by dissolving 3.75g of Sodium Sulfide into 500 mL reagent water	1) 0.025 N sodium thiosulfate prepared in lab. 2) Sodium Sulfide standard is prepared by dissolving 4.02g of Sodium Sulfide into 1000 mL reagent water
Sample preservation/handling		
Procedures	1) 190 mL of 0.25 N NaOH are added to each scrubber (absorber bottle) prior to distillation. 2) 15.0 mL of 6N HCl are added to the scrubber solution (sample distillate) to bring pH to <2; actual pH of scrubber solution is not verified. 3) 180 mL of 0.01N H ₂ SO ₄ is added to each of the addition graduated cylinders	1) 50 mL of 0.25 N NaOH are added to each scrubber, then diluted with water to obtain an adequate depth of liquid in the scrubber. 2) A small amount of scrubber solution (sample distillate) is titrated with 6N HCl to determine volume of HCl needed to acidify entire scrubber solution to pH <2; the small acidified aliquot is then combined with the remainder of the acidified scrubber solution. 3) Add enough sulfuric acid to fill the flask half full
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		

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FIGURE 1

EXAMPLE OF LOGBOOK PAGE

KATAHDIN ANALYTICAL							
REACTIVE SULFIDE							
EPA: 7.3.4.2/9030				PQL: 27 mg/kg			
REAGENTS:							
IODINE STANDARD: W7828				STS SOLUTION-0.0375N: SOL 2671			
Na2S SOLUTION: W7757				NaOH-0.25N: W7715			
HCl-6N:				H2SO4-0.01N: W7827			
STANDARDIZATION OF I2							
VOL(ml) I2	VOL(ml) Na2S2O3		CALC OF I2 N				
10	6.70		0.02519				
10	6.70						
10	6.75						
	X: 6.72						
STANDARDIZATION OF H2S							
VOL(ml) I2	VOL(ml) Na2S2O3	VOL(ml) Na2S	CALC OF H2S mg/L				
10	5.10	2.0	519.40				
10	5.25	2.0					
10	4.90	2.0					
	X: 5.08						
Time of Analysis	Sample ID	Sample Wt. (g)	NaOH Trap Vol.(ml)	Analysis Vol.(ml)	ml I2 Soln Added	ml STS to Endpoint	Comments
09:10	Blank	10.0	190	150	10	6.75	09:33
	LOG	10.0			25	11.65	09:36
	SC0797-1	10.223			10	6.85	09:41
	-1 dup	10.956			10	6.70	09:44
	10.020 -1 MS	#288			25	13.30	09:50
	V SC0797-2	10.083			10	6.80	09:55
11:30	SC0805-7	10.383			10	6.65	14:00
	SC0834-13	10.345			10	6.70	14:05
	SC0833-1	10.206			10	6.65	14:10
	V SC0833-2	11.080	V	V	10	6.60	14:13
NOTES:							
ANALYST: GJH				DATE: 07/24/09			
CHECKED BY: [Signature]				DATE: 07/25/09			

QAWL479

WG61171 R9126L5

0000051

TITLE: REACTIVE SULFIDE: SW-846 7.3.4.2

FIGURE 3
EXAMPLE OF BATCH SHEET

WET CHEMISTRY BATCH REPORT
Feb 25 2009, 10:16 am
Batch: WG61171

Parameter: Sulfide,Reactive
Date Analyzed: 24-FEB-09
Analyst Initials: GJH
Prep Date: 24-FEB-09
Prep Method: SW846 7.3.4
Prep Chemist: GJH

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SC0797-1	SAMP	SW846 7.3.4	10.223g	190.00mL	.98	- .08	U27 mg/Kg	48.	27	16.39	27		
SC0797-2	SAMP	SW846 7.3.4	10.083g	190.00mL	.99	- .05	U27. mg/Kg	76.	27	16.39	27		
SC0805-7	SAMP	SW846 7.3.4	10.363g	190.00mL	.96	.04	U27 mg/Kg	91.	27	16.39	27		
SC0834-13	SAMP	SW846 7.3.4	10.345g	190.00mL	.97	.01	U27 mg/Kg	94.	27	16.39	27		
SC0858-1	SAMP	SW846 7.3.4	10.206g	190.00mL	.96	.04	U27 mg/Kg	87.	27	16.39	27		
SC0858-2	SAMP	SW846 7.3.4	11.090g	190.00mL	.9	.07	U27 mg/Kg	90.	27	16.39	27		
WG61171-1	MBLANK	SW846 7.3.4	10.000g	190.00mL	1	- .02	U27. mg/Kg	NA	27	16.39	27		
WG61171-2	LCS	SW846 7.3.4	10.000g	190.00mL	1	3.09	410 mg/Kg	NA	27	16.39	27		80
WG61171-3	DUP	SW846 7.3.4	10.956g	190.00mL	.91	.01	U27 mg/Kg	NA	27	16.39	27	NC	
WG61171-4	MS	SW846 7.3.4	10.020g	190.00mL	1	2.1	280 mg/Kg	NA	27	16.39	27		54

Comments:

WG61171-1 SC0797-1
WG61171-2 SC0797-1
WG61171-3 SC0797-1
WG61171-4 SC0797-1

Entered by: *d*

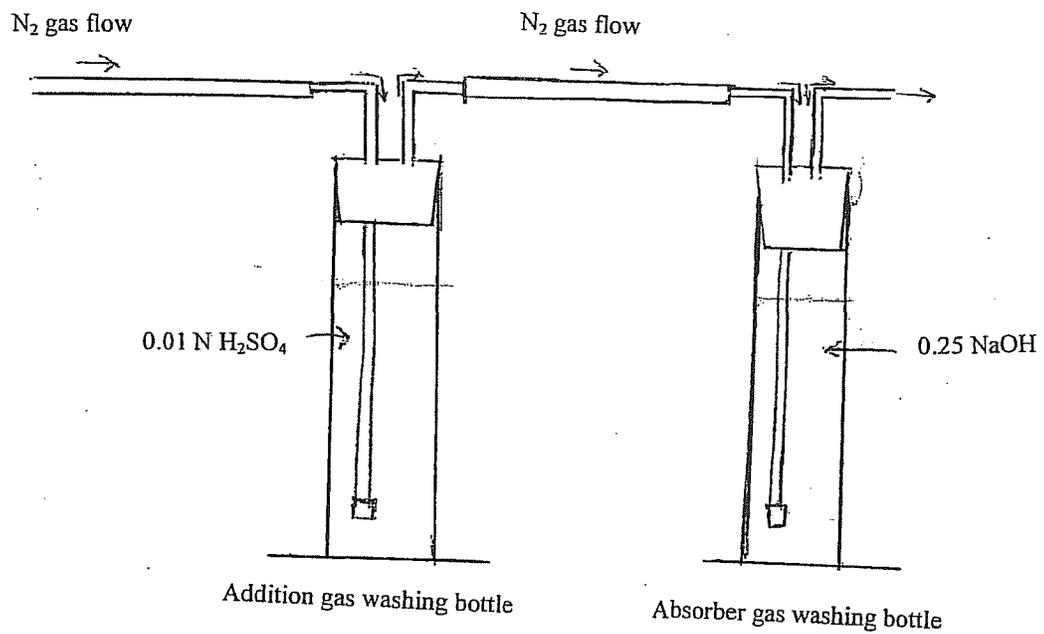
Date: 02/25/09

Accepted by: *[Signature]*

Date: 02/25/09

TITLE: REACTIVE SULFIDE: SW-846 7.3.4.2

FIGURE 4
REACTIVE SULFIDE APPARATUS



TITLE: TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER

Prepared By: Betsy Carbone Date: 8/9/0

Approved By:

Group Supervisor: Keith Ferguson Date: 012401

Operations Manager: J. Benton Date: 1/22/01

QA Officer: Dorothy J. Nadeau Date: 1.22.01

General Manager: Doreen L. Keenan Date: 1/22/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, added pollution prevention, p-xylene in duplicate, correction for barometric pressure	DN	1.22.01	1.22.01
02	Many changes throughout to reflect current practices. New fig. 2	LAD	031805	031805
03	Added method blank and LCS definitions	LAD	06108	06108

TITLE: TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER

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-736-03**, titled **TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **SOP CA-736-03**, titled **TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER**.

Recipient: _____ Date: _____

1.0 SCOPE AND APPLICATION

TITLE: TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER

The purpose of this SOP is to describe the procedure utilized by Katahdin Analytical Services, Inc. laboratory personnel to measure the tendency of a sample to form a flammable mixture with air under controlled laboratory conditions. The objective of the ignitability characteristic is to identify wastes that either present fire hazards under routine storage, disposal, or transportation or are capable of severely exacerbating a fire once started. The SOP is applicable to SW-846 method 1010 and ASTM method D 93-80.

The test method covers the determination of the flash point by Pensky-Martens closed-cup tester for fuel oils, lube oils, suspensions of solids, liquids that tend to form a surface film under test conditions and other liquids.

1.1 Definitions

Flash Point - The lowest temperature of the sample, corrected to a barometric pressure of 760 mm of Hg, at which application of the test flame ignites the vapor above the sample.

Laboratory Control Sample (LCS): LCS is a known standard carried through the entire analytical procedure in the same manner as a sample. The LCS determines the validity of the batch.

Method Blank - A Laboratory Reagent Grade Water sample that is carried through the entire analytical procedure in the same manner as a sample.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in flashpoint analysis by Pensky-Martens Closed-Cup method. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in flashpoint analysis by Pensky-Martens Closed-Cup method to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

TITLE: TEST METHOD FOR FLASH POINT BY PENSKEY-MARTENS CLOSED-CUP TESTER

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method have not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical 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 Waste Management Plan for further details on pollution prevention techniques.

p-Xylene must be stored separately and disposed of as a flammable liquid. All sample residues under this protocol are disposed of in satellite wastes for flammable liquids. Other wastes generated during the preparation of samples must be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

2.0 SUMMARY OF METHOD

Samples are heated at a slow, constant rate with continual stirring. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the test flame ignites the vapor above the sample.

3.0 INTERFERENCES

None determined.

TITLE: TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER

4.0 APPARATUS AND MATERIALS

- 4.1 Pensky-Martens Closed-Cup Flash Tester
- 4.2 Calibrated digital thermometer capable of reading up to 120°C
- 4.3 Barometer

5.0 REAGENTS

- 5.1 p-Xylene Reference Standard - Reagent grade, Flash point 27°C
- 5.2 Laboratory Reagent Grade Water

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Not applicable.

7.0 PROCEDURES

- 7.1 Record the ambient barometric pressure in inches of mercury. Multiply this number by 25.4 mm/in to obtain the barometric pressure in mm of mercury. Record this number in the appropriate place in the logbook (figure 1).
- 7.2 Preparation of Apparatus - Place the tester on the bench top located under a fume hood. Although the hood is turned off while performing the analysis, a draft is still present. The tester must be surrounded on three sides with a shield that is sufficient enough to prevent sputtering of the pilot flame.
- 7.3 Preparation of Sample - Samples of very viscous materials must be warmed until they are reasonably fluid before they are tested. However, no sample should be heated more than is absolutely necessary. Samples shall never be heated above a temperature of 17°C below the expected flash point.
- 7.4 Analytical Procedure
 - 7.4.1 Thoroughly clean and dry all parts of the cup and its accessories before starting the test, being sure to remove any solvent which had been used to clean the apparatus. Additional cleaning may be accomplished with the aid of sand or sandpaper.

TITLE: TEST METHOD FOR FLASH POINT BY PENSKEY-MARTENS CLOSED-CUP TESTER

- 7.4.2 Check to be sure that the orifice for the flame wick is not clogged. A piece of wire should fit into the opening.
- 7.4.3 For aqueous samples, fill the cup with the sample to be tested to the level indicated by the filling mark. For solid samples, fill the cup with the sample to be tested to the level indicated by the filling mark.
- 7.4.4 Place the lid on the cup and set the cup in the apparatus stove. Be sure to have the locking or locating device properly engaged.
- 7.4.5 Insert the thermometer.
- 7.4.6 If the flashpoint is known to be high, bring the material to be tested and the tester to a temperature of $25 \pm 5^{\circ}\text{C}$ or $15 \pm 5^{\circ}\text{C}$ lower than the estimated flash point, whichever is lower. Otherwise, start the flame when the samples are still cold (below room temperature)
- 7.4.7 Light the test flame and adjust it to 5/32 inch (4mm) in diameter.
- 7.4.8 Supply the stove heat at such a rate that the temperature increases 5 to $6^{\circ}\text{C}/\text{minute}$.
- 7.4.9 Turn the stirrer on (90-120 rpm), stirring in a downward direction.
- 7.4.10 Apply the test flame when the temperature is between 20 and 25°C .
- 7.4.11 Apply the test flame by operating the mechanism on the cover that controls the shutter and test flame burner so that the flame is lowered into the vapor space of the cup in 0.5 seconds, left in its lowered position for 1 second and quickly raised to its high position.
- NOTE:** Do not stir the sample while applying the test flame.
- 7.4.12 After 25°C , apply the test flame in increments of 2°C .
- 7.4.13 Continue applying the test flame at temperature increments of 2°C until the flash point of the sample or 71°C is reached, whichever comes first.
- 7.4.14 If the sample flashes between 25 and 71°C , obtain a fresh aliquot of the sample. Bring the sample material to a temperature $15 \pm 5^{\circ}\text{C}$ lower than the initially determined flash point. Apply the test flame and thereafter at temperature readings in increments of 2°C until the flash point of the sample is reached. Results obtained from Steps 7.4.13 and 7.4.14 should agree within $\pm 2^{\circ}\text{C}$.

TITLE: TEST METHOD FOR FLASH POINT BY PENSKEY-MARTENS CLOSED-CUP TESTER

7.4.15 Record the observed flash point as the temperature read on the thermometer at the time the test flame application causes a distinct flash in the interior of the cup. The lowest reading from the duplicate analyses (Steps 7.4.13 and 7.4.14) should be reported.

NOTE: Do not confuse the true flash with the bluish halo that sometimes surrounds the test flame at applications preceding the one that causes the actual flash.

7.4.16 If the sample flashes below 25°C, the reported value should be <25°C. If the sample did not flash, the reported value should be >71°C. If the sample was not heated to 71°C, record the highest temperature achieved.

7.4.17 The observed flash points must be corrected for the ambient barometric pressure. If the ambient barometric pressure at the time of analysis differs from 760 mm Hg (one atmosphere), the following formula must be used:

$$\text{Corrected flash} = (\text{observed flash} + 0.033 (760 \text{ mm Hg} - \text{ambient barometric pressure in mm Hg}))$$

Point (° C) point in ° C

Record all corrections in the logbook (Figure 1).

7.4.18 After completion of each test, the logbook must be signed and dated by the person performing the test.

7.4.19 The sample data results from the logbook, with any appropriate notations, are entered manually into the Katahdin Information Management System (KIMS) for calculation and reporting. Refer to the current revision of Katahdin Analytical Services SOP CA-762 ("Wet Chemistry Data Entry and Review Using Katahdin Information Management System") for further information.

7.4.20 All batch sheets and copies of the raw logbook data are filed with the Department Manager for approximately 3 months, for reference by analysts. Prior data are archived.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A typical analytical run consists of a tester calibration using p-Xylene (analyzed in duplicate), a blank consisting of laboratory reagent grade water (immediately following the p-Xylene), the samples to be analyzed and a duplicate sample analysis. A duplicate sample analysis is performed every ten samples, every daily batch, or for any sample that flashes, whichever is more frequent. If a sample flashes, that sample is run in duplicate. Refer to Table 1 for acceptance criteria and corrective actions.

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

TITLE: TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER

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

Refer to the current revision of Method 1010 for method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

ASTM, Test Methods for Flash Point by Pensky-Martens Closed Tester, D 93-80, 1981

“Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods”, SW-846, third Edition, Final Update III, December 1996, Pensky-Martens Closed-Cup Method for Determining Ignitability, Method 1010.

LIST OF TABLES AND FIGURES

Table 1	QC Requirements
Table 2	Summary of Method Modifications
Figure 1	Example of Flashpoint Logbook Page
Figure 2	Batch Sheet for Flashpoint

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TABLE 1
QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Flashpoint Pensky- Martens Closed-Cup, Method 1010	Method blank	One per prep batch	No flash	(1) Investigate source of contamination (2) Reprep and analyze method blank and all samples processed with the contaminated blank
	Sample Duplicate	One sample duplicate per ten samples	Results of sample and sample duplicate agree within ± 2 °C – Report the lowest value.	(1) If lab QC in criteria and duplicates do not agree within ± 2 °C , report the lowest value and narrate the other values. (2) Else, reanalyze
	LCS / p-xylene	In duplicate per batch of twenty samples or less	Flash point $27^{\circ}\text{C} \pm 2^{\circ}\text{C}$	1) Repeat analysis of reference standard and associated samples

TITLE: TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER

TABLE 2
SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-736-03	METHOD 1010, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures	Test flame is applied at 2 °C increments; if sample flashes during test, a second aliquot is tested for confirmation of flash point. The lowest value is reported unless the values do not agree within ± 2 °C. In these cases, the lowest value is reported and the others narrated.	Test flame is applied at 1 °C increments; single analysis is performed.
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER

FIGURE 1

EXAMPLE OF FLASHPOINT LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES, INC. - FLASHPOINT - CLOSED CUP LOGBOOK

METHOD: SW 846 1010		p-XYLENE: 27 Degrees Celcius		Ambient Barometric Pressure(*) = 765.81 mm Hg				
PQL: 25 Degrees Celcius		p-XYLENE Lot Number: SWL2444		(in. of Hg) X (25.4 mm/in) 30.15				
Sample ID	Analysis Time	Start Temperature (°C)	End Temperature (°C)	Flash? (Check appropriate box & record temp.)			Reported Flashpoint Corrected For Ambient Barometric Pressure	Comments
				YES	Temp.(°C)	NO		
p-Xylene (LCS)	800	25°C	29°C	/	29°C		28.80	WG 52291-2
p-Xylene (LCS)	915	25°C	29°C	/	29°C		29.80	↓ -3
Method Blank	930	25°C	71°C			X	>71.0°C	WG 52291-1
SB2919-1B	850	25°C	71°C			X	>71.0°C	
D4 -1B	915	25°C	71°C			X	>71.0°C	WG 52291-4
SB2930-1B	950	25°C	71°C			X	>71.0°C	
SB2947-2A	1045	25°C	71°C			X	>71.0	
SB2920-1A	1215	25°C	71°C			X	>71.0	
SB2932-1A	1300	25°C	71°C			X	>71.0	
↓ -2A	1330	25°C	71°C			X	>71.0	
WG 52291 R&Z084								
				Closed For		DEU SUB BY [Signature]		6-9-08

QAWL480

* = Ambient Pressure of the laboratory at the time of the test. When the pressure differs from 760 mm Hg, correct the flashpoint as follows:
Corrected Flashpoint = (Observed Flashpoint in °C) + 0.033 (760 - the ambient barometric pressure in mm Hg).

Analyst [Signature] Date 6-6-08
Reviewed By [Signature] Date 06/09/08

0000029

TITLE: TEST METHOD FOR FLASH POINT BY PENSKY-MARTENS CLOSED-CUP TESTER

FIGURE 2

BATCH SHEET FOR FLASHPOINT

MET CHEMISTRY BATCH REPORT
Jun 09 2008, 10:11 am
Batch: W552291

Parameter: Ignitability
Date Analyzed: 06-JUN-08
Analyst Initials: JF

Prep Date: N/A
Prep Method: N/A
Prep Chemist: N/A

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
SB2919-1	SAMP	SW846 1010	1.0000mL	1.0000mL	1	71	>71. Deg. C	NA	71	71	71		
SB2921-1	SAMP	SW846 1010	1g	1g	1	71	>71. Deg. C	88.	71	71	71		
SB2930-1	SAMP	SW846 1010	1.0000mL	1.0000mL	1	71	>71. Deg. C	NA	71	71	71		
SB2932-1	SAMP	SW846 1010	1g	1g	1	71	>71. Deg. C	16.	71	71	71		
SB2932-2	SAMP	SW846 1010	1g	1g	1	71	>71. Deg. C	20.	71	71	71		
SB2947-2	SAMP	SW846 1010	1.0000mL	1.0000mL	1	71	>71. Deg. C	NA	71	71	71		
W552291-1	MBLANK	SW846 1010	1.0000mL	1.0000mL	1	71	>71. Deg. C	NA	71	71	71		
W552291-2	LCS	SW846 1010	1.0000mL	1.0000mL	1	28.8	29. Deg. C	NA	71	71	71		107
W552291-3	LCSD	SW846 1010	1.0000mL	1.0000mL	1	28.8	29. Deg. C	NA	71	71	71	0	107
W552291-4	DUP	SW846 1010	1.0000mL	1.0000mL	1	71	>71. Deg. C	NA	71	71	71	NC	

Comments:

W552291-1 SB2919-1
W552291-2 SB2919-1
W552291-3 SB2919-1
W552291-4 SB2919-1

Entered by: *JF*

Date: 6-9-08

Accepted by: *JF*

Date: 06/09/08

ADDENDUM
SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Adrian Biscontini

Review Date: 3/11/09

SOP Number: CA-736

SOP Title: Ignitability

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature:

Date:

A. Brewer

03/16/09

QAO Signature:

Date:

Luseia Diamond

03.17.09

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 cwotchy 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 Band 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

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

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

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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.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

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:

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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.

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- 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.

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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

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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.

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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

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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

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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

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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.

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- 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.
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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, SO ₄ , NO ₂ , NO ₃)	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

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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

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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

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

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

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

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

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

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 = H2SO4 to pH<2
4 = HNO3 to pH<2
5 = NaOH to pH>12
6 = 1 mL 0.1M Na2S2O3 or 1 10 mg pellet
7 = 1 m/L 2NZnAc/L & NaOH
8 = 2 drops 1:1 HCl
9 = No headspace
10 = Na2S2O3, if chlorinated
11 = HCl to pH < 2
12 = 5 mL of HCL
13 = 15 mL of methanol
14 = methanol
15 = sodium bisulfate

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:			Requested Services		
Shipping:	UPS	Fed-Ex	Mail	Drop-Off	Standard Homeowner	Arsenic	Total Coliforms
Sample(s) Received on Ice?	Yes	No	Temperature if Iced:		Lead	Safety Test - Coliform & NHN	FHA/MSH
					Fluoride	Uranium	What's Included in the Standard Test and the FHA/MSH Test.
Sample Description (Sample Identification and/or Lot #)		Date Collected	Time Collected	No. of Cntrs.			
							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:

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 4

EXAMPLE OF KATAHDIN SAMPLE RECEIPT LOGBOOK

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 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.

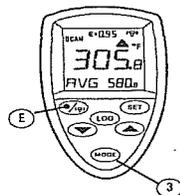
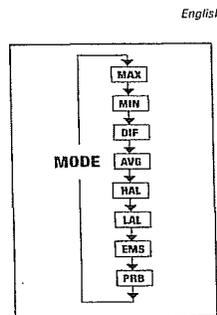
Pressing the MODE button also allows you to access the High Alarm (HAL), Low Alarm (LAL), Emissivity (EMS), Probe temperature (PRB—only available when the probe is connected), and Data logger (LOG). Each time you press MODE, you advance through the mode cycle. The diagram shows the sequence of functions 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.

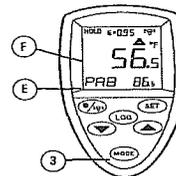
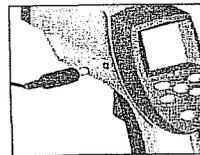
Selecting a Function

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.



9

English



10

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 11

EXAMPLE OF KATAHDIN WORK ORDER/LOGIN COC REPORT



Login Number: SA0395
Account: KATAHD001
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 GC Addresses:
Invoice GC Addresses:

Katahdin Analytical Services
Login Chain of Custody Report (Ino1)
Jan. 26, 2007
03:51 PM

Web

Login Information

ANALYSIS INSTRUCTIONS :
CHECK NO. :
CLIENT PO# :
COOLER TEMPERATURE : n/a
DELIVERY SERVICES : In-House
EDD FORMAT :
MAIL DATE :
PM : LAD
PROJECT NAME : QC Holding Blanks
QC LEVEL : 1
REGULATORY LIST :
REPORT INSTRUCTIONS :
SDG ID :
SDG STATUS :

Page: 1 of 1

Laboratory Sample ID	Client Sample Number	Collect Date/Time	Receive Date	Verbal PR Date	Due Date	Comments
SA0395-1	WHITE FRIDGE	26 JAN-07 15:50	26 JAN-07		08 FEB-07	
<i>Matrix</i>	<i>Product</i>	<i>Hold Date (shortest)</i>	<i>Bottle Type</i>	<i>Bottle Count</i>		
Aqueous	S SW8260FULLSML	09-FEB-07		2		
SA0395-2	BLUE FRIDGE	26 JAN-07 15:50	26 JAN-07		08 FEB-07	
<i>Matrix</i>	<i>Product</i>	<i>Hold Date (shortest)</i>	<i>Bottle Type</i>	<i>Bottle Count</i>		
Aqueous	S SW8260FULLSML	09-FEB-07		2		

Total Samples: 2 Total Analyses: 2

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 <u>no</u>
Jacobs		WW4883-1A	ICP	9/14/06 0100	→ 1000	DJJ	yes <u>no</u>
CES		WW4968	BOD	9/20/06 0930	9/20/06 1030	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 <u>no</u>
Irving		WW4994	BOD	9/21/06 1000	9/21/06 1015	CP	yes <u>no</u>
Hillmer		WW4992 [Ⓢ]	BOD	9/21/06 1015		CP	yes no
NATIONAL		WW5000	TS, PSEUDONITR PH, SP, & ATD	9/21/06 1100	9/21/06 1217	↓	yes <u>no</u>
WTC		WW5001	BOD	9/21/06 1300		CP	yes no
Arms		WW5016	N03	9/22/06 1100	09/22/06 1100	CP	yes <u>no</u>
RAISON		WW5010	↓	↓	↓	↓	yes <u>no</u>
EcoMaine		WW5029	BOD	9/22/06 1100		CP	yes no

QAQC143

0000095

TITLE: **SAMPLE DISPOSAL**

Prepared By: *Michael A. ...* Date: 2/01

Approved By:

Group Supervisor: _____ Date: _____

Operations Manager: *Jed C. Buntis* Date: 2/01

QA Officer: *Deborah J. Nadeau* Date: 2-01

General Manager: *Dennis F. Keegan* 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 disposal.	BH	2/01	2/01
02	Major rewrite to include more detail on hazardous waste regulations + to reflect current practices.	BH	02/05	02/05
03	Rewrite of section 7 to comply with current practices in new facility. Updated Figures 1 to 3.	BH	02-08	02-08
04	Added elementary neutralization to section 7.0. Other minor edits.	BH	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.

TITLE: SAMPLE DISPOSAL

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

TITLE: SAMPLE DISPOSAL

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.

TITLE: SAMPLE DISPOSAL

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.

TITLE: SAMPLE DISPOSAL

- 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.

TITLE: SAMPLE DISPOSAL

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.

TITLE: SAMPLE DISPOSAL

7.9.5 Dumping Basic samples (as determined by the basic preservative or by laboratory testing)

Aqueous samples get disposed of in waste stream "NH_i". 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 "NH_i". 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.

TITLE: SAMPLE DISPOSAL

- 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
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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		
SA5783-1	WC	10-17-07	✓									1-22-08	GN
SA5786-1		10-17-07	✓										
SA5787-1,2,4		10-17-07	✓										
SA5790-1		10-19-07		✓									
SA5793-1		10-17-07	✓										
SA5795-1-9		10-23-07	✓										
SA5797-1		10-23-07	✓										
SA5798-1,2		10-25-07			✓								
SA5799-1-5		10-31-07	✓										
SA5804-1,2		10-25-07	✓										
SA5809-1,2		10-25-07	✓				2				2		
SA5810-1-4		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)

TITLE: SAMPLE DISPOSAL

FIGURE 3

EXAMPLE OF HAZARDOUS WASTE STORAGE AREA DAILY CHECK

Daily Checklist for
HAZARDOUS WASTE STORAGE AREA

Week of: 1-28, 2008

QAQC315

Item/Day:	Monday	Tuesday	Wednesday	Thursday	Friday
1. Are containers closed? (Except when waste is being added)	<input checked="" type="radio"/> Yes / No				
2. Are containers properly labeled with a hazardous waste label?	<input checked="" type="radio"/> Yes / No				
3. Do you have access to each container and can you read the label? (3" x 5")	<input checked="" type="radio"/> Yes / No				
4. Is each container marked with the date storage begins?	<input checked="" type="radio"/> Yes / No				
5. Are the dates on the containers less than 90 days old?	<input checked="" type="radio"/> Yes / No				
6. Is container free of dents, bulges, rust, spills or leaks?	<input checked="" type="radio"/> Yes / No				
7. Are all containers on a firm working surface?	<input checked="" type="radio"/> Yes / No				
8. Inspection by: Name (No Initials)	<i>Dale Platin</i>				
9. Time of inspection	16:35	15:00	14:45	14:15	16:25
10. Verification of inspection (Name/Date)	<i>DL 1-28-08</i>				
Deficiency noted:					
Corrective action:					
By (Name/Date):					

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TITLE: SAMPLE DISPOSAL

FIGURE 4

CHARACTERISTIC TOXIC HAZARDOUS WASTE AND TCLP CONCENTRATIONS

Chemical Name	CAS Number	Waste Code	TCLP conc. liquid	Equivalent conc. In Soil
Arsenic	7440-38-2	D004	5.0 mg/L	100 mg/kg
Barium	7440-39-3	D005	100 mg/L	2000 mg/kg
Cadmium	7440-43-9	D006	1.0 mg/L	20 mg/kg
Chromium	7440-47-3	D007	5.0 mg/L	100 mg/kg
Lead	7439-92-1	D008	5.0 mg/L	100 mg/kg
Mercury	7439-97-6	D009	0.2 mg/L	4 mg/kg
Selenium	7782-49-2	D010	1.0 mg/L	100 mg/kg
Silver	7440-22-4	D011	5.0 mg/L	20 mg/kg
Endrin	72-20-8	D012	0.02 mg/L	0.4 mg/kg
Lindane	58-89-9	D013	0.4 mg/L	8 mg/kg
Methoxychlor	72-43-5	D014	10 mg/L	200 mg/kg
Toxaphene	8001-35-2	D015	0.5 mg/L	10 mg/kg
2,4-D	94-75-7	D016	10 mg/L	200 mg/kg
2,4,5-TP (Silvex)	93-72-1	D017	1.0 mg/L	20 mg/kg
Benzene	71-43-2	D018	0.5 mg/L	10 mg/kg
Carbon Tetrachloride	56-23-5	D019	0.5 mg/L	10 mg/kg
Chlordane	57-74-9	D020	0.03 mg/L	0.6 mg/kg
Chlorobenzene	108-90-7	D021	100 mg/L	2000 mg/kg
Chloroform	67-66-3	D022	6.0 mg/L	120 mg/kg
o-Cresol	95-48-7	D023	200 mg/L	4000 mg/kg
m-Cresol	108-39-4	D024	200 mg/L	4000 mg/kg
p-Cresol	106-44-5	D025	200 mg/L	4000 mg/kg
Cresol	1319-77-3	D026	200 mg/L	4000 mg/kg
1,4-Dichlorobenzene	106-46-7	D027	7.5 mg/L	150 mg/kg
1,2-Dichloroethane	107-06-2	D028	0.5 mg/L	10 mg/kg
1,1-Dichloroethylene	75-35-4	D029	0.7 mg/L	14 mg/kg
2,4-Dinitrotoluene	121-14-2	D030	0.13 mg/L	2.6 mg/kg
Heptachlor	76-44-8	D031	0.008 mg/L	0.16 mg/kg
Hexachlorobenzene	118-74-1	D032	0.13 mg/L	2.6 mg/kg
Hexachlorobutadiene	87-68-3	D033	0.5 mg/L	10 mg/kg
Hexachloroethane	67-72-1	D034	3.0 mg/L	60 mg/kg
Methyl Ethyl Ketone	78-93-3	D035	200 mg/L	4000 mg/kg
Nitrobenzene	98-95-3	D036	2.0 mg/L	40 mg/kg
Pentachlorophenol	87-86-5	D037	100 mg/L	2000 mg/kg
Pyridine	110-86-1	D038	5.0 mg/L	100 mg/kg
Tetrachloroethylene	127-18-4	D039	0.7 mg/L	14 mg/kg
Trichloroethylene	79-01-6	D040	0.5 mg/L	10 mg/kg

TITLE: SAMPLE DISPOSAL

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	

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 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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 289