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SAMPLING AND ANALYSIS PLAN

(Field Sampling Plan and Quality Assurance Project Plan)

Background Study

Naval Air Station
Brunswick, Maine

Naval Facilities Engineering Command
Mid-Atlantic

Contract Number N62467-04-D-0055
Contract Task Order 432

July 2009

BASAP Worksheet #1 -- Title and Approval Page
(UFP-QAPP Manual Section 2.1)

SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
October 16, 2008

Background Study
Naval Air Station Brunswick
Brunswick, Massachusetts

Prepared for:
Naval Facilities Engineering Command
Mid-Atlantic Division
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Prepared under:
Contract Number N62467-04-D-0055
"CLEAN" Contract Task Order No. 432

QAPP Worksheet #1b (UFP-QAPP Manual Section 2.1) -- Approval Page

Document Title: Final Sampling and Analysis Plan, (Field Sampling Plan and Quality Assurance Project Plan), October 2008, Background Study

Lead Organization: Naval Facilities Engineering Command Mid-Atlantic

Preparer's Name and Organizational Affiliation: Tetra Tech NUS, Inc.

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Preparation Date (Day/Month/Year): 07/14/2009

As per the State of Maine Department of Professional and Financial Regulations, Title 32, Chapter 73, the undersigned as reviewed the geological content of the attached report entitled: "*Sampling and Analysis Plan Background Study, Naval Air Station Brunswick, Brunswick, Maine*". Tetra Tech chemists prepared and reviewed the chemical analytical content of this document. Tetra Tech statisticians prepared the reviewed the statistical content of this document.

_____

Charles D. Race Date
State of Maine Certified Geologist No. GE243
Lead Project Hydrogeologist
TETRA TECH NUS, INC.
Wilmington, Massachusetts

Investigative Organization's Project Manager:

Signature/Date

Jeff Orient, P.G., Tetra Tech NUS, Inc.

Investigative Organization's Project QA Manager:

Signature/Date

Tom Johnston, PhD, Tetra Tech NUS, Inc.

Lead Organization's Project Manager:

Signature/Date

Todd Bober, U.S. Navy BRAC PMO Northeast

Lead Organization QA Officer:

Signature/Date

Sherri Eng, U.S. Navy Atlantic

Approval Signatures:

Signature/Date

Michael Daly, U.S. EPA

Signature/Date

Claudia Sait, MEDEP

EXECUTIVE SUMMARY

Sampling and Analysis Plan (UFP-SAP) describes Field Sampling Plan (FSP) requirements for the Background Study at Naval Air Station (NAS) Brunswick, in Brunswick, Maine. This SAP has been prepared by Tetra Tech NUS, Inc. (TtNUS) on behalf of the Base Realignment and Closure (BRAC) Program Management Office Northeast (PMO NE) under the Comprehensive Long-Term Environmental Action Navy (CLEAN) Contract Number N62467-04-D-0055, Contract Task Order (CTO) 432. This SAP was generated and complies with applicable Navy, State of Maine, and U.S. Environmental Protection Agency (EPA) Region 1 requirements and guidance, especially those concerning the UFP set forth by the EPA and Department of Defense/Department of Energy, and the Navy Policy on the use of Background Chemical Levels (CNO 2004).

The background study described herein is being performed to gather data on basewide background conditions in soil, groundwater, seeps, surface water, and sediment for current and future application to environmental site investigations and cleanups funded under the Navy's Environmental Restoration program and the Defense Base Closure and Realignment Act (BRAC) of 1990, Public Law 101-510 as amended. The primary goal of the study is to establish robust and statistically based background data sets for selected chemical constituents for soils from predominant soil types, groundwater from identified hydrologic units, surface water, and sediment from non-site areas for comparisons with environmental site data. Chemicals of interest include metals, pesticides and herbicides, and polycyclic aromatic hydrocarbons (PAHs), all of which may be present in media due to naturally occurring or anthropogenic processes. The data collection design will also support simple mathematical comparisons of site data to background concentrations ranges as well as more sophisticated data analyses as detailed within this SAP. To support this goal, the study anticipates and outlines various types of statistical methods for comparison of site data to background data, which are presented in greater detail in supporting guidance documents such as Navy guidance.

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APPENDICES (Submitted on CD)

A Sampling and Related SOPs
B Field Documentation Forms
C Analytical Specification
D Laboratory SOPs

ACRONYMS

amu	Atomic Mass Units
ASTM	American Society for Standards and Materials
BACSE	Brunswick Area Citizens for a Safe Environment
BEC	BRAC Environmental Coordinator
bgs	below ground surface
BRAC	Base Realignment and Closure
BTV	Background Threshold Values
ºC	degrees Celsius
CA	Corrective Action
CCC	Continuing Calibration Check
CCV	Calibration Check Verification
C.G.	Certified Geologist
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CERFA	Community Environmental Response Facilitation Act
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-Term Environmental Action Navy
CLLE	Continuous liquid-liquid extractor
CLP	Contract Laboratory Program
CNO	Chief of Naval Operations
COC	Contaminant of Concern
COPC	Contaminant of Potential Concern
CSM	Conceptual Site Model
CTO	Contract Task Order
DL	Detection Limits
DO	Dissolved Oxygen
DoD	Department of Defense
DPT	Direct Push Technology
DQI	Data Quality Indicator
DQO	Data Quality Objective
ECP	Environmental Condition of Property
EPA	Environmental Protection Agency
FFA	Federal Facilities Agreement
FOL	Field Operations Leader
FS	Feasibility Study

GC	Gas Chromatography
GC/ECD	Gas Chromatography/Electron Capture Detector
GC/MS	Gas Chromatograph/Mass Spectrometry
GIS	Geographic Information System
GPS	Global Positioning System
GW	Ground Water
HASP	Health and Safety Plan
HSM	Health and Safety Manager
IAS	Initial Assessment Study
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICP-MS	Inductively Coupled Plasma Mass Spectroscopy
ICS	Interference Check Solutions
ICV	Independent Calibration Verification
ID	Inside Diameter
IQR	Interquartile test
IR	Installation Restoration
IRP	Installation Restoration Program
IS	Internal Standards
K	Hydraulic Conductivity
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LTM	Long-Term Monitoring
MDD	Minimum Detectable Difference
MDL	Method Detection Limit
MEC	Munitions and Explosives of Concern
MECDC	Maine Center for Disease Control
ME CG	Maine Certified Geologist
MEDEP	Maine Department of Environmental Protection
MC	Munitions Constituents
MPC	Method Performance Criteria
MRP	Munitions Response Program
MRRA	Midcoast Regional Redevelopment Authority
MS	Matrix Spike
MSD	Matrix Spike Duplicate
msl	mean sea level
MW	Monitoring Well

NA	Not Applicable
NAPL	Non-Aqueous Phase Liquid
NAS	Naval Air Station
NAVFAC	Naval Facilities Engineering Command
NB	Number of Background Samples
NEESA	Naval Engineering & Environmental Support Activity
NEX	Navy Exchange
NFA	No Further Action
NFESC	Naval Facilities Engineering Service Center
NPL	National Priorities List
NS	Number of Site Samples
NTU	Nephelometric Turbidity Units
ORNL	Oak Ridge National Laboratory
ORP	Oxidation-Reduction Potential
OSHA	Occupational Safety and Health Administration
P-Level	Probability Level
PAH	Polycyclic Aromatic Hydrocarbon
PAL	Project Action Limit
PARCCS	Precision, Accuracy, Representativeness, Completeness, Comparability, and Sensitivity
PBW	Preparation Blank
PCBs	Polychlorinated Biphenyls
PDF	Portable Document Format
PID	Photoionization Detector
PM	Project Manager
PMO	Program Management Office
POL	Petroleum, Oil and Lubricant
PPE	Personal Protective Equipment
PQL	Practical Quantitation Limit
PQOs	Project Quality Objectives
PT	Proficiency Testing (previously known as performance evaluation (PE) sample)
PVC	Polyvinyl Chloride
QA	Quality Assurance
QAM	QA Manager
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit

QSM	Quality Systems Manual
RCRA	Resource Conservation and Recovery Act
RF	Response Factor
RI	Remedial Investigation
ROD	Record of Decision
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RSD	Relative Standard Deviation
RSL	Regional Screening Levels
RT	Retention Time
SAP	Sampling and Analysis Plan
SD	Standard Deviation
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SVOA	Semivolatile Organic Analytes
SVOC	Semivolatile Organic Compounds
SWMU	Solid Waste Management Unit
TAL	Target Analyte List
TBD	To Be Determined
TCL	Target Compound List
TOC	Total Organic Carbon
TtNUS	Tetra Tech NUS, Inc.
UFP	Uniform Federal Policy
UPL	Upper Prediction Limits
VOA	Volatile Organic Analytes
VOC	Volatile Organic Compounds
WRS	Mann-Whitney test

SAP Worksheet #2 -- SAP Identifying Information

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number: **Naval Air Station (NAS) Brunswick**
Operable Unit: **Background Study**
Contractor Name: **Tetra Tech NUS, Inc. (TtNUS)**
Contract Number: **N62467-04-D-0055**
Contract Title: **Naval Facilities Engineering Command (NAVFAC) Mid-Atlantic Comprehensive Long-Term Environmental Action Navy (CLEAN)**
Work Assignment Number (optional): Contract Task Order (CTO) 432

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

2. Identify regulatory program: 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
Navy – Maine Department of Environmental Protection (MEDEP) – EPA Region 1 – TtNUS – ECC	October 16 - 17, 2007
Brunswick Area Citizens for a Safe Environment (BACSE) (Citizens group) – Midcoast Regional Development Authority (MRRA) Local Redevelopment Authority	
Conference call with Navy: Base Realignment and Closure (BRAC) Program management Officer (PMO) NE, NAVFAC Chemists\TtNUS	June 24, 2008

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

Title	Date
Draft Background Study Work Plan (ECC)	March 2007
Internal Draft SAP (TtNUS)	April 2008

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

EPA Region 1, Regulatory Oversight
MEDEP, Regulatory Oversight
NAS Brunswick, Property Holder
NAVFAC, Responsible Party Representative
TtNUS, Contractor to NAVFAC
MRRA, Brunswick Local Redevelopment Authority, Designated Property Recipient
BACSE, Citizens Group
BRAC Program Management Office, Northeast (BRAC PMO NE), Lead Agency

7. Lead organization (see WS 7 for detailed list of data users)

Navy BRAC PMO NE

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:

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

Sample Collection		
26	Sample Handling System, Documentation Collection, Tracking, Archiving and Disposal Sample Handling Flow Diagram	**Not applicable**
27	Sample Custody Requirements, Procedures/SOPs Sample Container Identification Example Chain-of-Custody Form and Seal	**Not applicable**
Quality Control Samples		
28	QC Samples Table Screening/Confirmatory Analysis Decision Tree	**Not applicable**
Data Management Tasks		
29	Project Documents and Records Table	**Not applicable**
30	Analytical Services Table Analytical and Data Management SOPs	**Not applicable**
C. Assessment Oversight		
31	Planned Project Assessments Table Audit Checklists	**Not applicable**
32	Assessment Findings and Corrective Action (CA) Responses Table	**Not applicable**
33	QA Management Reports Table	**Not applicable**
D. Data Review		
34	Verification (Step I) Process Table	**Not applicable**
35	Validation (Steps IIa and IIb) Process Table	**Not applicable**
36	Validation (Steps IIa and IIb) Summary Table	**Not applicable**
37	Usability Assessment	**Not applicable**

SAP Worksheet #3 -- Distribution List

(UFP-QAPP Manual Section 2.3.1)

Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address	Document Control Number (Optional)
Todd Bober	Remedial Project Manager	NAVFAC Atlantic	215-897-4911	Todd.bober@navy.mil	NA
Paul Burgio	BRAC Environmental Coordinator (BEC)	BRAC PMO NE	215-897-4915	Paul.burgio@navy.mil	NA
Lisa Joy	Environmental Director	NAS Brunswick	207-921-1717	Lisa.joy@navy.mil	NA
Mike Fagan	Installation Restoration (IR) Coordinator	NAS Brunswick	207 921-1717	Mike.Fagan1@navy.mil	NA
Amy Van Dercook	Geologist/Technical Support	NAVFAC Atlantic	757 322-4764	amy.vandercook@navy.mil	NA
Mike Daly	EPA Remedial Project Manager	EPA Region I	617-918-1386	Daly.mike@epa.gov	NA
Claudia Sait	Maine Remedial Project Manager (RPM)	MEDEP	207-287-7713	claudia.b.sait@maine.gov	NA
Carolyn Lepage	Hydrogeologist	BACSE	207-777-1049	calepage@adelphia.net	NA
Victoria Boundy	redevelopment	Brunswick Local Redevelopment Authority	207-798-6512	victoriab@mrra.us	NA
Jeff Orient	TtNUS Project Manager (PM)	661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	412-921-8778	jeff.orient@tetratech.com	NA

Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address	Document Control Number (Optional)
Charles Race	TtNUS Lead Hydrogeologist/Main e Certified Geologist (ME CG)	Tetra Tech NUS, Inc. 55 Jonspin Road Wilmington, MA 01887	978-474-8437	charles.race@tetratech.com	NA
Tom Johnston	TtNUS CLEAN QA Manager (QAM)	Tetra Tech NUS, Inc. 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	412-921-8615	tom.johnston@tetratech.com	NA
Matt Soltis	TtNUS Health and Safety Manager (HSM)	Tetra Tech NUS, Inc. 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	412-921-8912	matt.soltis@ttnus.com	NA
Lucy Guzman	TtNUS Project Chemist	Tetra Tech NUS, Inc. 55 Jonspin Road Wilmington, MA 01887	978-474-8416	lucy.guzman@tetratech.com	NA
Chelsea Fellows-Swenson	TtNUS Field Operations Leader (FOL)	Tetra Tech NUS, Inc. 55 Jonspin Road Wilmington, MA 01887	978-474-8411	Chelsea.Fellows-Swenson@tetratech.com	NA

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

(UFP-QAPP Manual Section 2.3.2)

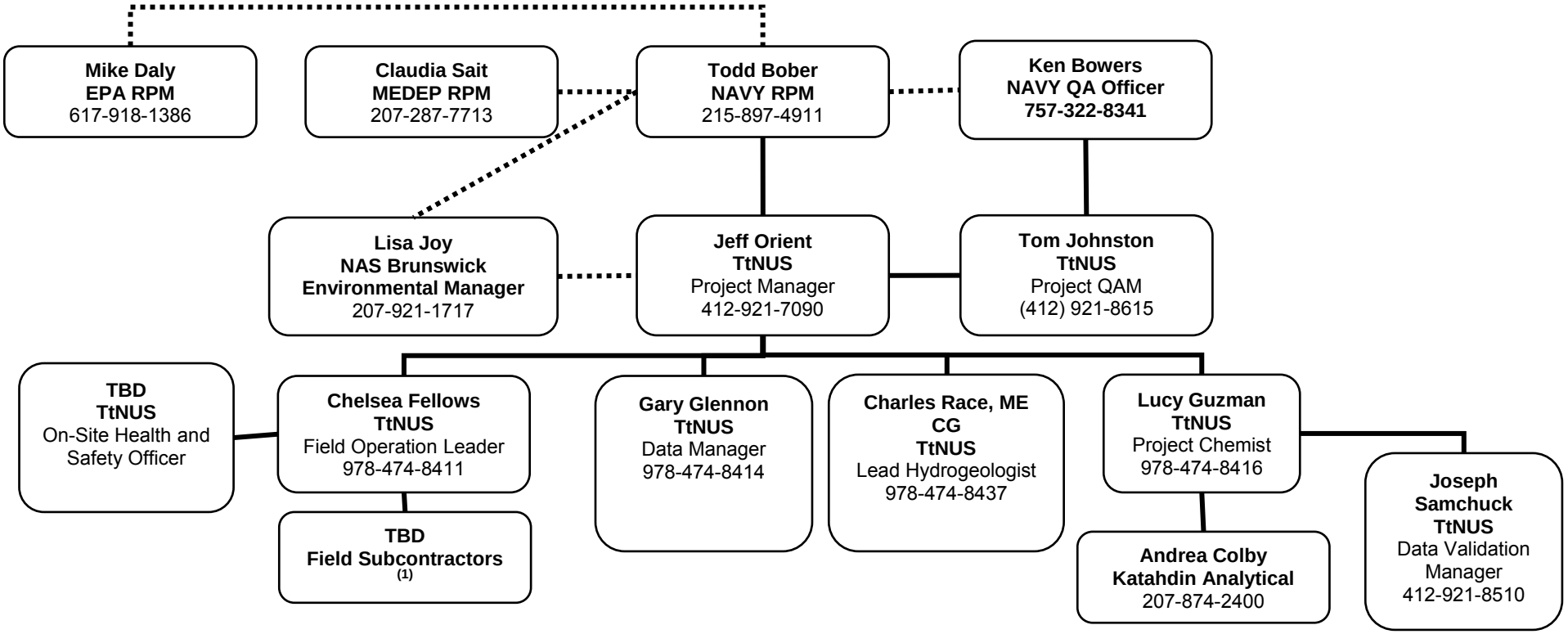
Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Jeff Orient	TtNUS PM	412-921-8778			
Charles Race	TtNUS Lead Hydrogeologist, hydrogeology oversight and review	978-474-8437			
Tom Johnston	TtNUS, CLEAN QAM, data quality review oversight	412-921-8615			
Lucy Guzman	TtNUS Project Chemist, laboratory procurement oversight, data quality review, and chemistry support	978-474-8416			
Matt Soltis	TtNUS HSM, health and safety management	412-921-8912			
Andrea Colby	Katahdin Analytical Services, PM	207-874-2400			
Chelsea Fellows-Swenson	TtNUS FOL	978-474-8411			
TBD	TtNUS, Site Safety Officer	TBD			
Joseph Samchuck	TtNUS Data Validation Manager (DVM)	412-921-8510			
TBD	Field Contractors (1)	TBD			

(1) Field subcontractors will be determined before finalization of the SAP.

SAP Worksheet #5 -- Project Organizational Chart

(UFP-QAPP Manual Section 2.4.1)

Lines of Authority ▬▬▬▬▬ ┅┅┅┅┅ Lines of Communication



(1) Field subcontractors will be determined after finalization of the SAP.

SAP Worksheet #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.)
QAPP amendments	Navy RPM	Todd Bober	215-897-4911	Send scope change to TtNUS Program office
Changes in schedule	TtNUS PM	Jeff Orient	412-921-8778	Inform Navy via schedule impact letter as soon as impact is realized
Issues in the field that result in changes in scope of field work	TtNUS FOL TtNUS PM TtNUS Lead Hydrogeologist	Chelsea Fellows-Swenson Jeff Orient Charles Race	978-474-8411 412-921-8778 978-474-8437	FOL inform PM/ME CG; PM inform RPM; RPM issue scope change if warranted; Scope change to be implemented before work is executed. Document changes on field task modification request (FTMR) form.
Recommendations to stop work and initiate work upon CA	TtNUS FOL TtNUS PM TtNUS QAM TtNUS Site Safety Officer Navy RPM	Chelsea Fellows-Swenson Jeff Orient Tom Johnston TBD Todd Bober	978-474-8411 412-921-8778 412-921-8615 TBD 215-897-4911	Responsible Party immediately informs subcontractors, the Navy, and project team
Analytical data quality issues	Katahdin Analytical Services TtNUS Project Chemist	Andrea Colby Lucy Guzman	207-8743-2400 978-474-8416	Immediately notify TtNUS Project Chemist Notify Data Validation Staff and TtNUS PM if necessary

FTMR = Field Task Modification Form

SAP Worksheet #7 -- Personnel Responsibilities and Qualifications Table

(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
Jeff Orient	Project Manager	TtNUS	Oversee project, financial, schedule, and technical day to day management of the project.	M.S. Geology, B.S. Earth Science, 29 years environmental experience, P.G. in Pennsylvania, North Carolina, Tennessee, and Kentucky.
Charles Race	Lead Hydrogeologist/ME CG	TtNUS	Oversee geology/hydrogeology related tasks and review report.	M.S. Geology, B.A. Geological Sciences, ME CG, N.H. Certified Geologist, Mass. Licensed Site Professional, 26 years geology experience
Chelsea Fellows-Swenson	FOL	TtNUS	Supervise, coordinate, and perform field sampling activities. Prepare all geological interpretation and text.	Information can be provided at a later date
Tom Johnston	QAM	TtNUS	Ensure quality aspects of the CLEAN program.	B.S. Chemistry, PhD Analytical Chemistry, 30 years environmental experience
Lucy Guzman	Project Chemist	TtNUS	Participate in scoping, prepare lab scope, and coordinate with lab. Oversee data quality review and quality assurance of data validation deliverables.	M.S. Chemistry, more than 25 years experience in environmental analysis and quality control.
Kelly Carper	QA Officer	TtNUS	Manages day to day quality assurance activites and interfaces with QAM on matters of quality.	Can be provided upon request
TBD	Site Safety Officer	TtNUS	Oversee site safety during field work	Information can be provided at a later date
TBD	Field crew members, database specialists, and Geographic Information Systems (GIS) specialists	TtNUS	Collect, package, and ship samples in accordance with work plan.	Sufficient training to be able to implement TtNUS work plans and Standard Operating Procedures (SOPs) to meet project objectives.

SAP Worksheet #8 -- Special Personnel Training Requirements Table

(UFP-QAPP Manual Section 2.4.4)

All field personnel will have appropriate training to conduct the field activities to which they are assigned. Additionally, each site worker 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). Safety requirements are addressed in greater detail in the site-specific Health and Safety Plan (HASP), prepared under separate cover.

The selected analytical laboratory (Katahdin Analytical Services, Inc.) has successfully completed the laboratory evaluation process required as part of the Naval Facilities Engineering Service Center (NFESC) QA Program and described in “Department of Defense Quality Systems Manual (DOD QSM)” January 2006. The NFESC approval letter for Katahdin Analytical Services, Inc. is included in Appedix C.

SAP Worksheet #9 -- Project Scoping Session Participants Sheet

(UFP-QAPP Manual Section 2.5.1)

Project Name:	**NAS Brunswick**	Site Name:	**Background Study**
Projected Date(s) of Sampling:	**TBD**	Site Location:	**Brunswick, Maine**
Project Manager:	**Arnold Ostrofsky, (subsequently replaced by Jeff Orient)**		
Date of Session:	**October 16 -17, 2007**		
Scoping Session Purpose:	**Data Quality Objectives (DQO) meeting**		

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Claudia Sait	RPM	MEDEP	207-287-7713	claudia.b.sait@maine.gov	Project management
Eric Frohmberg	Risk	MECDC			Risk support
Chris Evans	Project Hydrogeologist	MEDEP	207-287-2651	gordon.c.evans@maine.gov	Tech support-geology
Amy Van Dercook	Geologist	NAVFAC	--	amy.vandercook@navy.mil	Tech support
Carolyn Lepage	Hydrogeologist	BACSE	--	calepage@adelphia.net	BACSE Tech Advisor
Carol Warren	Vice Chair	Brunswick Local Redevelopment Authority	--	carol@wacubu.com	Redevelopment Advisor
David Chipman	--	Town of Harpswell	--		
Lisa Joy	Env. Director	NAS Brunswick	207-921-1717	lisa.joy@navy.mil	Management
Dawn Kincaid	Env. Coordinator	BRAC PMO NE	215-897-4915	dawn.kincaid@navy.mil	BRAC Env. Coord.
Lonnie Monaco	RPM/Env. Engr.	NAVFAC	215-897-4911	orlando.monaco@navy.mil	RPM
Cornell Rosiu	Ecological	U.S. EPA			Ecological Risk
Christine Williams	RPM	U.S. EPA	617-918-1386	daly.mike@epa.gov	RPM
Mike Daly	RPM	U.S. EPA			RPM
Dale Mosher	Environmental Support	NAS Brunswick	207-921-1719	Dale.mosher@navy.mil	Tech support
Deb Stahler	Chemist	MEDEP	207-287-7878	--	Chemist
Lucy Guzman	Chemist	TtNUS	978-474-8416	lucy.guzman@tetratech.com	Chemist
Arnold Ostrofsky	Project Manager	TtNUS	978-474-8431	arnold.ostrofsky@tetratech.com	PM
Gina Calderone	Hydrogeologist	ECC	845 687-0427	gcalderone@ecc.net	Hydrogeologist
Jackson Kiker	Chemist	ECC	508 229-2270	jkiker@ecc.net	Chemist

Project Name: **NAS Brunswick** Projected Date(s) of Sampling: **TBD** Project Manager: **Arnold Ostrofsky, (subsequently replaced by Jeff Orient)**			Site Name: **Background Study** Site Location: **Brunswick, Maine**		
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Comments/Decisions: Included in DQOs, Worksheets 10 and 11.

Action Items: Included in DQOs, Worksheets 10 and 11.

Consensus Decisions: Included in DQOs, Worksheets 10 and 11.

SAP Worksheet #10 -- Problem Definition

(UFP-QAPP Manual Section 2.5.2)

10.1 BACKGROUND STUDY GOALS AND OBJECTIVES

This background study at NAS Brunswick is being performed to gather data on basewide background conditions in soil, groundwater, seeps, surface water, and sediment for current and future application to environmental site investigations and cleanups funded under the Navy's Environmental Restoration program and the Defense BRAC Act of 1990, Public Law 101-510 as amended.

In 2005, NAS Brunswick was designated for closure under the BRAC. Operational closure is scheduled for September 2011. BRAC requires that the Navy must meet the notification and covenant requirements of CERCLA Section 120(h), regarding transfer of property upon which storage or release of CERCLA hazardous substances and petroleum products has occurred, and if releases have occurred, the Navy must document that all remedial actions required to protect human health and the environment have been taken. CERCLA Section 104(3)(A) requires that the government not provide for removal or remedial action for naturally occurring substances.

There are currently 15 active environmental sites on the base and several items identified through the BRAC environmental condition of property (ECP) study that will be investigated and could potentially become sites. The Navy is reevaluating conditions at some of the sites and is seeking to accelerate assessment and cleanup of others. In accordance with its policy on the use of background chemical levels, the Navy needs to distinguish between evidence of release or disposal of hazardous substances in excess of reportable quantities caused by the Navy (a site release) and basewide background chemical levels (naturally occurring and anthropogenic) [Chief of Naval Operations (CNO), 2004]. The Navy policy requires basewide background levels in non-site areas to be established to ensure that cleanup levels for sites (spills, releases, disposal areas) are not less than background concentrations (both naturally occurring and anthropogenic).

The intent of the study is to establish robust and statistically based background constituent data sets for soils from predominant soil types at NAS Brunswick, groundwater from identified hydrologic units, and surface water, sediment, and seeps from non-site areas for comparisons of site data to basewide background data. The scope of this background study does not include the actual comparison of site data to background. The data collected for this project will be used in other projects to help identify site-related contamination, identify contaminants of concern (COCs), and develop cleanup goals that are not below background levels.

This SAP details the overall approach and the activities planned for obtaining consensus on sample locations, field data collection, and the evaluation of background sample data. So that future comparisons of site data to background are valid, the background database and descriptive statistics may have to be segregated into subsets according to soil type or hydrological regime if chemical concentrations are found to vary with these characteristics. Background data sets must support statistical analyses of several types in accordance with Navy guidance for background comparisons (Navy, 2002, 2003, 2004).

As referenced in Navy guidance (Navy, 2004), for mobile media (i.e., groundwater, surface water, and sediment), the term "background conditions" is used in this document to represent local conditions, usually upgradient or upstream, side gradient, or well removed from a site, that are unimpacted by either the site or any other point source contaminant release. In contrast, the term "baseline conditions" refers to local conditions that are not impacted by the site, but which could possibly be impacted by a source not related to the site. The goals of the background study are to define base-wide "background conditions" as opposed to "baseline conditions" at particular NAS Brunswick sites, since the latter is a problem that requires considering lines of evidence that are relevant only to the specific site in question.

10.2 NAS BRUNSWICK SITE HISTORY, LOCATION, AND DESCRIPTION

The following short summary of NAS Brunswick base and site history, description, and location is provided to facilitate understanding and interpretation of the areas proposed for background sampling at NAS Brunswick and the relationship to particular sites of interest. NAS Brunswick is an active base, owned and operated by the Federal government through the Department of the Navy, and is located in Brunswick, Cumberland County, Maine, south of the Androscoggin River (Figure 10-1). The base supports the Navy's antisubmarine warfare operations in the Atlantic Ocean with several squadrons of P-3 maritime patrol aircraft.

The base was commissioned in 1943 as a base for inshore and offshore patrol squadrons. It expanded through the war years, but was decommissioned in the fall of 1946. Between 1947 and 1951 it was leased to non-military tenants including the University of Maine and Bowdoin College and a civilian flying service and school. The base was recommissioned in 1951 as an antisubmarine NAS. In the 1950s, there was significant expansion including construction of the 8000 ft duel runways and 222 structures and buildings. The NAS has continued as an active airbase through the present.

The base occupies approximately 3,094 acres of land including the Main Station (also referred to as "the Base"), and several remote areas including McKeen Street Housing, Topsham Annex, the Former East

Brunswick Radio Transmitter Site (Figure 10-1), and two small rake stations (not shown). The operational area of the Main Station lies east of the two parallel runways and consists of numerous office buildings, barracks, recreational facilities, hangars, repair shops, and other facilities to support NAS Brunswick aircraft. Forested areas, grasslands, shrub land, marsh, and open water comprise approximately 83 percent of the base with the remaining 17 percent of the base consisting of an operations area and paved areas (primary flight ramps and runways). The southern edge of the Base borders Harpswell and Buttermilk Coves, estuaries of the Gulf of Maine.

Undisturbed topography at NAS Brunswick is characterized by low undulating hills with deeply incised brooks. Ground surface elevations range from mean sea level (msl) in lowland drainage areas and the Harpswell Cove estuary to more than 110 ft above msl west and southwest of the southern end of the runways. Topography in the developed areas of the base has been modified by construction, with ground surface elevations generally ranging from 50 to 75 ft above msl.

Property uses surrounding NAS Brunswick are primarily suburban and rural residential, with some commercial and light industrial uses along bordering highways. An elementary school and a college are located within a 1-mi radius of the western base boundary.

Groundwater on the Base is not used for potable purposes. The industrial and residential portions of the Base are connected to a public water supply administered by the Brunswick-Topsham Water District, but the remainder of the Base, that has not been developed does not have access to public water. Several environmental sites exist on Base and are shown in Figure 10-2.

10.3 ENVIRONMENTAL INVESTIGATIONS

The Navy prepared an Initial Assessment Study (IAS) in 1983 to identify and assess potential sites due to contamination from past hazardous materials operations [Naval Engineering & Environmental Support Activity (NEESA), 1983]. In 1987, NAS Brunswick was placed on the National Priorities List (NPL) by the U.S. EPA. In 1990, the Navy issued a Remedial Investigation (RI) report for multiple sites, and entered into a Federal Facilities Agreement (FFA) with EPA and MEDEP. The Navy has been addressing various environmental sites under the Installation Restoration Program (IRP) and in accordance with MEDEP petroleum regulations. In addition, the Navy is currently conducting Site Inspections at several sites under the DoD's Military Munitions Response Program (MRP).

The environmental sites are shown on Figure 10-2. The Navy identified 18 environmental sites that it has addressed under the IRP.

Sites 1 & 3, 2, 4, 7, 9, 11 and 13 were characterized in the RI (EC Jordan, 1990). Sites 5, 6, 8, 9, 11, 12, and 14, were further characterized in the Supplemental RI (EC Jordan, 1991). Sites 5, 6, and 8, were closed with No Further Action (NFA) Record of Decision (ROD). Site 10, off base near Harpswell Cove (not shown in figure 10-2), was addressed as a petroleum site by the Defense Logistics Agency and the Defense Energy Support Center. Sites 14, 15, 16, and 18 were closed with a Consensus Statement dated July 18, 2001 recommending no further action. Samples from environmental media at the following active sites are being collected for laboratory analyses in support of long-term monitoring (LTM), site delineation and characterization, and remedial/removal actions, and the background study will support data analysis for these projects:

<u>IRP Sites 1 & 3, 2, 7, 9, 17; and the Eastern Plume (Sites 4, 11, and 13)</u>

- Site 1, Orion Street Landfill – North and Site 3, Hazardous Waste Burial Area, are two closed hazardous waste landfills. The selected remedy included a slurry wall around the landfill, a low permeability cap, and extraction wells which are currently off-line. LTM is ongoing.

- Site 2, Orion Street Landfill – South, is an inactive 2-acre landfill that was closed with a soil cover in 1955. The selected remedy in the 1998 ROD was institutional controls, removal of surface debris and monitoring. LTM is ongoing. Concerns remain that metal concentrations in leachate seep samples along Mere Brook could be elevated. Additional data will be collected under other projects to further delineate the nature and extent of landfilling activity and contamination.

- Site 7, the Old Acid Caustic Pit site, was used for liquid waste disposal in the 1950s and 1960s. Cadmium and manganese were COCs in groundwater. Elevated concentrations of cadmium, polycyclic aromatic hydrocarbons (PAHs), and pesticides were detected in the soil. After a soil removal action to remove the source(s) of contamination, LTM for groundwater (ECC August 2008) indicated that both cadmium and manganese exceed current drinking water criteria. New data collection for source delineation and site characterization is planned under another project.

- The Eastern Plume is the groundwater contamination attributed to past solvent disposal practices at three sites, Site 4 (Acid Caustic Pit), Site 11 (Fire Training Area), and Site 13 (Defense Reuse and Marketing Office -DRMO). The dissolved-phase plume was found to consist primarily of chlorinated Volatile Organic Compounds (VOCs), including tetrachloroethene, 1,1,1-trichloroethane, and trichloroethene. Investigations and LTM are ongoing under other projects.

- Site 9, Neptune Drive Disposal Site, is a 20 acre area that contained an incinerator used in the 1940s, an ash landfill/dump area, and disposal area. Excavation of the landfill began in 2006 and is ongoing. Further delineation of the site is planned under another project.

- Site 17 was associated with storage, mixing, and disposal of pesticides and herbicides. Herbicides and pesticides were detected in the soil and on the structures, and low concentrations of pesticides and inorganics were reported in groundwater samples. The Navy conducted building demolition and soil removal in the early 1990s. In 1994, some contaminated soil was excavated and then placed in a 6-inch layer within a rectangular area, or "soil strip," of previously excavated material and covered with 2 feet of fill. The Navy will collect additional data under another project to further characterize the nature and extent of contamination and perform human health and ecological risk assessments.

<u>Two active oil, petroleum, lubricant (POL) sites:</u>

- The Navy Exchange [NEX) Service Station is currently being addressed through a removal action.
- At the Old Navy Fuel Farm, the Navy conducts LTM.

<u>Six active military munitions sites for which a Site Inspection is underway</u>:

- At Site 12, the Explosive Ordnance Detonation Training Area, disposal and open burning/open detonation of small quantities of ordnance, pyrotechnics, privately manufactured explosive devices occurred. Munitions and explosives of concern (MEC) and munitions constituents (MC) are potentially present. Originally investigated under the IRP.

- The former Machine Gun Bore Sight Range was active in the 1950s, and machine gun ammunition including .30-caliber and .50-caliber was used. MEC is not suspected and the primary MC constituent is lead.

- At the former Skeet Range, the primary MC constituents are PAHS from clay targets, lead and potentially antimony, arsenic and nickel and black powder. Lead shot from expended shot gun ammunition is not considered MEC so the site is not suspected to contain MEC.

- The former Munitions Bunker West was used for munitions training by Marines. MEC and MC are potentially present in surface soil.

- The Quarry was allegedly used as a munitions disposal site. MEC and MC are potentially present.

- The Topsham Annex Skeet Range (not shown in Figure 10-2) has concerns similar to those identified for the former Skeet Range.

The Navy will also perform some screening level investigations to support property transfer determinations in non-site areas of NAS Brunswick. The Navy prepared an ECP Report for NAS Brunswick (NAVFAC-Midlant, 2006), which summarized the environmental condition of the NAS Brunswick property and a Community Environmental Response Facilitation Act (CERFA) Identification of Uncontaminated Property report (NAVFAC Mid-Atlantic, 2007) to identify real property on which no hazardous substances or petroleum products or their derivatives have been released or disposed. Navy believes some areas warrant further investigation to update the ECP. These areas were identified through further Navy evaluations of historical practice, regulator comments on the ECP and CERFA reports, and citizen input and will be designated Phase II ECP Items. Any analytical data collected from media at "ECP Items" will be screened against human health and ecological benchmarks and background values for NAS Brunswick, to determine whether these items could be CERCLA sites.

10.4 CONCEPTUAL SITE MODEL (CSM) – GENERAL SUMMARY

In the absence of NAS Brunswick, concentrations of various chemicals such as metals and PAHs would exist in the environment at detectable concentrations. Some of these chemicals (e.g., metals) occur naturally in minerals in rocks, soil, sediment, surface water and groundwater and would be present in the absence of all human activities. Other chemicals (e.g., PAHs) are deposited through non-human activities such as forest fires caused by lightning. Others may be deposited through airborne emissions (from cars, buses, planes, local industries); activities such as use of pesticides in accordance with manufacturers instructions; runoff from pavements or parking lots; deposition of road salts, etc. The concentrations of these anthropogenic and naturally occurring chemicals are classified as "background" concentrations (CNO, 2004). However, activities resulting in release or disposal of hazardous substances at concentrations above reportable quantities or petroleum products may add to the background concentrations of some chemicals. The increased concentrations potentially increase the health risks to humans and ecological receptors exposed to the media. These areas where spills, releases, or disposal of hazardous substances have occurred due to Navy activities are the environmental sites that are under investigation on the Base.

It is Navy policy that remedial activites will not be based on non-site-related chemicals (whether naturally occurring or anthropogenic) and therefore, this investigation is needed to determine basewide

background concentrations of chemicals. In keeping with CNO (2004), background for this study is defined as all background chemical concentrations, regardless of their origin, that are present naturally in the environment plus chemicals that are present in the environment due to man-made non-site-related contaminant sources (i.e. anthropogenic sources unrelated to the site that are not point sources having a localized and isolated range of influence. Included in the category would be chemicals such as pesticides and herbicides that are used in a legal manner during application to control pests and vegetation. Some of the data collected in this study may be useful to distinguish subgroups of anthropogenic background; however, the study was not designed to achieve that goal.

The Navy has considered three classes of chemicals that are typically found in soils because they are background sources: metals, PAHs, and pesticides/herbicides. These chemical constituents may be present as background conditions or they also may be contaminants of concern at sites where release or disposal of hazardous substances containing these chemicals has occurred. The data from the background study will support efforts to distinguish between the two.

There may be different chemical concentrations in surface, versus subsurface soil because surface soil may receive runoff from roads, atmospheric deposition, or legal pesticide application. For example, where orchards are present or have been present, arsenic concentrations may be greater in surface soil than in subsurface soil because of historical pesticide use. Therefore, two data sets, one for surface soil and one for subsurface soil will emerge.

The chemical groups to be analyzed for each medium are developed in Worksheet #18.

Characteristics that affect the concentration of chemicals in background areas are described below for individual matrices.

10.4.1 CSM – Soils

The chemical content of soils can vary widely based on soil type alone. Factors that could determine chemical differences from one soil type to another include the geologic parent material, the surficial soil type, the depositional environment and whether the soil is fill.

Soil categorized as fill will be avoided for background sampling wherever non-native geologic materials are indicated. However, due to the sandy nature of surface soils at NAS Brunswick, it is very difficult and nearly impossible in many cases to reliably distinguish between native sandy soil versus sandy soil that has been regraded and technically considered disturbed soil.

Plausible risk exposure scenarios drive the depths of soil that are investigated. Direct contact soil exposure to either human or ecological receptors occurs within the shallowest 6 inches to 1 foot below ground surface (bgs), so the depth range of 0 to 1 foot bgs is considered a distinct soil sampling category. Future construction activities, road development, utility work, or landscaping might cause soils presently located at depths between one foot and 10 feet bgs to be disturbed and redistributed at the ground surface, so this 1 to 10-feet depth interval will also be sampled as part of the background study. Contamination deeper than 10 feet is typically only a concern from a fate and transport perspective, e.g., contamination leaching from subsurface soils to groundwater.

There are four targeted background soil units that have been described in previous reports (EC Jordan 1990, 1991) and can be correlated with the surficial units identified by the Maine Geologic Survey (Maine Office of Geographic Information Systems, Maine Geological Survey, 2006). The mapped surficial units are shown on Figure 10-2, and the targeted soil types are described as follow:

- Upper Sand - Glaciomarine Deposits, Coarse Grained Facies - Sand, gravel, and minor amounts of silt. Also described locally on base as tan to light brown fine sand, trace to little medium sand and silt.

- Transitional Unit and Lower Sands - Glaciomarine Deposits, Fine Grained Facies - Silt, clay, and minor amounts of gravel. Commonly, a clayey silt (the Presumpscot Formation). Sand is dominant in some places, but may be underlain by finer grained sediments. Locally fossiliferous. The map unit includes small areas of till and other units that are not completely covered by marine sediments. Described locally on base as predominantly gray very fine to medium sand interbedded with silt, silty clay, clay layers. Lower Sands are described as gray-brown very fine to coarse sand.

- Presumpscot Clay - Glaciomarine Deposits, Fine Grained Facies - Silt, clay, and minor amounts of gravel. Commonly, a clayey silt (the Presumpscot Formation). Locally described as gray silty clay to clayey silt (rock flour), medium to high plasticity, very soft.

- Till - Heterogeneous mixture of sand, silt, clay, and stones. May include many boulders. Generally massive, but in many places contains beds and lenses of variably washed and stratified sediments.

A typical cross section from the Eastern Plume study is shown on Figure 10-3. This is the one of the areas that has been studied most intensively. It is likely that only Upper Sand, and Transitional Unit, will be encountered within the upper 10 ft bgs at most locations. The Lower Sand and Till will not be encountered in the upper 10 ft bgs at most, if not all locations; therefore, these two geologic units are

excluded from the Background Study SAP. This leaves two geologic units (Upper Sand and Transition Unit) to be sampled for soil.

The RI at NAS Brunswick evaluated two soil types for its study of inorganics in background: the sand unit and the clay unit (EC Jordan, 1990). The sand unit was described as predominantly fine to medium quartz sands with trace silt (Upper Sand). The clay of the Presumpscot Formation was described as a mixture of clay minerals, fine sand, silt and clay-sized particles derived from glacially abraded bedrock. The TAL metals concentrations in the sand were typically low, with aluminum, iron and manganese predominating and lower levels of chromium and zinc. The clays, in contrast contained more metals at higher concentrations including aluminum, iron, magnesium, and manganese, and a larger array of trace minerals including chromium, lead, zinc and arsenic. The RI noted that the occurrence of the metals was consistent with rock flour derived from bedrock containing pyrite, iron-titanium oxides ilmenite and hematite, and mica and clay minerals illite and vermiculite (EC Jordan, 1990).

The Navy anticipates that there will be similar differences in metals concentrations in the targeted soil units but until the data are analyzed, it is not known whether the four target soil units will emerge as statistically distinguishable subsets.

10.4.2 CSM – Groundwater and Seeps

Groundwater is water that exists within soils and bedrock as well as seeps. For this project, seeps are defined as expressions of groundwater at elevations greater than nearby steams. It was not known whether background seeps (i.e., seeps unaffected by base operations) can be found. A satisfactory background seep would flow through the same geologic parent material as a site seep (same depositional environment and possibly same bedrock) but would not be impacted by site-related activities. Vertical gradients, soil units, bedrock units will be considered to assess where seep water originates but evaluations will be made on a seep-by-seep basis.

A background area walkover was performed by the Navy, EPA, MEDEP, MRRA and BCSE on April 28-29, 2009. Area 2 was determined acceptable for surface water, sediment, and seeps. However, debris were present in the vicinity of most seeps, resulting in only one or two seeps available. Therefore, the Navy and regulators agreed to exclude seep sampling from the Background Study.

The representative hydrogeologic units for groundwater are the Upper Sand, Transition Unit, Lower Sand, and bedrock. A typical cross section from the Eastern Plume area is shown on Figure 10-3. The Upper Sand unit has the greatest horizontal hydraulic conductivity (K) measurments (in the 10^{-3} cm/sec range).

The Transition Unit, with its stratified, sand, silt, and clay had measured K values one to two orders of magnitude lower (10^{-4} to 10^{-5} cm/sec). The Lower Sand also had K values in the 10^{-3} cm/sec range and have been found to be a preferential pathway for groundwater and contaminant movement. Laboratory K values for the clay unit obtained from Sites 1 and 8 ranged from 10^{-7} to 10^{-8} cm/sec (EC Jordan 1990). The K for till was not evaluated as part of the RI, but till typically has K values that range from 10^{-4} to 10^{-11} cm/sec (Freeze and Cherry, 1979). Both the clay and the till have low hydraulic conductivities and are not anticipated to be significant for contaminant transport and are not included in the background study. Bedrock in the most of the base is a metamorphic highly foliated mica (biotite) schist that in boring logs is described as slightly to moderately weathered and not fractured. Groundwater flow is through fractures and joints in the bedrock, and the horizontal and vertical distribution of groundwater in these water-bearing zones is believed to be highly variable. Upper portions of the bedrock have higher permeability than deeper zones (E.C. Jordan 1990). According to the Bedrock Map for Bath 1:100000 Quadrangle (Maine Geological Survey, 2002), the Cape Elizabeth Formation underlies overburden in the majority of the developed portion of the Base to the eastern Base boundary, and is in contact with the Cushing Formation beneath the western portion of the Base.

An overview of basewide groundwater movement is shown on Figure 10-4 (EC Jordan 1990). Flow patterns in the upper unconfined units have been assessed based on water level data from wells installed at the IR sites, interpolated from known points, and supported with surveyed surface water elevations in the streams. Figure 10-5 shows more recent interpretation of the water table surface in the eastern portion of the base, where most of the monitoring wells have been installed. Figure 10-6 shows interpreted flow in the Lower Sand unit. From these interpretations, areas that are upgradient from or geographically distant from the known environmental sites, and distant from the current or past industrial areas, the runways, taxiways, aprons, and operations areas have been identified. As further described in Section 11, new monitoring wells will be installed and screened discretely in each of the three identified hydrogeologic units. Currently, it is anticipated that four offbase existing wells in bedrock will be sampled. The four off-Base existing bedrock wells are completed in the Cape Elizabeth Formation, which covers most of the Base. The Cushing Formation is mapped generally in the western one-third of the Base; the Quarry is the only known site underlain by the Cushing Formation. It is unknown whether groundwater from these units is distinctly different. Therefore, if bedrock groundwater at the Quarry or at any other identified site is underlain by the Cushing Formation, then upgradient bedrock wells will be considered to evaluate background concentrations.

Data from each of these four units (Upper Sand, Transition Unit, Lower Sand, Bedrock) will initially represent a data subset for background groundwater. If the concentrations of analytes are found to be statistically similar between these hydrologic units, then the background data sets will be consolidated to

achieve a larger data set for future use in comparing to site data. Details are provided in subsequent discussions.

10.4.3 CSM – Sediments

For this project, sediment is defined as any solid geological material that is submerged in less than 1 foot of water for all or virtually all of the year. Background sediment samples will be collected from the same types of locations (i.e., depositional areas) as sediment samples collected during environmental investigations and will include a range of sediment types. For human health risk assessment only the top 6" of sediment represents an exposure concern. The rationale is that if the water is deeper than this, the bottom sediments will be washed off skin shortly after contact, virtually eliminating any significant or prolonged exposure. Ecological risk estimates are typically based on data from the top 6 inches. The study will also evaluate whether the concentrations of analytes may be affected by the amount of organic matter or soil classification as silt, clay, and sand. If the chemical distribution of substances is found to be dependent on these attributes, then site sediments should be compared to background sediments having a similar composition.

Contaminants may be carried into sediments directly by precipitation or from overland runoff or groundwater discharge. Sediment flows within drainage channels to lower elevations and carries suspended and adsorbed contaminants along the way. Background sediment conditions are represented by sediments in areas that have not been impacted by site-related activities at spills, releases, or disposal areas (sites) on base. Depositional areas within streams and ponds/lakes accumulate sediments and provide the best opportunity for obtaining representative sediments, but should have similar composition to site sediment (i.e., percent fines, percent organic carbon).

10.4.4 CSM – Surface Water

Contaminants may be carried into the surface water directly by precipitation or from overland runoff or groundwater discharge. Surface water flows within drainage channels to lower elevations and carries or picks up suspended and dissolved contaminants along the way. The streams and water bodies are shown in Figure 10-1. General drainage divides on the base are shown on Figure 10-7 (EC Jordan, 1990). Background conditions are represented by surface water in areas that have not been contaminated by site-related activities. Areas upstream of sites, and areas that do not receive runoff or groundwater discharge from contaminated sites on base are needed. Surface water and sediment locations should be co-located and sampled at the same time to help interpret whether observed surface water concentrations are currently contributing to depositional processes in sediments.

10.4.5 Statistical Model

Background data will eventually be compared to site data. The background data sets may need to be segregated by subtype – for example, soil types, groundwater hydrologic units, seeps, and sediment type. If analyte concentrations are not significantly different among subtypes, the various matrix subtypes may be combined to create a larger background data set for a particular matrix. Future data set comparisons between sites and background will be based on the concept of using one or more statistical methods to distinguish a site data set from a corresponding background data set. Navy and EPA guidance were followed to develop a strategy for comparing site to background for all media (EPA, 1989, 1992, 2002, Navy, 2002, 2003, 2004).

In general, background data can be statistically compared to site-specific data in several different ways. Consistent with Navy guidance (Navy, 2004) and EPA guidance (EPA, 1989, 1992), statistical tests called "site-wide comparisons" (Navy, 2004) can be used to compare the aggregate data from site samples to the pooled background data. Site-wide comparisons evaluate whether the site concentrations, considered as a whole, exceed the levels that would be expected in the background population. Site-wide tests can be applied using the Hypothesis Test Form II (see explanation in Section 11) that was recommended by stakeholders for NAS Brunswick.

Full TAL metals analysis rather than analysis of a more limited list of metals allows a flexibility of approach for a particular site, with the option of performing either site-wide comparison tests with two sample hypothesis using Test Form II or a geochemical evaluation of the correlation of specific minerals to naturally occurring analytes when comparing sites to background. Therefore, TAL metals are of interest for this investigation.

If it is not possible to perform site-wide comparison tests for various reasons discussed in Section 11, then the alternative is to perform a separate comparison of each site sample to background using individual comparison tests (Navy, 2004). Individual comparison tests are also referred to as background threshold values (BTVs) (EPA, 2004) or upper prediction limits (UPLs) (EPA, 1989, 1992). Concentrations from one site sample are compared to a threshold level that is statistically derived so as to include a certain proportion of the background population (for example, 95 percent). However, Navy recommends UPL comparisons should not be the only basis for making a final decision on background.

When performing site-wide comparisons, it is not possible to detect an infinitesimally small difference between site versus background, so a minimum detectable difference (MDD) must be established. This difference was set at two standard deviations of the background chemical concentration. Therefore,

enough data must be collected for each background data set to facilitate the detection of a site data set that has a mean concentration that is greater than the background mean concentration by two standard deviations. The number of data points that must be collected for the background data sets depends on the number of data points in the site data sets. This varies from site to site. Therefore an assumption was made that the background and site data sets would contain the same minimum number of data points. If the site data set has fewer data points, it might be necessary to have more data points in the background data set to be able to distinguish a two-standard deviation difference from background concentrations. If soils of ostensibly different types are found to be chemically similar, however, the data sets may be combined. The resulting larger data set will offset the limitations associated with smaller site data set. The mathematics of site-wide comparisons requires the computation of a pooled variance for the two data sets. Formulas describing the relationship between the required controls over Type I and Type II errors, the number of samples belonging to the site and background data sets, and the minimum detectable difference (MDD) are given for several types of statistical tests in Table 11-2.

10.5 PROBLEM SUMMARY

For soil, groundwater, surface water, and sediments conditions representing an absence of NAS Brunswick site-related contamination must be established. These concentrations, representing background conditions, will be used to help determine when chemical concentrations at environmental sites are elevated relative to background conditions. The background conditions will represent chemical concentrations that occur naturally plus contributions from background activities. The data from background areas for each environmental medium must be analyzed to establish a limited number of background values and data sets representing significantly different background conditions for the various combinations of soil type, etc., as described above. At least one background (or upgradient) data set must be established for each of the following media:

- Surface Soil - this will be divided initially into two subtypes: Upper Sand, and Transition Unit
- Subsurface Soil (this will be divided initially into two subtypes: Upper Sand, and Transition Unit)
- Sediment
- Surface water
- Groundwater (this will initially be subdivided based on major hydrogeologic units of Upper Sand, Transition Unit, Lower Sand, and bedrock)

Differences and similarities among data sets representing subtypes and major units will be established based on geology as well as chemical characteristics of the various environmental media. The MDD between site and background data will be two standard deviations of the background concentrations.

A protocol for using the background data sets is presented in this SAP. This protocol will support comparisons of background conditions to site data.

SAP Worksheet #11 -- Project Quality Objectives/Systematic Planning Process Statements

(UFP-QAPP Manual Section 2.6.1)

11.1 OVERVIEW OF THE DQO PROCESS: DEVELOPMENT OF PROJECT QUALITY OBJECTIVES (PQOS)

PQOs document the environmental decisions that need to be made and the level of data quality needed to ensure that those decisions are based on sound scientific data. PQOs are described below.

11.2 IDENTIFY THE GOALS OF THE STUDY

The principal goal of this study is to collect enough data for each environmental medium to estimate the background concentration of each chemical of interest, so that the data may be used to support the goals of future studies that attempt to discern a difference between site and background data. To achieve this, background data sets for the various subunits will be compared in an attempt to establish the minimum number of data groups required to uniquely represent particular environmental media. When sufficient background data have been collected for each medium, stop the data collection for that medium.

A secondary goal has been addressed. That goal was to establish a protocol for using the background data generated for this project. This protocol will be used to compare site data to background data to determine whether site data exceed background concentrations. The protocol for comparing site data to background data is explained in section 37.0.

Project Decision Statement for Data Collection:

If enough data have been collected to measure a two standard deviation difference between site data and background data for each of the media described in Sections 10.4.1 through 10.4.4, stop data collection. If enough data have not been collected reconvene the project team to determine whether additional data collection is necessary.

Whether additional data collection is necessary will depend on which combinations of chemicals and environmental media do not have enough data. For example, if thallium in groundwater is the only chemical for which enough data have not been collected, it is unlikely that more data will be collected because thallium is not normally detected in groundwater and mere detection could possibly be used to establish a contaminated site. On the other hand, if all metals in groundwater do not have enough data,

continued data collection would be likely. If insufficient data have been collected but it is not clear whether additional data should be collected, the project team will be reconvened to determine this.

11.3 DEFINE WHAT INFORMATION IS NEEDED FOR THE STUDY

Data that must be collected make the decision identified in Section 11.2 are:

- Groundwater elevation data from Fall 2008 field activities (even if preliminary) that may used to refine the CSM and understanding of groundwater flow directions to support placement of background wells.

- Geologic boring logs from previous investigations and those completed as part of this Background Study will be considered to refine the understanding of the location of the Lower Sand Unit.

- Background locations for monitoring wells, soil, sediment, and surface water. Field surveys were conducted by the Navy on April 8-9, 2009, and on April 28-29, 2009 by the Navy, EPA, MEDEP, MRRA, and BCSE representatives to ground-truth the planned locations for groundwater, soil, surface water, and sediment sampling, thus verifying that the locations represent true background areas unaffected by site operations. This included verification of the required background soil types identified above, and assessing whether upgradient and upstream areas may contribute to point-source contamination and thereby affect the general applicability of any sample location as representative of background. Targeted background areas are further discussed in Section 11.4, in Table 11-1 and are identified on Figure 10-2 and Figure 17-1.

- Sufficient numbers of surface soil, subsurface soil, groundwater, surface water, and sediment samples from background locations, in each of the identified soil types and hydrogeologic units.

- Soil descriptions and detailed boring logs so that the targeted soil types and hydrogeologic units can be identified and used later to match to site samples that must be compared to background data.

- Groundwater and surface water quality parameters (see Worksheet 17).

- Grain-size and Total Organic Carbon (TOC) analyses for sediment and TOC for soil.

- Chemical concentrations of each chemical in each of the environmental media identified in specific chemical lists arranged by analytical fraction can be found on Worksheet 15 and the analytical scope

is described in Worksheet 18 of this SAP. The analytical scope was modified since its inception and the analyses listed in Worksheet #18 reflect the current scheduled analyses.

- Lowest representative numerical criteria (project action levels) to which data will be compared during site investigations [e.g., or Contaminants of Potential Concern (COPC) selection or project decision making]. These criteria will be used in establishing the analytical method sensitivity requirements for this project.

- Statistical tests for evaluating the data and consolidating the data into the fewest possible unique data sets and for comparing data sets for geochemical similarities and dissimilarities.

In addition, chloride and bromide data will be collected from groundwater to assess whether elevated sodium in the groundwater is associated with relic seawater or deicing salts.

11.4 STEP 4 OF THE DQO PROCESS: DEFINE THE STUDY BOUNDARIES

There are no exposure units per se for this project, just general classes of media, areas, and depths to which various receptors might be exposed at contaminated sites. The background study boundaries for each environmental medium are restricted to those areas which meet the definition of background stated in Section 10.1 and which are on or in close proximity to NAS Brunswick and belong to the relevant media sub-classifications identified in Section 10.4.

NAS Brunswick site-related sampling locations comprise certain depth ranges that are considered possible contact points for future human exposure. To support valid comparisons of site and background data, the background data should represent similar depths and geological classifications as the soil, sediment, surface water, and groundwater at the sites but be from locations on Base that have not been known to have been impacted by sites.

The Project Team has identified seven general areas on the Main Station Base (Areas 1, 2, 3, 4, 5 6N, 6S), two off Base areas (Area 7 and Bowdoin Pines) and an area for bedrock groundwater east of the Eastern Plume (Figure 10-2). These areas have been identified because they do not appear to be impacted by site-related activities; are located either upgradient, sidegradient, or upstream of known sites and base activities (or geographically distant from sites if gradients are not known); and are likely to contain the geologic and hydrogeologic units of interest. These areas and the rationale for their selection are described in Table 11-1. Specific sampling locations within the areas will be chosen and vetted with the Project Team, through site walks for ground truthing. Factors impacting sample locations include

information from review of aerial photos, discussion of previous and current uses, topography, accessibility, location with respect to ground water or surface water divides, and location with respect to background structures (e.g. golf course, former utility pipeline, former homesteads). For the evaluation of soils and mobile media such as groundwater, sediments, and surface water, the Navy prefers on-Base sampling areas because these areas are more likely to represent the nearest upgradient locations relative to specific NAS Brunswick sites. Ground truthing verified whether these locations are representative of background areas.

The total soil populations comprise the collection of all potential samples that could be collected from the environmental media of interest. Soils are divided into surface (0 to 1 feet bgs) and subsurface (1 to 10 feet bgs) units. Soil concentrations are not expected to change rapidly hence there are no temporal constraints on sample collection. As shown in Table 11-1 and on Figure 10-2, it is anticipated that a sufficient number of samples of the two soil types (Upper Sand, Transition Unit) can be collected in areas on Base representing Base-wide background conditions, if encountered within 10 feet of ground surface.

As shown on Table 11-1 and Figure 10-2, the soil sample and temporary well locations are distributed across a wide area near the Base perimeter and in suitable areas outside the Base (Bowdoin Pines and Area 7). The Navy and regulators have agreed on the Background Areas and number of soil, groundwater, surface water and sediment samples, and sampling frequency. Further, the location of soil borings/temporary wells are contingent upon the Navy obtaining an access agreement with the property owner of Bowdoin Pines. The locations of soil borings/temporary wells in the remaining Background Areas will be determined in a similar manner as that at Bowdoin Pines (i.e., distributed evenly across each area, while maintaining a minimum setback of 200 feet from highways, golf courses and avoiding potential impacts from drainage from these areas and septic leachfields (if upgradient). A summary of soil sample locations is summarized in the Table below;

Background Area	Soil Unit	Number of Soil Sample Locations
1	Upper Sand	5
4	Transition Unit	9
5	Transition Unit	3
6N	Upper Sand	5
6S	Transition Unit	3
7	Upper Sand	2
Bowdoin Pines	Upper Sand	3
	Total	30

The number of sampling locations for groundwater are summarized in the table below:

Background Area	Hydro-stratigraphic Unit	Number of Wells
1	Upper Sand	4
3	Lower Sand (if present)	1
4	Transition Unit	8
5	Transition Unit	3
6N	Upper Sand	4
6N	Lower Sand (if present)	3
6S	Transition Unit	3
7	Upper Sand	4
Bowdoin Pines	Upper Sand	3
Existing Wells	Transition Unit	1
Existing Wells	Lower Sand	8
East of Merriconeag Stream	Bedrock	4
Area 6N	Lower Sand	3
Area Northwest of MW-303	Lower Sand	3
Total Number of Upper Sand wells		15
Total Number of Transition Unit wells		15
Total Number of Lower Sand wells		15
Total Number of Bedrock Wells		4

For the bedrock unit, the Navy proposes not to install new wells but to sample four offsite wells that have been previously sampled and found to be not contaminated. Groundwater concentrations are known to fluctuate over time and to exhibit seasonal variations. Representative groundwater was agreed by the project team to be most consistently collectible in Spring (e.g., April) after snowmelt and Fall (e.g., September) corresponding to the months when samples have been collected as part of the Long Term Monitoring Program so those periods are targeted for sampling. No additional on-Base bedrock wells are identified as acceptable for use as background wells by the project team; however, contaminants of concern in the bedrock have historically been VOCs and 1,4-dioxane and in one bedrock well (MW-308) that is located at the top of a buried bedrock hill where the fractured bedrock is in hydraulic connection with the Transition Unit or Lower Sand unit.

Sediment and surface water samples are to be co-located. The areas supporting this sampling are shown on Figure 10-2 and discussed in Table 11-2. The primary location for surface water and sediment is Area 2, where Mere Brook enters the base, and west of the bridge that crosses the Brook. The bridge washed out and was replaced in 2008, and therefore, locations upstream of the new bridge are preferred. Further, the area for background surface water and sediment sampling will be along a reach downstream from a fence where vegetation has created a pond, to the confluence of Mere Brook and a tributary. The tributary to Mere Brook is considered to not be a suitable background area because it receives drainage from an area of man-made land that could include imported fill. Based on risk evaluation methods, the top 6-inch depth interval of sediments represents the entire range of depths of interest because this interval represents exposure for human health and/or ecological risks. For surface water in Mere Brook, and Coffins Ice Pond, the targeted depth will be 6 to 8 inches below the water surface. This is viewed to be a representative depth that is not so deep that it would prevent sample collection. Storm events could alter what would be considered normal background conditions, therefore sample collection will not occur during or immediately after a storm event.

11.5 STEP 5 OF THE DQO PROCESS: FORMULATE THE STUDY'S ANALYTIC DECISION RULES

It is anticipated that the data collected from this background investigation will be used to make two types of decisions: to detect differences between site and background data sets, and to detect differences between subgroups of background data associated with different media types. The former represents an end use of the background study results and the latter is an intermediate step in performing the background study to determine what data sets should properly be consolidated for future applications to sites having certain associated soil types. These data comparison decisions are described in the following subsections.

11.5.1 Minimum Difference Between Site and Background Needed to Make a Decision

In any background study one of the most important criteria used to establish success for the project is the minimum difference that can be detected between site and background data sets for each combination of parameter being measured and the medium in which it is measured. For this study, the MDD was set at two standard deviations (SDs) (2-sigma) of the background data set. This corresponds to a significance level of 5 percent which allows for a five percent chance (or less) that a background and non-background data set barely contaminated over background levels would be indistinguishable. As the non-background data set becomes increasingly contaminated the chance of making this error rapidly decreases so the 5

percent was viewed to be a reasonable upper bound. In general, the MDD decreases with increased number of samples collected. If data from multiple targeted compartments within a particular medium can be combined, the increased number of data values improves the ability to discriminate small differences between data sets. It also lessens the chance that a real difference would go undetected. Using two-SDs as the MDD, the decision rule used to address the investigation objective is:

> If enough data points of adequate quality for each analyte for each of the environmental media have been collected to yield MDD ≤ 2-SDs of background data, and the data are verified through inspection of the sampling locations versus targeted sampling areas to represent background (or upgradient) conditions, stop collecting data; otherwise the project team representatives will consider the need to collect additional data. Considerations to be addressed when deciding to collect additional data include:
>
> 1. the MDD achievable with the data that have already been collected versus the target of 2-SDs of background data, and
> 2. the cost of collecting additional background samples *versus* collecting additional Solid Waste Management Unit (SWMU) investigation samples.
>
> The Navy, Maine DEP and EPA Region 1 representatives from the project team will be involved in the decision to collect more data or not. The tendency to collect additional data will increase with decreased number of collected target samples and with increased likelihood that the targeted medium will yield the intended number of samples. When data collection is completed, the smallest difference that can be detected between site and background data sets will be estimated for each medium.

The minimum number of samples (both site and background) required to achieve MDD ≤ 2 SDs of background data for each medium is computed in Step 6 below. A variety of statistical tests, as described below in this section will be used to evaluate and characterize the background data. The ability of a statistical test to find a difference between two data sets improves when the number of samples in either the site data set or background data set increases. Hence, increasing either site or background sample size is beneficial to the ability to perform a statistical comparison that is able to detect differences in concentrations.

Note that the number of samples needed for each type of statistical tests is dependent on the number of non-detected observations. Analysis results at concentrations less than the Method Detection Limits (MDLs) will be reported as the value of the Detection Limit (DL) with a “U” flag. The “U” flag signifies that

the sample was analyzed for the analyte and that the analyte was not detected at a concentration greater than or equal to the MDL. This is true for both organic and inorganic parameters because the laboratory will report all positive results greater than the MDL. The positive results above MDL but below the laboratory's quantitation limit will be reported as estimated values qualified with a "J" for both organic and metals analyses.

For any sample analytical result to be suitable for inclusion in the site or background data set, data quality will be defined as adequate if no anomalies are identified that compromise the usability of the data for background comparisons and if bias and precision indicators are judged to be adequate in accordance with specifications found in Worksheets 19, 22, 24, and 28. More detail on data usability assessment is provided in Worksheet #37.

11.5.2 Comparing Several Background Data Subsets: The Hypothesis and the Decision Rule

As previously discussed, background data subsets will be compared to one another to determine whether each soil subtype or groundwater hydrological regime is associated with significantly different analyte concentrations. There are two ways to formulate the decision making null hypothesis (baseline condition) for the simple case that involves comparing only two data sets:

- Assume data group A concentrations are greater than data group B and let the burden of proof be on the data to show otherwise (hypothesis test form II).

- Assume data group A concentrations are not greater than data group B and let the burden of proof be on the data show otherwise (hypothesis test form I).

If there are only two background groups to be compared, then two-way hypothesis tests using Test Form I will be used with an overall alpha of 0.05. Note that this is mathematically equivalent to conducting two separate one-way comparisons (one with the null hypothesis A<=B and the other with B<=A), each using an alpha of 0.025.

If there are more than two background groups involved, then either a parametric or nonparametric analysis of variance (ANOVA) test will be employed. A parametric ANOVA test assumes normality and homogeneity of variance for each of the subgroups compared. This assumption may not be met, particularly if there are many groups involved, so that the nonparametric Kruskal Wallis ANOVA test may be useful in many cases.

ANOVA initially determines whether there is a difference somewhere among groups but does not identify which particular group(s) are affected. Therefore, a second step is required after the initial ANOVA, using either t-tests or nonparametric post-hoc tests, to determine which groups are different. To select an alpha for post-hoc tests, it is necessary to balance the advantages of ensuring the overall chance of finding any random difference from all the post-hoc comparisons for one substance does not exceed 0.05 with the disadvantages of reducing alpha too far in connection with increasing the number of groups compared – as an illustration, an alpha of 0.005 if there are 10 separate two-group post-hoc comparisons to be performed. A small alpha can make it very difficult to prove any differences at all and might overlook substantive differences between subgroups. If site data are later compared to an aggregate background data set comprised of heterogeneous and multi-modal subpopulations, then site-wide statistical test results cannot be relied upon to yield a valid conclusion. Therefore, post-hoc comparisons will be performed with and without adjustments to the initial alpha, which will be set equal to 0.05. The outcomes of all series of post-hoc tests will be considered as lines of evidence and compared to the differences between groups visualized on box plots to make a final decision involving segregating or combining background data sets.

11.5.3 Statistical Tests to Compare Two Background Data Subsets

The means of two background data sets may be statistically compared provided that both data sets are first shown to match the shape of a normal distribution based on normal probability plots that are linear and based upon the Shapiro Wilk normality test performed at a level of significance of 0.05. If there are only two background groups to be compared, for normally distributed data, two variants of the t-test will be used. If the standard deviations of the two data sets are found to be indistinguishable based on Bartlett's test for equal variance, then a student's t-test will be used; otherwise, Satterthwaite's t-test will be used. The t-test will be employed only if all values within each data set constitute positive detects, there are at least three sampling points in each data set, and the pooled standard deviation is not zero. The t-test will not be performed using log-transformed data sets because of the potential for misinterpretation of the true underlying distribution that tends to occur with lognormal fitting (EPA, 1997).

If the background data are not normally distributed, then alternative non-parametric tests will be applied to determine whether two background data sets belong to populations with identical medians and rank distributions. The Mann-Whitney test and the analogous Gehan's test (which is used in cases involving many non-detects) require combining the two data sets, ranking results from smallest to largest, and evaluating whether the two sets have a similar distribution of data within the range of low to high ranks. The prevalence and magnitude of non-detects can bias the outcome of the Mann-Whitney U test, but

does not adversely bias the accuracy of Gehan's test, so Gehan's test will replace the Mann-Whitney U test whenever non-detects are present in either of the data sets being compared.

The quantile test or upper ranks test (EPA, 1992b, 2006a) is a type of hot spot test that will be applied to comparisons involving data sets having only two subgroups. The test combines two subgroups of background data into one set and determines whether an upper rank subset displays an unusually large proportion of data belonging to one subgroup rather than a mixture having the expected proportion of data from both groups. The null hypothesis is that the two subgroups come from the same population. If calculations show there is less than a five percent chance that k or more samples could belong to one subgroup among a subset of the r largest upper ranks of the data, then the test concludes that k samples from one subgroup have statistically elevated concentrations, which might reflect one or more hot spots in that data set.

If there are only two background groups to be compared, then in the event that none of the above quantitative comparative statistical tests yields a definite "yes" or "no" decision, a test of proportions will be used to determine if the percentage of positively detected results is greater in one background data subgroup versus the other. When only a very small portion of results are detected (less than 10 percent), this test is recommended (EPA, 2006a, 1989).

11.5.4 Statistical Tests to Compare More Than Two Background Data Subsets

If there are more than two background data groups to be compared, for normally distributed data, a parametric ANOVA test will be employed to determine if there is a difference somewhere among the means of the groups. The parametric ANOVA test is valid only if the population variances are equal (homoscedastic) and either the population distributions are normal or the sample sizes are large. If the Fisher F-statistic exceeds the critical value, then the ANOVA test concludes that at least one of the means among groups is different. To determine which of the group means are different, post-hoc comparisons will be performed using several types of tests having different degrees of conservatism in balancing the goals of controlling the individual test error rate and the overall family-wise error rate. Post-hoc tests may include t-tests (with or without the Bonferonni adjustment), Tukey's Honestly Significant Difference (HSD) test, and the Ryan-Einot-Gabriel-Welsch (REGW) test, the latter of which in many situations has more power than other techniques which control family-wise error.

When the background data groups do not meet the assumptions for a parametric ANOVA test, then the rank sums among groups will be compared using the Kruskal Wallis ANOVA test to determine if there is a

difference between groups. If differences are found, then post-hoc nonparametric comparisons will be performed with and without Bonferonni adjustments to the alpha for each test.

11.5.5 Hypothesis, Decision Rules, and Statistical Tests for Comparing Site to Background

For the evaluation of site data, either site-wide tests or individual comparison tests will be used to compare to background. Site-wide comparison tests will be performed following the approach recommended by EPA stakeholders, which formulated the hypothesis as test form II. This hypothesis form assumes the site is "dirty" by an amount at least as great as the MDD. A feasible and effective value for the MDD was adopted based on two times the background standard deviation. The same types of statistical tests (t-test, Mann-Whitney, and Gehan's Test) will be used for site-wide comparisons. In addition, a test of proportions will be used to make a decision only if the number of non-detects precludes use of the former tests.

Note that site-wide comparison tests require that the site and background media data sets being compared meet certain general assumptions and also specific assumptions for groundwater in particular. General requirements for any medium (soil, groundwater, , surface water, or sediment) are that a specific minimum number of samples be included in the site and background so that the test conclusions are confident, as outlined in Section 11.6.4. In addition, for groundwater, for the conclusions of a site-wide comparison test to be valid, all site wells included in the test should belong to a homogeneous statistical population having a common mean and variance. These assumptions will not be met if certain wells in the site data set are impacted by the site to a greater degree relative to other wells (for example, as a result of proximity to unanticipated contaminant sources or unexpected gradient directions). This may be evident if substances related to the source are more prevalent in some wells.

If site-wide comparisons cannot be used, then the alternative is to perform a separate comparison of each site sample to background using individual comparison tests (Navy, 2004). Two types of individual comparison tests will be available, either BTV comparisons or geochemical comparisons. BTVs based on a UPL will be established for each substance in each subcategory (soil type, groundwater hydrologic regime). The UPL is based on the null hypothesis that individual site concentrations are less than or equal to background (test form I). Concentrations from one site sample are compared to a threshold level that is statistically derived so as to include 95 percent of the background population. The 95% UPL will be based on the t distribution if the data follow a normal distribution; otherwise, a nonparametric 95% UPL will be estimated. Future site-to-background Individual comparisons involving UPLs are possible for all media (soil, groundwater, surface water, and sediment).

Note that Navy recommends that individual comparisons (i.e., BTVs) "only be considered as a means to suggest the need for additional investigation of whether or not a chemical is a COPC, and should never be the only test applied to determine if a chemical is a COPC" (Navy, 2004). This background study addresses only the initial establishment of BTVs using the background data sets, so if any other lines of evidence are needed to make a final decision on background, this can be addressed in the work plan as well as stakeholder discussions for a particular site.

Another type of individual comparison test, based on geochemical prediction limits, will be available as a more robust way of comparing site data to background data. It is anticipated that geochemical prediction limits will be useful and feasible for metals in soil, as samples of this medium will represent large background populations that encompass variability extending across geologic subtypes. In applying the geochemical method, the background data set will be graphed using a scatter plot of a trace metal (on the y-axis) against a reference metal such as iron or aluminum (x-axis), and a linear regression, with the associated 95% prediction envelope, will be computed.. Several scatter plots of background soil data will be prepared to compare each metal of interest to concentrations of various candidate metals (or linear combination of metals). For each metal, an optimal scatter plot will be selected that demonstrates high correlation and a strong linear relationship across background soil types. A linear regression will then be used to draw the best fit line through the background data points and to establish 95 percent upper prediction limit (UPL) envelopes which define the expected range for observed background soil concentrations. When this linear regression and the associated prediction limits are superimposed on a scatter plot of site data, any site samples falling outside the prediction limits are subject to further scrutiny. They may belong to the 5% of the background population lying outside of the background-derived prediction envelope, or they may be contaminated.

The proper limits for the expected prediction domain will be established so that the linear relationship is not extrapolated beyond the proven region where background soil data points fall within the regression line.

11.6 STEP 6 OF THE DQO PROCESS: PERFORMANCE AND ACCEPTANCE CRITERIA FOR STATISTICAL EVALUATION AND DECISION MAKING

Control over the accuracy of decision making requires development of a tolerance criterion for each type of hypothesis test utilized. As discussed in Section 11.5.1, for this project, the recommendations from EPA are to employ the hypothesis test form II (site is assumed "dirty" until the hypothesis is rejected based on a statistical test). In applying this hypothesis form, the desired criteria to be controlled include the false positive rate, the false negative rate, and the MDD. To achieve the selected performance

criteria, the number of samples will have to be greater than or equal to a certain minimum and the number of non-detected values will have to fall within an acceptable range to allow certain statistical tests.

11.6.1 False Positive Rate: Alpha Level

When using the hypothesis test form II for site-wide comparison tests, the alpha level is defined as probability of rejecting the assumption that the site is contaminated when in fact the site population is greater than background by an amount equal to the MDD. The probability level (P-level) for alpha has been selected by the project team as 0.05 for all statistical tests to be utilized to compare site data to background. In other words, the alpha level is the probability that the test suggests the site is similar to background, when the site is actually greater than background. A conservative alpha of 0.05 ensures that the probability of overlooking contamination is low.

When individual comparison tests are applied, test form I will be utilized, along with the assumption of a P-level of 0.05. The alpha level of 0.05 will be applied to both univariate UPLs based on one analyte and to multivariate geochemical UPLs.

When comparing background data groups to each other, an overall alpha level of 0.05 will be applied when looking for any type of difference among the background groups. In the case where only two groups are compared, this is mathematically equivalent to performing two separate tests with opposite null hypothesis assumptions and with each test using a one-sided decision-making P-level of 0.025. In the case of more than two groups, the ANOVA method will apply an initial alpha of 0.05, followed by a series of post-hoc comparisons wherein the individual test alpha level is set once to 0.05 and then also adjusted downward following alternative procedures to better control the family-wise error rate.

11.6.2 False Negative Rate: Beta Level

When using the hypothesis test form II with site-wide comparison tests, the beta level is defined as the probability of concluding that the site is contaminated when in fact the site population is NOT greater than background by an amount equal to the MDD. The beta probability has been selected as 0.05 for all two sample statistical tests to be utilized. A conservative beta of 0.05 renders unlikely the conclusions that remediation is required if in fact site concentrations are indistinguishable from background concentrations, within the specified MDD.

When using test form I with individual comparison tests, the beta level depends on the number of samples. Therefore, an established minimum number of samples is required to ensure that the beta level does not exceed 0.05.

11.6.3 Power of the Test

The power of the test is the mathematical relation of 1 minus the beta level (1 – 0.05). When using the hypothesis test form II, power is defined as the probability that the test will conclude that site is NOT greater than background plus MDD, when in fact the site equals background plus MDD. In other words, high power indicates that the test is capable of correctly and confidently concluding that a site is similar to background. When power is high it is unlikely that remediation will be required if in fact site conditions are actually similar to background concentrations levels.

11.6.4 Number of Samples Needed to Achieve Desired Statistical Power, MDD, and Sensitivity

Based on the statistical relationships between alpha, beta, and number of samples, for this project 15 samples were determined to be necessary for each background soil data category (Upper Sand, Transition Unit), groundwater hydrologic regime category (Upper Sand, Transition Unit, Lower Sand, bedrock), and for background data sets for surface water and sediment. Statistical equations published in the guidance documents cited in Table 11-2 were applied to estimate the MDD associated with various numbers of samples collected, to ensure that the project DQO of 2 standard deviations MDD can be achieved by the background sampling design. After collecting two groundwater sampling rounds in 15 monitoring wells screened discretely in the Upper Sand, Transition Unit and Lower Sand, (and two rounds for 4 bedrock wells a statistical test will be performed to determine whether the data for common analytes can be combined for additional statistical power. Similarly, after all soil samples are collected, statistical tests will be performed to determine whether various soil subcategories and surface and subsurface categories can be combined to achieve increased statistical power. Geochemical soil data sets will be evaluated to attempt to establish strong predictive linear relationships between trace background analytes and primary mineral components that are valid across all subcategories of soil.

Table 11-2 utilizes published formula to estimate the MDD given pre-selected alpha (0.05) and beta (0.05) probabilities and given specified numbers of site and background samples. The MDD is shown for two types of site-wide statistical tests, the t-test and the Mann-Whitney (abbreviated WRS) test. This table illustrates that if the number of background samples is 15, the MDD goal of 2-sigma can be achieved with as few as 4 site samples for the t-test and as few as 8 site samples for the WRS test. However, allowance should be made for 10 percent rejected analytical data, so that with only 13 usable background samples, 4 site samples would be required for the t-test and 9 site samples for the WRS test. Although 4 site samples would suffice, the supporting distributional shape test is not reliable with less than 8 to 10 samples, so that the underlying assumptions for the t-test would be difficult to verify. With

the WRS test, no supporting distributional tests are required, so that a realistic goal for site-wide tests is a minimum of 8 or 9 usable data points representing site samples and 13 or 14 usable background points.

TABLE 11-1

Base Back-ground Area	Location	Rationale for Use as Background Area	Media to Be Sampled	Surficial Geologic Unit	Comments
Area 1	Northwest corner of base	Near base boundary. CERFA Report area A-5, former residential area. There has been no use or development since the government acquired property. Some areas have remained forested. Some solid waste present. No known release or disposal of petroleum or hazardous substances. Groundwater flow is expected to be to the north toward the Androscoggin River with a ground water divide to the south.	Surface soil, subsurface soil groundwater	Upper Sand	Install 5 soil borings and collect 5 surface soil and 5 subsurface soil samples in Upper Sand. Install 4 monitoring wells in, the first 4 borings in the Upper Sand.
Area 2	West Mere Brook	Mere Brook enters the base from a forested area offbase to the west. North of the brook, there is a forested area and south of brook and a current disturbed zone, there is more forested area near base boundary. No known release or disposal of petroleum or hazardous substances.	Sediment Surface Water	Upper Sand	Install 2 surface water gauges along Mere Brook west of the runway. Collect 8 surface water and 8 sediment samples in Mere Brook – (see Figure 17-1 for Area 2). Sample 8 surface water,and 8 sediment locations (co-located) in Fall and at the same locations in Spring.
Area 3	Southeast of south end of runway.	Forested area west of former access road and building of unknown use. Potential Lower Sand monitoring well location identified in north-central of Area 3 during Site Walkover on April 28-29, 2009 with Navy, MEDEP and EPA.	Groundwater	Lower Sand	Install 1 electrical conductivity (EC) boring into Presumpscot clay to determine the depth interval of the Lower Sand. Provide EC data to ME CG, to determine location of well screen.
Area 4	South of Golf course	Areas topographically above the golf course provide areas for surface and subsurface soil sampling. Area A-4 in the CERFA report indicated no known or suspected release or disposal of hazardous substances in areas 200 feet (minimum) away from golf course, fuel pipeline, Sites 15 and 16.	Surface soil Subsurface soil Groundwater	Transition Unit	Install 9 borings and collect 9 surface and 9 subsurface soil samples. Install 8 monitoring wells In the Transition Unit– one in each of the first 8 borings.

Base Back-ground Area	Location	Rationale for Use as Background Area	Media to Be Sampled	Surficial Geologic Unit	Comments
Area 5	Southeastern area, north of EOD Range	Proposed for transfer to National Guard. Area A-3 in CERFA report. Historically forested. Undeveloped land with no known or suspected release or disposal of petroleum or hazardous substances. Small pond may be suitable for sampling.	Surface soil Subsurface soil Groundwater	Transition Unit	Install 3 borings and collect 3 surface and 3 subsurface soil samples. Install 3 monitoring wells in Transition Unit, one in each of the borings.
Area 6N	Northeast of Picnic Pond	Undeveloped forested land east of Picnic Pond. No known or suspected release or disposal of petroleum or hazardous substances. Bedrock high to east suggests surface and groundwater flow from unimpacted off base area on to base.	Surface soil Subsurface soil Groundwater	Upper Sand Lower Sand	Install 5 borings and collect 5 surface soil and 5 subsurface soil samples. Install 4 wells in Upper Sand. Also advance EC probe at 3 locations to determine if Lower Sand is present. If the Lower Sand is present, install 3 temporary wells in the Lower Sand.
Area 6S	East of Picnic Pond.	Same rationale as Area 6N.	Surface soil Subsurface soil Groundwater	Transition Unit	Install 3 borings. Collect surface and subsurface soil samples. Install 3 monitoring wells – one in each of the borings in the Transition Unit.
Area 7	North of Main Station	This area provides additional spatial coverage. Boring/well locations should be at least 200 feet north of Rt. 24 (Old Bath Rd.)	Surface soil, Subsurface soil Groundwater	Upper Sand	Install 2 borings and collect 2 surface soil and 2 subsurface soil samples of the Upper Sand. Install 2 monitoring wells in the Upper Sand – one in each of the borings. Install 2 additional monitoring wells in the Upper Sand.
Area north of MW-303	North of MW-303	Location where Lower Sand should be present.	Groundwater	Lower Sand	Advance EC probe at 3 locations to determine if Lower Sand is present. If the Lower Sand is present, install 3 temporary wells in the Lower Sand.
Offsite	East of Eastern Plume	Bedrock wells MW-316A,B and MW 317A, B are located on private property but have been sampled previously.	Groundwater	Bedrock	Collect 2 rounds of samples from 4 wells, unless other on-base uncontaminated bedrock wells can be identified and are acceptable to the

Base Back-ground Area	Location	Rationale for Use as Background Area	Media to Be Sampled	Surficial Geologic Unit	Comments
					project team.
Bowdoin Pines	North of Rt 24 (Old Bath Road), south of railroad tracks, and immediately west of Car Quest Auto Parts Store.	Undeveloped area, forested area (tall pine trees). Area used for recreation (walking trails).	Surface soil Subsurface soil, groundwater	Upper Sand	Install 3 borings, and collect 3 surface and 3 subsurface soil samples from Upper Sand. Install 3 monitoring wells – one in each of the borings in the saturated Upper Sand. (Pending Access agreement between Navy and landowner.)
Coffins Ice Pond	Off-base, - an impound-ment along Mere Brook	Surface water and sediments samples from an impoundment are needed for comparison with surface water and sediments within impoundments on Base.	Surface water, sediment	Upper Sand Transition Unit	Collect 8 surface water samples and 8 sediment samples, one set after a dry period (e.g., September), and the other after a wet period (e.g. April).

TABLE 11-2
MINIMUM DETECTABLE DIFFERENCE FOR HYPOTHESIS TEST FORM 2 (H0: SITE > BACK + MDD)

MDD for t-Test for Various Sample Sizes			
Alpha = 0.05	Beta = 0.05		
1 - alpha = 0.95	1 – beta = 0.95		
Z(1-alpha) = 1.645	Z(1-beta) = 1.645		
No. Site Samples (NS)	NS=15	NS=10	NS=4
No. Back. Samples (NB)	NB=15	NB=15	NB=15
MDD =	1.23	1.38	1.92
No. Site Samples (NS)	NS=15	NS=10	NS=4
No. Back. Samples (NB)	NB=14	NB=14	NB=14
MDD =	1.25	1.40	1.94
No. Site Samples (NS)	NS=15	NS=10	NS=4
No. Back. Samples (NB)	NB=13	NB=13	NB=13
MDD =	1.28	1.43	1.96

MDD for WRS Test for Various Sample Sizes			
Alpha = 0.05	Beta = 0.05		
1 – alpha = 0.95	1 – beta = 0.95		
Z(1-alpha) = 1.645	Z(1-beta) = 1.645		
No. Site Samples (NS)	NS=15	NS=10	NS=8
No. Back. Samples (NB)	NB=15	NB=15	NB=15
MDD =	1.45	1.72	1.95
No. Site Samples (NS)	NS=15	NS=10	NS=8
No. Back. Samples (NB)	NB=14	NB=14	NB=14
MDD =	1.48	1.76	2.00
No. Site Samples (NS)	NS=15	NS=10	NS=9
No. Back. Samples (NB)	NB=13	NB=13	NB=13
MDD =	1.53	1.81	1.91

WRS = Wilcoxon Rank Sum test, also known as Mann-Whitney test
MDD - Minimum Detectable Difference, expressed in units of shift in mean divided by standard deviation.
Alpha - Probability the test concludes Site is NOT > Background + MDD if the true shift in the 2 populations' mean = MDD .
Beta - Probability the test concludes that Site > Background + MDD if the true shift in the mean = MDD.
Power - Probability test will NOT conclude Site > Background + MDD if true shift in the mean = MDD.
Z(value) - Normal distribution function of probability shown in parenthesis.

<u>Sample Size Equation for t-Test, Given N1 not equal to N2</u>:

Dixon, W.J., 1983. Introduction to Statistical Analysis. McGraw-Hill Book Co., New York, New York.

<u>Sample Size Equation for WRS Test, Given N1 not equal to N2</u>:

EPA, 1992. Methods for Evaluating the Attainment of Cleanup Standards. Volume 3: Reference-Based Standards for Soils and Solid Media. Office of Policy, Planning, and Evaluation. Washington, DC.

<u>Sample Size Equation for t-Test, Given N1 = N2</u>:

EPA, 1992. Methods for Evaluating the Attainment of Cleanup Standards. Volume 3: Reference-Based Standards for Soils and Solid Media. Office of Policy, Planning, and Evaluation. Washington, DC.

<u>Sample Size Equation for WRS Test, Given N1=N2</u>:

EPA, 1996. Guidance for Data Quality Assessment. EPA QA/G-9, EPA/600/R-96084. Office of Research and Development, Quality Assurance Division. Washington, D.C.

11.6.5 Analyzing Data Sets with Nondetect (ND) Observations

Because non-detects can bias the outcome of some statistical tests to an unpredictable extent, certain tests will be avoided whenever there are non-detects. For site-wide statistical tests, only nonparametric tests will be employed if there are non-detects present among either site or background data. Distributional shape tests involving non-detects will not be applicable as a prerequisite to site-wide statistical tests involving non-detects since no parametric tests will be used in such cases.

In the event of non-detects occurring in site-wide comparison tests, Gehan's test will be used instead of the t-test or the Mann-Whitney test (EPA, 2008, Navy, 2002). Detailed estimates of the MDD using Gehan's test in the presence of non-detects have not been previously published in guidance documents, so test form I will be applied for all site-wide comparisons which require Gehan's test. In addition, the quantile test will be applied for a nonparametric comparison of just the upper ranks of two data sets, and will avoid using any data points with a rank at or below the largest detection limit.

Univariate individual comparison tests consist of a calculated BTV that is used as an upper concentration limit on background for comparison to an individual site sample result. BTVs are available in ProUCL with adjustments to impute proxy positive concentrations for all non-detected values (EPA, 2007a, 2007b). The magnitude, range, and frequency of non-detected background values affect the overall uncertainty contributed to the estimated upper prediction limit or BTV. There is also an alternative method of estimating BTVs using 95th quantiles, which contributes more variance and uncertainty as the size of the background data set becomes smaller. When there are non-detects, no across-the-board recommendation can be made as to identifying one of these methods as always being more reliable than the other. For each substance, both types of candidate BTVs will be presented in the background study, from which a selection will be made that represents either an upper prediction limit with imputed detection limits or a nonparametric 95th quantile, with a decision made on a case-by-case basis considering all relevant statistical recommendations and software output.

11.6.6 Develop the Plan for Obtaining the Data

Part of the development of the plan for obtaining the data for this project was done under Step 4 during the planning session because of the time constraints. As agreed during the DQO meeting, the rest was completed as part of UFP-SAP development and is presented in Worksheet #17.

SAP Worksheet #12a -- Measurement Performance Criteria Table note matrix in table entry

(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table –Groundwater, Surface Water, , Soil, Sediment - Field QC Samples

QC Sample	Analytical Group[1]	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Temperature Blank	PAHs, pesticides, herbicides, TOC, metals (no PAHs for groundwater samples), chloride and bromide (groundwater only)	One per cooler	Accuracy/bias/ representativeness	4° C $\pm$ 2° C	S
Rinsate Blank	PAHs, pesticides, herbicides, metals (no PAHs for groundwater), chloride and bromide (groundwater only)	One per 20 samples	Bias / contamination	No target analytes > ½ Quantitation Limit (QL). Exception: for common laboratory contaminants, no target analytes > QL.	S & A
Field Blank	PAHs, pesticides, herbicides, metals (no PAHs for groundwater), chloride and bromide (groundwater only)	One per water source	Bias/contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.	S & A

QC Sample	Analytical Group[1]	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Field Duplicates	PAHs, pesticides, herbicides, TOC, metals (no PAHs for groundwater samples), chloride and bromide (groundwater only)	One per 10 samples	Precision/comparability	Organic—Relative Percent Difference (RPD) ≤ 50% Soils; RPD ≤ 30% waters. If sample results are < 2x QL, professional judgment is used. Inorganic--Values ≥ 5xPractical Quantitation Limit (PQL): RPD ≤ 50% Soils; RPD ≤ 30% waters. Values < 5xPQL: Absolute Difference ≤ 4xPQL Soils; Absolute Difference ≤ 2xPQL Waters.	S & A

1 - Refer to Worksheet #20 for a listing of analytical groups applicable for each matrix.

SAP Worksheet #12b -- Measurement Performance Criteria Table

(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table –Surface Water– Analytical PAH QC Samples

QC Sample	Analytical Group[1]	Frequency	DQIs	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	PAHs	One per analytical batch	Bias/contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.	A
Reagent Blank	PAHs	One per lot	Bias/contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.	A
Surrogate Standards	PAHs	Each field and QC sample	Accuracy/bias	See Worksheet #28a	A
Internal Standards	PAHs	Every field and QC samples	Accuracy/bias	See Worksheet #28a	A
Laboratory Control Sample (LCS)	PAHs	One per analytical batch	Accuracy/bias	See Worksheet #28a	A
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	PAHs	One set per 20 samples	Accuracy/Precision	See Worksheet #28a	A
All samples	PAHs	All samples	Sensitivity	QLs < project action limits (PALs) listed in Worksheet # 15	A
All samples	PAHs	All samples	Data completeness	95% overall	S & A

1- Refer to Worksheet #2 for a listing of analytical groups applicable for each matrix.

SAP Worksheet #12c -- Measurement Performance Criteria Table

(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table –Soil/ Sediment – Analytical PAH, Pesticide, and Herbicide QC Samples

QC Sample	Analytical Group[1]	Frequency	DQIs	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	PAHs, Pesticides, Herbicides	One per analytical batch	Bias/contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.	A
Reagent Blank	PAHs, Pesticides, Herbicides	One per lot	Bias/contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.	A
Surrogate Standards	(1) PAHs, (2) Pesticides (3) Herbicides	Each field and QC sample	Accuracy/bias	(1) See Worksheet #28b (2) See Worksheet #28c (3) See Worksheet #28d	A
Internal Standards	PAHs	Each field and QC sample	Accuracy/bias	See Worksheet #28b	A
LCS	(1) PAHs, (2) Pesticides, (3) Herbicides	One per analytical batch	Accuracy/Bias	(1) See Worksheet #28b (2) See Worksheet #28c (3) See Worksheet #28d	A
MS/MSD	(1) PAHs, (2) Pesticides, (3) Herbicides	One set per 20 samples	Accuracy/Precision	(1) See Worksheet #28b (2) See Worksheet #28c (3) See Worksheet #28d	A
All samples	PAHs, Pesticides, Herbicides	All samples	Sensitivity	QLs < PALs listed in Worksheet # 15	A
All samples	PAHs, Pesticides, Herbicides	All samples	Data completeness	95% overall	S & A

1 - Soil analytical groups: PAHs, TCL Pesticides, Herbicides
Sediment analytical groups: PAHs, TCL Pesticides, Herbicides

SAP Worksheet #12d -- Measurement Performance Criteria Table

(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table – Groundwater/Surface Water – Analytical Total and Dissolved Metals and Chloride/Bromide QC Samples

QC Sample	Analytical Group[1]	Frequency	DQIs	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Independent Calibration Verification (ICV)	TAL Metals (Dissolved and Total), Chloride and Bromide	Immediately after calibration	Accuracy/bias	Recovery 90-110%	A
Initial Calibration Blank (ICB)	TAL Metals (Dissolved and Total)	Immediately after ICV	Accuracy/bias/ contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.	A
PQL (low level) Standard	TAL Metals (Dissolved and Total)	Daily after one-point initial calibration	Accuracy/bias	Recovery 70-130%	A
Method blank	TAL Metals (Dissolved and Total) , Chloride and Bromide	One per 20 samples	Bias and Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.	A
LCS	TAL Metals (Dissolved and Total) , Chloride and Bromide	One set per 20 samples	Accuracy/bias	Recovery 80-120%	A
Inductively-Coupled Plasma (ICP) Interference Check Sample	TAL Metals (ICP only) (Dissolved and Total)	Once at beginning of run	Accuracy/bias	Interference Check Solution (ICS)-A: Unspiked analytes ≤ 2x MDL, if interferent concentrations in sample > 50% interferent concentration in ICS-A solution. ICS-AB: Recovery of each analyte within ± 20% of true value.	A
MS	TAL Metals (Dissolved and Total)	One per 20 samples	Accuracy/bias	Recovery 75-125%	A
MS/MSD	Chloride and Bromide	One per 20 samples	Accuracy/precision	Recovery 80-120% RPD ± 20%	A

QC Sample	Analytical Group[1]	Frequency	DQIs	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Post-Digestion Spike	ICP Metals (Dissolved and Total)	When MS or serial dilution fails	Accuracy/bias	Recovery 75-125%	A
Laboratory Duplicate	TAL Metals (Dissolved and Total)	One per 20 samples	Precision	Values ≥ 5xPQL: RPD ± 20% Values < 5xPQL: Absolute Difference ≤ PQL	A
ICP Serial Dilution	TAL Metals (ICP only) (Dissolved and Total)	One per 20 samples	Accuracy & Precision	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 15% of the original result.	A
Internal Standard (IS)	Metals Inductively Coupled Plasma/Mass Spectrometry (ICP-MS) only (Dissolved and Total)	Appropriate IS in every sample	Accuracy/Bias	IS relative intensity 30-120% for field samples	A
All samples	TAL Metals (Dissolved and Total)	Each target analyte	Sensitivity	QLs < PALs listed in Worksheet # 15	S&A
All samples	TAL Metals (Dissolved and Total)	Calculated for the data set	Data Completeness	95% Overall	S&A

1 - Dissolved TAL metals samples provide dissolved metals results. Total TAL metals samples provide total metals results. Chloride and bromide analysis is for groundwater only.

SAP Worksheet #12e -- Measurement Performance Criteria Table

(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table –Soil/Sediment – Analytical Metals, TOC, Grain Size QC Samples

QC Sample	Analytical Group[1]	Frequency	DQIs	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
ICV	TAL Metals	Immediately after calibration	Accuracy/bias	Recovery 90-110%	A
ICB	TAL Metals	Immediately after ICV	Accuracy/bias/ contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.	A
PQL (low level) Standard	TAL Metals	Daily after one-point initial calibration	Accuracy/bias	Recovery 70-130%	A
Method blank	TAL Metals, TOC	One per 20 samples	Bias and Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.	A
LCS	TAL Metals, TOC	One set per 20 samples	Accuracy/bias	Metals: Recovery within vendor supplied limits TOC: Recovery 80-120%	A
ICP Interference Check Sample	TAL Metals (ICP only)	Once at beginning of run	Accuracy/bias	ICS-A: Unspiked analytes ≤ 2x MDL, if interferent concentrations in sample > 50% interferent concentration in ICS-A solution. ICS-AB: Recovery of each analyte within ± 20% of true value.	A
MS	TAL Metals	One per 20 samples	Accuracy/bias	Recovery 75-125%	A
Post-Digestion Spike	ICP Metals	When MS or serial dilution fails	Accuracy/bias	Recovery 75-125%	A

QC Sample	Analytical Group[1]	Frequency	DQIs	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Laboratory Duplicate	TAL Metals, TOC	One per 20 samples	Precision	Metals: Values ≥ 5xPQL: RPD ± 35%; Values < 5xPQL: Absolute Difference ≤ 2x PQL TOC: RPD ± 20%	A
ICP Serial Dilution	TAL Metals (ICP only)	One per 20 samples	Accuracy & Precision	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 15% of the original result.	A
IS	Metals by ICP-MS	Appropriate IS in every sample	Accuracy/Bias	IS relative intensity 30-120% for field samples	A
All samples	TAL Metals	Each target analyte	Sensitivity	QLs < PALs listed in Worksheet # 15	S&A
All samples	TAL Metals, TOC, Grain Size	Calculated for the data set	Data Completeness	95% Overall	S&A

1 - Grain size (sieve analysis only) is analyzed only for sediment samples.

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

(UFP-QAPP Manual Section 2.7)

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
Data from previous LTM events at various sites at NAS Brunswick	Monitoring Event 31 for various sites (ECC 2008)	Originating organization: ECC Data types: groundwater data and water level measurements	The data will be used to evaluate concentration and water level trends.	Data quality is otherwise adequate and comparable to data that will be generated under this study.
New data generated through field activities in Fall 2008	Field reports from Site 7, Site 9, Site 2, and Picnic Pond	Originating organization: ECC, TtNUS Data types: preliminary water level measurements, soil boring data	If available data will be used to support background well location	Preliminary data.

SAP Worksheet #14 -- Summary of Project Tasks

(UFP-QAPP Manual Section 2.8.1)

Field Tasks - The field tasks are described below. The sampling design is summarized on Worksheet #17. Sample collection locations and methods are summarized on Worksheet #18. Analytical methods/SOP requirements are summarized on Worksheet #19.

- Mobilization/Demobilization – includes mobilization of equipment and staff to the site, field team orientation, and demobilization.

- Background Area Reconnaissance – consists of an inspection of proposed background areas to assure they are suitable for this project. The background areas were observed by the project team on April 28-29, 2009. The final locations will be presented to the project team for approval prior to sample collection.

- Utility Clearance - A DIGSAFE number and NAS Brunswick utility clearance will be obtained at all drilling locations prior to mobilizing drilling equipment.

- Surface and Subsurface Soil Sampling –an estimated 30 soil borings will be located within the proposed areas for continuous soil sampling. When the minimum number of soil samples per soil type and geologic type are collected, the soil sampling program will be considered completed. For statistical analysis, 15 surficial soil samples and 15 subsurface soil samples for each of the predominant surficial geologic units present are required. Four soil types are identified in Worksheet 10: Upper Sand, Transition Unit, Presumpscot clay and till; and recommended background Areas 1-7 are shown on Figure 10-2. The Lower Sand, Presumpscot Clay and till do not occur within 10 feet of ground surface; therefore, soil samples will not be collected from these units. 30 surface soil samples (2 soil types x 15 samples/soil type) and 30 subsurface soils (2 soil types x 15 samples/soil type) are required. The total number of soil samples is 60. It will be necessary to classify surface soil samples and subsurface soil samples into the following groups upon collection:

  - Upper Sand
  - Transitional Unit

- Monitoring Well Construction/Development – 37 new overburden monitoring wells will be completed and developed (see Table 17-1). Fifteen wells will be completed in the Upper Sand, 14 will be

completed in the Transition Unit, and 8 new wells will be completed in the Lower Sand unit. All wells will be sampled two times to obtain sufficient samples for statistical analysis to evaluate whether common analytes from different units can be combined. The two sets of groundwater samples will also represent temporal groundwater chemical concentration variation, because one set will be collected after a dry period (e.g. September) and the other set after a wet period (e.g April). No bedrock wells will be installed. Four existing bedrock wells located adjacent to the Base can be sampled; each will be sampled twice to obtain representative background concentrations in bedrock groundwater. These wells have been sampled previously as part of the Eastern Plume Study. Alternately, if the project team agrees, other existing on-base bedrock wells can be used. It will be necessary to classify groundwater samples into the following groups upon collection:

- Upper Sand
- Transition Unit
- Lower Sand
- Bedrock

- Groundwater Sampling – A total of 98 groundwater samples will be obtained (3 units x 15 samples per unit) including four bedrock wells all sampled during two separate mobilizations. All groundwater samples will be collected following EPA's Low Flow (Low Stress) sampling protocol.

- Surface Water Sampling –Eight surface water samples will be collected from Mere Brook and eight surface water samples from Coffins Ice Pond in the spring, and eight locations will be sampled from both surface-water bodies in the fall. The sample location coordinates will be determined using Global Positioning Systems (GPS).

- Sediment Sampling –Eight sediment samples will be collected from Mere Brook and in the spring, eight surface water samples from Coffins Ice Pond, and eight locations will be sampled from both surface-water bodies in the fall. Sediment sample locations will be co-located with surface water. Sample location coordinates will be determined using GPS.

- Monitoring Well Survey – Maine Licensed Surveyor - locations relative to UTM/WGS 84/UTM zone 19N in meters; elevations relative to msl.

- Equipment decontamination – will be performed to avoid potential cross contamination between sample locations.

- Investigative Derived Waste (IDW) Characterization and Disposal - will be performed after all IDW has been containerized. IDW shall be handled in accordance with SOP SA-7.1

Field Quality Control Tasks – QC samples will be collected, including field duplicates and field blanks. See Worksheet #20 for a table summarizing field quality control samples.

Analytical Tasks

- Analytical SOPs - see Worksheet #23 for lists of the applicable SOPs.
- Analytical instrument calibration -- see Worksheet #24 for calibration requirements.
- Analytical instrument and equipment maintenance – see Worksheet #25 for requirements.

Sample Collection Tasks

- Sample handling system including documentation, tracking, archival and disposal - see Worksheet #26 for applicable requirements.

- Sample custody requirements, procedures/SOPs, and sample container identification, and example Chain-of-Custody form and seal - see Worksheet #27.

Quality Control Samples - See Worksheet #28 for a list of laboratory QC samples.

Data Management Tasks

- Project documentation and records
  - Field sample collection and field measurement records - See Worksheet #29 for documentation requirements.
  - Laboratory data package deliverables - See Worksheet #29 and the Analytical Technical Specification in Appendix C.
  - Data assessment documents and records – See Worksheet #29 for these requirements.

- Data recording formats - See Worksheet #27 for these requirements.
- Data handling and management – See Worksheet #29 for these requirements.
- Data tracking and control – See Worksheet #29 for these requirements.
- Analytical Services – See Worksheet #30 for these specifications.

Assessment Oversight

- Planned assessments - See Worksheet #31. An assessment is not currently planned at the project level but may be conducted at the TtNUS programmatic level.
- Assessment findings and CAs – See Worksheet #32.
- QA management reports - See Worksheet #33.

Data Review

- Data verification – See Worksheet #34.
- Data validation – See Worksheets #35 and #36.
- Usability assessment – See Worksheet #37.

Reports – A data package and memorandum documenting the results of the reconnaissance and data review, and presenting the final sample locations will be prepared for project team review. The data package will include an electronic data deliverable (EDD) in the MEDEP's EDD Verision 5.0. The data package will include field parameter data, water level and flow data, and laboratory analytical data for soil, surface water and groundwater. Laboratory analytical data will include field quality control sample results, surrogate recoveries in percent, and matrix spike/matric duplicate recoveries in percent. Comments on the data package will be discussed and the memorandum will be finalized.

A draft background data report will be prepared to document field activities, field observations/measurements, analytical results, quality assurance/data usability assessment, statistical evaluation, summary and conclusions, and recommendations. The draft report will be reviewed and responses provided. After responses are addressed, then a final report will be issued. The ME CG will sign and stamp the final reports. Additionally, an interim soils background report will be generated prior to the draft background data report because soil sampling is scheduled to be complete approximately 8 months prior to the completion of groundwater sampling.

SAP Worksheet #15a -- Reference Limits and Evaluation Table, Soil Samples

(UFP-QAPP Manual Section 2.8.1)

Matrix: Soil

Analytical Group:

Analyte	CAS Number	PAL[1,2] (mg/Kg)	Analytical Method	PQL Goal (mg/Kg)	Laboratory-specific	
					QLs (mg/Kg)	MDLs (mg/Kg)
PAHs						
2-Methylnaphthalene	91-57-6	29	SW846 8270C SIM	0.33	0.020	0.0028
Acenaphthene	83-32-9	29	SW846 8270C SIM	0.33	0.020	0.0015
Acenaphthylene	208-96-8	29	SW846 8270C SIM	0.33	0.020	0.0013
Anthracene	120-12-7	29	SW846 8270C SIM	0.33	0.020	0.0026
Benzo(a)anthracene	56-55-3	0.15	SW846 8270C SIM	0.020	0.020	0.0025
Benzo(a)pyrene	50-32-8	*0.015*	SW846 8270C SIM	0.020	0.020	0.0021
Benzo(b)fluoranthene	205-99-2	0.15	SW846 8270C SIM	0.020	0.020	0.0022
Benzo(g,h,i)perylene	191-24-2	1.1	SW846 8270C SIM	0.33	0.020	0.0033
Benzo(k)fluoranthene	207-08-9	1.1	SW846 8270C SIM	0.33	0.020	0.0019
Chrysene	218-01-9	1.1	SW846 8270C SIM	0.33	0.020	0.0026
Dibenzo(a,h)anthracene	53-70-3	*0.015*	SW846 8270C SIM	0.020	0.020	0.0038
Fluoranthene	206-44-0	29	SW846 8270C SIM	0.33	0.020	0.0018
Fluorene	86-73-7	29	SW846 8270C SIM	0.33	0.020	0.0016
Indeno(1,2,3-c,d)pyrene	193-39-5	0.15	SW846 8270C SIM	0.020	0.020	0.0043

Analyte	CAS Number	PAL[1,2] (mg/Kg)	Analytical Method	PQL Goal (mg/Kg)	Laboratory-specific	
					QLs (mg/Kg)	MDLs (mg/Kg)
Naphthalene	91-20-3	3.9	SW846 8270C SIM	0.33	0.020	0.0018
Phenanthrene	85-01-8	29	SW846 8270C SIM	0.33	0.020	0.0043
Pyrene	129-00-0	1.1	SW846 8270C SIM	0.33	0.020	0.0069
Pesticides						
4,4' DDD	72-54-8	0.021	SW846 8081A	0.0033	0.0033	0.00071
4,4'-DDE	72-55-9	0.021	SW846 8081A	0.0033	0.0033	0.00070
4,4'-DDT	50-29-3	0.021	SW846 8081A	0.0033	0.0033	0.00082
Aldrin	309-00-2	0.00006	SW846 8081A	0.0017	0.0017	0.00068
alpha-BHC	319-84-6	*0.00072*	SW846 8081A	0.0017	0.0017	0.00071
alpha-Chlordane	5103-71-9	0.00003	SW846 8081A	0.0017	0.0017	0.00091
beta-BHC	319-85-7	0.0026	SW846 8081A	0.0017	0.0017	0.00076
delta-BHC	319-86-8	0.00005	SW846 8081A	0.0017	0.0017	0.00097
Dieldrin	60-57-1	0.004	SW846 8081A	0.0033	0.0033	0.00069
Endosulfan I	959-98-8	0.0001	SW846 8081A	0.0017	0.0017	0.00072
Endosulfan II	33213-65-9	0.0001	SW846 8081A	0.0033	0.0033	0.00069
Endosulfan sulfate	1031-07-8	0.0001	SW846 8081A	0.0033	0.0033	0.00080
Endrin	72-20-8	0.00004	SW846 8081A	0.0033	0.0033	0.00077
Endrin aldehyde	7421-93-4	0.00004	SW846 8081A	0.0033	0.0033	0.00091
Endrin ketone	53494-70-5	0.00004	SW846 8081A	0.0033	0.0033	0.00077
gamma-BHC (Lindane)	58-89-9	0.00005	SW846 8081A	0.0017	0.0017	0.00073

Analyte	CAS Number	PAL[1,2] (mg/Kg)	Analytical Method	PQL Goal (mg/Kg)	Laboratory-specific	
					QLs (mg/Kg)	MDLs (mg/Kg)
gamma-Chlordane	5103-74-2	0.00003	SW846 8081A	0.0017	0.0017	0.00082
Heptachlor	76-44-8	0.0007	SW846 8081A	0.0017	0.0017	0.00082
Heptachlor epoxide	1024-57-3	0.0000002	SW846 8081A	0.0017	0.0017	0.00076
Methoxychlor	72-43-5	31	SW846 8081A	0.017	0.017	0.0009
Toxaphene	8001-35-2	0.44	SW846 8081A	0.17	0.17	0.022
Herbicides						
2,4-D	94-75-7	1.5	SW846 8151A	0.033	0.033	0.0070
2,4-DB	94-82-6	36	SW846 8151A	0.033	0.033	0.016
2,4,5-TP (Silvex)	93-72-1	20	SW846 8151A	0.033	0.033	0.0048
2,4,5-T	93-76-5	11	SW846 8151A	0.033	0.033	0.0058
Dalapon	75-99-0	180	SW846 8151A	0.170	0.170	0.0114
Dicamba	1918-00-9	13	SW846 8151A	0.033	0.033	0.0059
Dichloroprop	120-36-5	--	SW846 8151A	0.067	0.067	0.0046
Dinoseb	88-85-7	0.37	SW846 8151A	0.170	0.170	0.0097
MCPA	94-74-6	*3.1*	SW846 8151A	5	5	0.960
MCPP	93-65-2	6.1	SW846 8151A	3.3	3.3	1.07
Metals						
Aluminum	7429-90-5	170	SW846 6010B	30	30	2.21
Antimony	7440-36-0	0.27	SW846 6020	0.1	0.1	0.027
Arsenic	7440-38-2	*0.39*	SW846 6020	0.5	0.5	0.169

Analyte	CAS Number	PAL[1,2] (mg/Kg)	Analytical Method	PQL Goal (mg/Kg)	Laboratory-specific	
					QLs (mg/Kg)	MDLs (mg/Kg)
Barium	7440-39-3	330	SW846 6010B	10	0.5	0.0312
Beryllium	7440-41-7	4	SW846 6010B	0.5	0.5	0.0124
Cadmium	7440-43-9	0.36	SW846 6020	0.25	0.1	0.00639
Calcium	7440-70-2	--	SW846 6010B	500	5.0	2.96
Chromium	7440-47-3	23	SW846 6010B	4	1.5	0.0461
Cobalt	7440-48-4	3.3	SW846 6020	2.5	0.1	0.00382
Copper	7440-50-8	28	SW846 6010B	2.5	2.5	0.133
Iron	7439-89-6	5500	SW846 6010B	10	10	0.611
Lead	7439-92-1	11	SW846 6010B	1	0.5	0.122
Magnesium	7439-95-4	--	SW846 6010B	500	5.0	1.08
Manganese	7439-96-5	180	SW846 6010B	5	0.5	0.196
Mercury	7439-97-6	0.1	SW846 7471A	0.04	0.040	0.0034
Nickel	7440-02-0	38	SW846 6010B	4	4.0	0.0131
Potassium	7440-09-7	--	SW846 6010B	500	100	3.71
Selenium	7782-49-2	0.52	SW846 6020	0.5	0.5	0.0640
Silver	7440-22-4	4.2	SW846 6010B	1.5	1.5	0.0677
Sodium	7440-23-5	--	SW846 6010B	500	100	2.21
Thallium	7440-28-0	0.51	SW846 6020	0.2	0.2	0.0130
Vanadium	7440-62-2	7.8	SW846 6010B	2.5	2.5	0.0316
Zinc	7440-66-6	46	SW846 6010B	6	2.5	0.171

Analyte	CAS Number	PAL[1,2] (mg/Kg)	Analytical Method	PQL Goal (mg/Kg)	Laboratory-specific	
					QLs (mg/Kg)	MDLs (mg/Kg)
TOC	--	--	SW846 9060A		400	85

Notes:

1 - The PAL represents the lowest risk-based or regulatory criterion applicable for the project. References (criteria and sources) used to determine PALs are provided below in Table 15a. The lowest criterion for each analyte is shown in **bold font** in Table 15a.

2 - The *shading and italicizing* of a PAL indicates that laboratory-specific QL exceeds the action limit using the analytical methods conventionally available (and specifically the methods cited in this table), but the MDL is less than the PAL. The laboratory will evaluate results down to the MDL, and will report positive detections between the QL and MDL as estimated due to uncertainty of quantitation below the QL. In the case of arsenic, the metal typically occurs naturally at concentrations greater than the indicated QL and PAL, and the arsenic QL is within these background concentrations so the PAL exceedance is not a concern. The shading (without italics) of a PAL indicates that the laboratory-specific QL and MDL exceed the action limit using the analytical methods conventionally available (and specifically the methods cited in this table). Because site investigations at NASB frequently involve the use of analytical methods with quantitation and DLs comparable to those listed in this table, more sensitive methods were not sought for this project. If, however, a specific project would require more sensitive analytical methods, additional background data may need to be collected for that project.

Abbreviations:

-- - Not available

TABLE 15A – DETERMINATION OF PALS, SOILS

Analyte	CAS Number	ORNL Regional Screening Levels, Residential Soil[2] (mg/kg)	USEPA Soil Screening Level[3] (mg/kg)		Maine Remedial Action Guidelines for Contaminated Soil[4] (mg/kg)				Ecological Soil Screening Level (mg/kg)[13]	Source of Ecological Soil Screening Level	Lowest Criterion[1] (mg/kg)
			Soil to Air	Soil to GW DAF = 20	Residen-tial	Residen-tial Basis	Transfer to GW	Transfer to GW Basis			
PAHs											
2-Methylnaphthalene	91-57-6	31	--	--	--		--		**29**	EPA ECO-SSL	29
Acenaphthene	83-32-9	340	--	630	--		--		**29**	EPA ECO-SSL	29
Acenaphthylene	208-96-8	340 [5]	--	--	--		--		**29**	EPA ECO-SSL	29
Anthracene	120-12-7	1700	--	13000	--		--		**29**	EPA ECO-SSL	29
Benzo(a)anthracene	56-55-3	**0.15**	--	3.2	--		--		1.1	EPA ECO-SSL	0.15
Benzo(a)pyrene	50-32-8	***0.015***	--	8.2	2	Ing., C	8	SSL	1.1	EPA ECO-SSL	0.015
Benzo(b)fluoranthene	205-99-2	**0.15**	--	9.8	--		--		1.1	EPA ECO-SSL	0.15
Benzo(g,h,i)perylene	191-24-2	170 [6]	--	--	--		--		**1.1**	EPA ECO-SSL	1.1
Benzo(k)fluoranthene	207-08-9	1.5	--	9.8	--		--		**1.1**	EPA ECO-SSL	1.1
Chrysene	218-01-9	15	--	3.2	--		--		**1.1**	EPA ECO-SSL	1.1
Dibenzo(a,h)anthracene	53-70-3	***0.015***	--	30	--		--		1.1	EPA ECO-SSL	0.015
Fluoranthene	206-44-0	230	--	6300	--		--		**29**	EPA ECO-SSL	29
Fluorene	86-73-7	230	--	810	--		--		**29**	EPA ECO-SSL	29
Indeno(1,2,3-c,d)pyrene	193-39-5	**0.15**	--	28	--		--		1.1	EPA ECO-SSL	0.15
Naphthalene	91-20-3	**3.9**	170	61	245	Inh., NC	84	SSL	29	EPA ECO-SSL	3.9
Phenanthrene	85-01-8	170 [6]	--	--	--		--		**29**	EPA ECO-SSL	29
Pyrene	129-00-0	170	--	4600	--		--		**1.1**	EPA ECO-SSL	1.1

Analyte	CAS Number	ORNL Regional Screening Levels, Residential Soil[2] (mg/kg)	USEPA Soil Screening Level[3] (mg/kg)		Maine Remedial Action Guidelines for Contaminated Soil[4] (mg/kg)				Ecological Soil Screening Level (mg/kg)[13]	Source of Ecological Soil Screening Level	Lowest Criterion[1] (mg/kg)
			Soil to Air	Soil to GW DAF = 20	Residen-tial	Residen-tial Basis	Transfer to GW	Transfer to GW Basis			
Pesticides											
4,4' DDD	72-54-8	2.0	--	14	--		--		**0.021**	EPA ECO-SSL	0.021
4,4'-DDE	72-55-9	1.4	--	45	--		--		**0.021**	EPA ECO-SSL	0.021
4,4'-DDT	50-29-3	1.7	750	26	50	Ing., C	32	SSL	**0.021**	EPA ECO-SSL	0.021
Aldrin	309-00-2	0.029	3.4	0.49	--		--		**6E-05**	TV	0.00006
alpha-BHC	319-84-6	0.077	0.75	***0.00072***	--		--		0.003	TV	0.00072
alpha-Chlordane	5103-71-9	1.6 [7]	72	9.6	--		10	SSL	**3E-05** [14]	TV	0.00003
beta-BHC	319-85-7	0.27	6	**0.0026**	--		--		0.009	TV	0.0026
delta-BHC	319-86-8	0.077 [8]	--	--	--		--		**5E-05** [15]	TV	0.00005
Dieldrin	60-57-1	0.03	1.1	0.0046	0.3	Ing. & Inh., C	**0.004**	SSL	0.0049	EPA ECO-SSL	0.004
Endosulfan I	959-98-8	37 [9]	--	20	--		18	SSL	**0.0001**	TV	0.0001
Endosulfan II	33213-65-9	37 [9]	--	20	--		--		**0.0001**	TV	0.0001
Endosulfan sulfate	1031-07-8	37 [9]	--	--	--		--		**0.0001**	TV	0.0001
Endrin	72-20-8	1.8	--	0.99	60	Ing. & Inh., NC	1	SSL	**4E-05**	TV	0.00004
Endrin aldehyde	7421-93-4	1.8 [10]	--	--	--		--		**4E-05**	TV	0.00004
Endrin ketone	53494-70-5	1.8 [10]	--	--	--		--		**4E-05**	TV	0.00004
gamma-BHC (Lindane)	58-89-9	0.52	--	0.0094	--		--		**5E-05**	TV	0.00005
gamma-Chlordane	5103-74-2	1.6 [7]	72	9.6	--		--		**3E-05** [14]	TV	0.00003
Heptachlor	76-44-8	0.11	4.1	23	4	Ing., C	23	SSL	**0.0007**	TV	0.0007

Analyte	CAS Number	ORNL Regional Screening Levels, Residential Soil[2] (mg/kg)	USEPA Soil Screening Level[3] (mg/kg)		Maine Remedial Action Guidelines for Contaminated Soil[4] (mg/kg)				Ecological Soil Screening Level (mg/kg)[13]	Source of Ecological Soil Screening Level	Lowest Criterion[1] (mg/kg)
			Soil to Air	Soil to GW DAF = 20	Residen-tial	Residen-tial Basis	Transfer to GW	Transfer to GW Basis			
Heptachlor epoxide	1024-57-3	0.053	4.7	0.67	--		--		**2E-07**	TV	2E-07
Methoxychlor	72-43-5	**31**	--	160	--		--		--	--	31
Toxaphene	8001-35-2	**0.44**	87	31	--		--		--	--	0.44
Herbicides											
2,4-D	94-75-7	69	--	**1.5**	--		--		--	--	1.5
2,4-DB	94-82-6	49	--	**36**	--		--		--	--	36
2,4,5-TP (Silvex)	93-72-1	49	--	**20**	--		--		--	--	20
2,4,5-T	93-76-5	61	--	**11**	1900	Ing., NC, Ceiling	--		--	--	11
Dalapon	75-99-0	**180**	--	--	--		--		--	--	180
Dicamba	1918-00-9	180	--	**13**	--		--		--	--	13
Dichloroprop	120-36-5	--	--	--	--		--		--	--	--
Dinoseb	88-85-7	6.1	--	**0.37**	5	Inh., NC	--		--	--	0.37
MCPA	94-74-6	***3.1***	--	--	--		--		--	--	3.1
MCPP	93-65-2	**6.1**	--	--	--		--		--	--	6.1
Metals											
Aluminum	7429-90-5	7700	7090000	**170**	--		--		pH [16]	EPA ECO-SSL	170
Antimony	7440-36-0	3.1	--	5.4	--		--		**0.27**	EPA ECO-SSL	0.27
Arsenic	7440-38-2	***0.39***	769	5.8	10	Ing., C	29	SSL	18	EPA ECO-SSL	0.39
Barium	7440-39-3	1500	709000	1600	10000	Ceiling	1600	SSL	**330**	EPA ECO-SSL	330

Analyte	CAS Number	ORNL Regional Screening Levels, Residential Soil[2] (mg/kg)	USEPA Soil Screening Level[3] (mg/kg)		Maine Remedial Action Guidelines for Contaminated Soil[4] (mg/kg)				Ecological Soil Screening Level (mg/kg)[13]	Source of Ecological Soil Screening Level	Lowest Criterion[1] (mg/kg)
			Soil to Air	Soil to GW DAF = 20	Residen-tial	Residen-tial Basis	Transfer to GW	Transfer to GW Basis			
Beryllium	7440-41-7	16	1380	63	**4**	Ing., C	63	SSL	21	EPA ECO-SSL	4
Cadmium	7440-43-9	7.0	1840	7.5	27	Dermal, NC	8	SSL	**0.36**	EPA ECO-SSL	0.36
Calcium	7440-70-2	--	--	--	--		--		--	--	--
Chromium	7440-47-3	**23** [11]	276	42	--		38	SSL	26	EPA ECO-SSL	23
Cobalt	7440-48-4	--	1180	**3.3**	--		--		13	EPA ECO-SSL	3.3
Copper	7440-50-8	310	--	11000	650	New Jersey	--		**28**	EPA ECO-SSL	28
Iron	7439-89-6	**5500**	--	--	--		--		pH [16]	EPA ECO-SSL	5500
Lead	7439-92-1	40	--	--	375	DHS, Ceiling	--		**11**	EPA ECO-SSL	11
Magnesium	7439-95-4	--	--	--	--		--		--	--	--
Manganese	7439-96-5	**180**	70900	2200	--		--		220	EPA ECO-SSL	180
Mercury	7439-97-6	0.67 [12]	2.9	2.1	60	Ing., NC	2	SSL	**0.1**	ORNL Invert	0.1
Nickel	7440-02-0	160	--	950	3800	Ing., NC	130	SSL	**38**	EPA ECO-SSL	38
Potassium	7440-09-7	--	--	--	--		--		--	--	--
Selenium	7782-49-2	39	--	5.2	950	Ing., NC	5	SSL	**0.52**	EPA ECO-SSL	0.52
Silver	7440-22-4	39	--	31	950	Ing., NC	34	SSL	**4.2**	EPA ECO-SSL	4.2
Sodium	7440-23-5	--	--	--	--		--		--	--	--
Thallium	7440-28-0	**0.51**	--	1.1	--		--		1	ORNL Plant	0.51
Vanadium	7440-62-2	**39** [17]	--	5100	--		--		7.8	EPA ECO-SSL	7.8

Analyte	CAS Number	ORNL Regional Screening Levels, Residential Soil[2] (mg/kg)	USEPA Soil Screening Level[3] (mg/kg)		Maine Remedial Action Guidelines for Contaminated Soil[4] (mg/kg)				Ecological Soil Screening Level (mg/kg)[13]	Source of Ecological Soil Screening Level	Lowest Criterion[1] (mg/kg)
			Soil to Air	Soil to GW DAF = 20	Residen-tial	Residen-tial Basis	Transfer to GW	Transfer to GW Basis			
Zinc	7440-66-6	2300	--	14000	1500	New Jersey	12000	SSL	**46**	EPA ECO-SSL	46

Notes:

DAF Dilution Attenuation Factor
TCL Target Compound List
TAL Target Analyte List
PAH Polycyclic Aromatic Hydrocarbons
SIM Selective Ion Monitoring
SSL Soil Screening Level
PRG Preliminary Remediation Goal

GW Groundwater
C Carcinogen
NC Noncarcinogen
Ing. Ingestion
Inh. Inhalation
sat Soil saturation concentration
max Ceiling limit
-- There is no criterion for this particular analyte.

The specific criterion/standard is shaded and italicized if it is exceed by the QL but not by the MDL. The criterion/standard is shaded (without italics) if it is exceeded by both the QL and the MDL.

1 - The lowest criterion for each analyte represents the PAL, presented in Worksheet #15.
2 - Oak Ridge National Laboratory (ORNL) Regional Screening Levels (RSLs) for Chemical Contaminants at Superfund Sites, Residential Soil, July 7, 2008 (available online at http://epa-prgs.ornl.gov/chemicals/index.shtml). One-tenth values are displayed for noncarcinogenic compounds.
3 - USEPA Soil Screening Levels. USEPA Internet Site at http://risk.lsd.ornl.gov/calc_start.htm.
4 - Maine, May 1997. Draft Maine Remedial Action Guidelines for Contaminated Soil.
5 - Acenaphthene is used as a surrogate.
6 - Pyrene is used as a surrogate.
7 - Chlordane is used as a surrogate.
8 - Alpha-BHC is used as a surrogate.
9 - Endosulfan is used as a surrogate.

10 - Endrin is used as a surrogate.
11 - Value is for hexavalent chromium.
12 – Saturation concentration is lower than the screening level.
13 - Sources (soil):
Eco SSL = USEPA Ecological Soil Screening Levels, August 2003. Individual Eco-SSL documents available at http://www.epa.gov/ecotox/ecossl
CCME = CCME, 2006. Recommended Canadian Soil Quality Guidelines. Canadian Council of Ministers of the Environment. Ottawa, Ontario. November.
ORNL Plant = Efroymson, R.A., M.E. Will, G.W. Suter II, and A.C. Wooten. 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision. Oak Ridge National Laboratory. November. ES/ER/TM-85/R3 and ES/ER/TM-126/R2.
ORNL Invert = Efroymson, R.A., M.E. Will, and G.W. Suter II. 1997. Toxicological Benchmarks for Contaminants of Potential Concern for Effects on
Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision. Oak Ridge National Laboratory. November. ES/ER/TM- 126/R2.
TV = MHSPE, 2000. Circular on target values and intervention values for soil remediation. Ministry of Housing, Spatial Planning, and Environment.
DBO/1999226863. Department of Soil Protection, The Netherlands. February 4.
14 - Total value used.
15 - Gamma-BHC is used as a surrogate.
16 - pH- Aluminum is only considered a COPC when the soil pH is less than 5.5; Iron is only considered a COPC when the soil pH is less than 5.0 or greater than 8.
17 – The value for vanadium and compounds is presented.

SAP Worksheet #15b -- Reference Limits and Evaluation Table, Sediment Samples

(UFP-QAPP Manual Section 2.8.1)

Matrix: Sediment

Analytical Group:

Analyte	CAS Number	PAL[1,2] (mg/Kg)	Analytical Method	PQL Goal (mg/Kg)	Laboratory-specific	
					QLs (mg/Kg)	MDLs (mg/Kg)
PAHs						
2-Methylnaphthalene	91-57-6	0.02	SW846 8270C SIM	0.020	0.020	0.0028
Acenaphthene	83-32-9	0.29	SW846 8270C SIM	0.020	0.020	0.0015
Acenaphthylene	208-96-8	0.16	SW846 8270C SIM	0.020	0.020	0.0013
Anthracene	120-12-7	0.0572	SW846 8270C SIM	0.020	0.020	0.0026
Benzo(a)anthracene	56-55-3	0.108	SW846 8270C SIM	0.020	0.020	0.0025
Benzo(a)pyrene	50-32-8	0.15	SW846 8270C SIM	0.020	0.020	0.0021
Benzo(b)fluoranthene	205-99-2	1.5	SW846 8270C SIM	0.33	0.020	0.0022
Benzo(g,h,i)perylene	191-24-2	0.17	SW846 8270C SIM	0.020	0.020	0.0033
Benzo(k)fluoranthene	207-08-9	0.24	SW846 8270C SIM	0.020	0.020	0.0019
Chrysene	218-01-9	0.166	SW846 8270C SIM	0.020	0.020	0.0026
Dibenzo(a,h)anthracene	53-70-3	0.033	SW846 8270C SIM	0.020	0.020	0.0038
Fluoranthene	206-44-0	0.423	SW846 8270C SIM	0.33	0.020	0.0018
Fluorene	86-73-7	0.0774	SW846 8270C SIM	0.020	0.020	0.0016
Indeno(1,2,3-c,d)pyrene	193-39-5	0.2	SW846 8270C SIM	0.020	0.020	0.0043

Analyte	CAS Number	PAL[1,2] (mg/Kg)	Analytical Method	PQL Goal (mg/Kg)	Laboratory-specific	
					QLs (mg/Kg)	MDLs (mg/Kg)
Naphthalene	91-20-3	0.176	SW846 8270C SIM	0.020	0.020	0.0018
Phenanthrene	85-01-8	0.204	SW846 8270C SIM	0.020	0.020	0.0043
Pyrene	129-00-0	0.195	SW846 8270C SIM	0.020	0.020	0.0069
Pesticides						
4,4' DDD	72-54-8	0.00488	SW845 8081A	0.0033	0.0033	0.00071
4,4'-DDE	72-55-9	*0.00316*	SW845 8081A	0.0033	0.0033	0.00070
4,4'-DDT	50-29-3	0.00416	SW845 8081A	0.0033	0.0033	0.00082
Aldrin	309-00-2	0.002	SW845 8081A	0.0017	0.0017	0.00068
alpha-BHC	319-84-6	0.006	SW845 8081A	0.0017	0.0017	0.00071
alpha-Chlordane	5103-71-9	3.24	SW845 8081A	0.0017	0.0017	0.00091
beta-BHC	319-85-7	0.005	SW845 8081A	0.0017	0.0017	0.00076
delta-BHC	319-86-8	0.005	SW845 8081A	0.0017	0.0017	0.00097
Dieldrin	60-57-1	*0.0019*	SW845 8081A	0.0033	0.0033	0.00069
Endosulfan I	959-98-8	0.0055	SW845 8081A	0.0017	0.0017	0.00072
Endosulfan II	33213-65-9	0.0055	SW845 8081A	0.0033	0.0033	0.00069
Endosulfan sulfate	1031-07-8	0.0055	SW845 8081A	0.0033	0.0033	0.00080
Endrin	72-20-8	*0.00222*	SW845 8081A	0.0033	0.0033	0.00077
Endrin aldehyde	7421-93-4	*0.00222*	SW845 8081A	0.0033	0.0033	0.00091
Endrin ketone	53494-70-5	*0.00222*	SW845 8081A	0.0033	0.0033	0.00077
gamma-BHC (Lindane)	58-89-9	0.00237	SW845 8081A	0.0017	0.0017	0.00073
gamma-Chlordane	5103-74-2	0.00324	SW845 8081A	0.0017	0.0017	0.00082

Analyte	CAS Number	PAL[1,2] (mg/Kg)	Analytical Method	PQL Goal (mg/Kg)	Laboratory-specific	
					QLs (mg/Kg)	MDLs (mg/Kg)
Heptachlor	76-44-8	0.068	SW845 8081A	0.0017	0.0017	0.00082
Heptachlor epoxide	1024-57-3	0.00247	SW845 8081A	0.0017	0.0017	0.00076
Methoxychlor	72-43-5	*0.019*	SW845 8081A	0.017	0.017	0.0009
Toxaphene	8001-35-2	*0.028*	SW845 8081A	0.033	0.033	0.022
Herbicides						
2,4-D	94-75-7	690	SW846 8151A	0.033	0.033	0.0070
2,4-DB	94-82-6	490	SW846 8151A	0.033	0.033	0.016
2,4,5-TP (Silvex)	93-72-1	490	SW846 8151A	0.033	0.033	0.0048
2,4,5-T	93-76-5	610	SW846 8151A	0.033	0.033	0.0058
Dalapon	75-99-0	1800	SW846 8151A	0.17	0.17	0.0114
Dicamba	1918-00-9	1800	SW846 8151A	0.033	0.033	0.0059
Dichloroprop	120-36-5	--	SW846 8151A	0.067	0.067	0.0046
Dinoseb	88-85-7	61	SW846 8151A	0.17	0.17	0.0097
MCPA	94-74-6	31	SW846 8151A	5	5	0.960
MCPP	93-65-2	61	SW846 8151A	3.3	3.3	1.07
Metals						
Aluminum	7429-90-5	25500	SW846 6010B	30	30	2.21
Antimony	7440-36-0	3	SW846 6010B	1	0.8	0.0562
Arsenic	7440-38-2	3.9	SW846 6010B	1	0.8	0.265
Barium	7440-39-3	48	SW846 6010B	10	0.5	0.0312
Beryllium	7440-41-7	160	SW846 6010B	0.5	0.5	0.0124

Analyte	CAS Number	PAL[1,2] (mg/Kg)	Analytical Method	PQL Goal (mg/Kg)	Laboratory-specific	
					QLs (mg/Kg)	MDLs (mg/Kg)
Cadmium	7440-43-9	0.99	SW846 6020	0.5	0.1	0.00639
Calcium	7440-70-2	--	SW846 6010B	500	5.0	2.96
Chromium	7440-47-3	43.4	SW846 6010B	4	1.5	0.0461
Cobalt	7440-48-4	50	SW846 6010B	5	3.0	0.0273
Copper	7440-50-8	31.6	SW846 6010B	2.5	2.5	0.133
Iron	7439-89-6	20000	SW846 6010B	10	10	0.611
Lead	7439-92-1	35.8	SW846 6010B	1	0.5	0.122
Magnesium	7439-95-4	--	SW846 6010B	500	5.0	1.08
Manganese	7439-96-5	460	SW846 6010B	5	0.5	0.196
Mercury	7439-97-6	0.18	SW846 7471A	0.04	0.04	0.0034
Nickel	7440-02-0	22.7	SW846 6010B	4	4.0	0.0131
Potassium	7440-09-7	--	SW846 6010B	500	100	3.71
Selenium	7782-49-2	1	SW846 6020	0.5	0.5	0.0640
Silver	7440-22-4	0.5	SW846 6020	0.2	0.1	0.0145
Sodium	7440-23-5	--	SW846 6010B	500	100	2.21
Thallium	7440-28-0	5.1	SW846 6010B	2.5	1.5	0.0638
Vanadium	7440-62-2	57	SW846 6010B	5	2.5	0.0316
Zinc	7440-66-6	121	SW846 6010B	6	2.5	0.171
TOC	--	--	SW846 9060A		400	85

Notes:

1 – The PAL represents the lowest risk-based or regulatory criterion applicable for the project. References (criteria and sources) used to determine PALs are provided below in Table 15a. The lowest criterion for each analyte is shown in **bold font** in Table 15b.

2 - The *shading and italicizing* of a PAL indicates that laboratory-specific QL exceeds the action limit using the analytical methods conventionally available (and specifically the methods cited in this table), but the MDL is less than the PAL. The laboratory will evaluate results down to the MDL, and will report positive detections between the QL and MDL as estimated due to uncertainty of quantitation below the QL. The shading (without italics) of a PAL indicates that the laboratory-specific QL and MDL exceed the action limit using the analytical methods conventionally available (and specifically the methods cited in this table). Because site investigations at NASB frequently involve the use of analytical methods with quantitation and DLs comparable to those listed in this table, more sensitive methods were not sought for this project. If, however, a specific project would require more sensitive analytical methods, additional background data may need to be collected for that project.

Abbreviations:

-- - Not available

TABLE 15B – DETERMINATION OF PALS, SEDIMENT SAMPLES

Analyte	CAS Number	Human Health Sediment Screening Level[2] (mg/kg)	Ecological Sediment Screening Level (mg/kg)[11]	Source of Ecological Sediment Screening Value	Lowest Criterion[1] (mg/kg)
PAHs					--
2-Methylnaphthalene	91-57-6	310	**0.0202** [12]	NOAA	0.0202
Acenaphthene	83-32-9	3400	**0.29**	NOAA	0.29
Acenaphthylene	208-96-8	3400 [3]	**0.16**	NOAA	0.16
Anthracene	120-12-7	17000	**0.0572**	TEC	0.0572
Benzo(a)anthracene	56-55-3	1.5	**0.108**	TEC	0.108
Benzo(a)pyrene	50-32-8	0.15	**0.15**	TEC	0.15
Benzo(b)fluoranthene	205-99-2	**1.5**	1.8 [13]	NOAA	1.5
Benzo(g,h,i)perylene	191-24-2	1700 [4]	**0.17**	OMOE	0.17
Benzo(k)fluoranthene	207-08-9	15	**0.24**	OMOE	0.24
Chrysene	218-01-9	150	**0.166**	TEC	0.166
Dibenzo(a,h)anthracene	53-70-3	0.15	**0.033**	TEC	0.033
Fluoranthene	206-44-0	2300	**0.423**	TEC	0.423
Fluorene	86-73-7	2300	**0.0774**	TEC	0.0774
Indeno(1,2,3-c,d)pyrene	193-39-5	1.5	**0.2**	OMOE	0.2
Naphthalene	91-20-3	39	**0.176**	TEC	0.176
Phenanthrene	85-01-8	1700 [4]	**0.204**	TEC	0.204
Pyrene	129-00-0	1700	**0.195**	TEC	0.195

Analyte	CAS Number	Human Health Sediment Screening Level[2] (mg/kg)	Ecological Sediment Screening Level (mg/kg)[11]	Source of Ecological Sediment Screening Value	Lowest Criterion[1] (mg/kg)
Pesticides					
4,4' DDD	72-54-8	20	**0.00488**	TEC	0.00488
4,4'-DDE	72-55-9	14	***0.00316***	TEC	0.00316
4,4'-DDT	50-29-3	17	**0.00416**	TEC	0.00416
Aldrin	309-00-2	0.29	**0.002**	OMOE	0.002
alpha-BHC	319-84-6	0.77	**0.006**	OMOE	0.006
alpha-Chlordane	5103-71-9	16 [5]	**3.24**	TEC	3.24
beta-BHC	319-85-7	2.7	**0.005**	OMOE	0.005
delta-BHC	319-86-8	0.77 [6]	**0.005** [14]	OMOE	0.005
Dieldrin	60-57-1	0.3	***0.0019***	TEC	0.0019
Endosulfan I	959-98-8	370 [7]	**0.0055** [15]	SCV	0.0055
Endosulfan II	33213-65-9	370 [7]	**0.0055** [15]	SCV	0.0055
Endosulfan sulfate	1031-07-8	370 [7]	**0.0055** [15]	SCV	0.0055
Endrin	72-20-8	18	***0.00222***	TEC	0.00222
Endrin aldehyde	7421-93-4	18 [8]	***0.00222*** [16]	TEC	0.00222
Endrin ketone	53494-70-5	18 [8]	***0.00222*** [16]	TEC	0.00222
gamma-BHC (Lindane)	58-89-9	5.2	**0.00237**	TEC	0.00237
gamma-Chlordane	5103-74-2	16 [5]	**0.00324**	TEC	0.00324
Heptachlor	76-44-8	1.1	**0.068**	SCV	0.068
Heptachlor epoxide	1024-57-3	0.53	**0.00247**	TEC	0.00247

Analyte	CAS Number	Human Health Sediment Screening Level[2] (mg/kg)	Ecological Sediment Screening Level (mg/kg)[11]	Source of Ecological Sediment Screening Value	Lowest Criterion[1] (mg/kg)
Methoxychlor	72-43-5	310	***0.019***	SCV	0.019
Toxaphene	8001-35-2	4.4	***0.028***	EcoTox	0.028
Herbicides					
2,4-D	94-75-7	**690**	--	--	690
2,4-DB	94-82-6	**490**	--	--	490
2,4,5-TP (Silvex)	93-72-1	**490**	--	--	490
2,4,5-T	93-76-5	**610**	--	--	610
Dalapon	75-99-0	**1800**	--	--	1800
Dicamba	1918-00-9	**1800**	--	--	1800
Dichloroprop	120-36-5	**--**	--	--	--
Dinoseb	88-85-7	**61**	--	--	61
MCPA	94-74-6	**31**	--	--	31
MCPP	93-65-2	**61**	--	--	61
Metals					
Aluminum	7429-90-5	77000	**25500** [17]	NOAA	25500
Antimony	7440-36-0	31	**3**	NOAA	3
Arsenic	7440-38-2	**3.9**	9.79	TEC	3.9
Barium	7440-39-3	15000	**48** [13]	NOAA	48
Beryllium	7440-41-7	**160**	--	--	160
Cadmium	7440-43-9	70	**0.99**	TEC	0.99

Analyte	CAS Number	Human Health Sediment Screening Level[2] (mg/kg)	Ecological Sediment Screening Level (mg/kg)[11]	Source of Ecological Sediment Screening Value	Lowest Criterion[1] (mg/kg)
Calcium	7440-70-2	--	--	--	--
Chromium	7440-47-3	230 [9]	**43.4**	TEC	43.4
Cobalt	7440-48-4	--	**50**	OMOE	50
Copper	7440-50-8	3100	**31.6**	OMOE	31.6
Iron	7439-89-6	55000	20000	OMOE	20000
Lead	7439-92-1	400	**35.8**	TEC	35.8
Magnesium	7439-95-4	--	--	--	--
Manganese	7439-96-5	1800	**460**	OMOE	460
Mercury	7439-97-6	6.7 [10]	**0.18**	TEC	0.18
Nickel	7440-02-0	1600	**22.7**	TEC	22.7
Potassium	7440-09-7	--	--	--	--
Selenium	7782-49-2	390	**1** [13]	NOAA	1
Silver	7440-22-4	390	**0.5**	OMOE	0.5
Sodium	7440-23-5	--	--	--	--
Thallium	7440-28-0	**5.1**	--	--	5.1
Vanadium	7440-62-2	390 [18]	**57** [13]	NOAA	57
Zinc	7440-66-6	23000	**121**	TEC	121

Notes:
DAF Dilution Attenuation Factor
TCL Target Compound List

TAL Target Analyte List
PAH Polycyclic Aromatic Hydrocarbons
SIM Selective Ion Monitoring
PRG Preliminary Remediation Goal

The specific criterion/standard is shaded and italicized if it is exceed by the QL but not by the MDL. The criterion/standard is shaded (without italics) if it is exceeded by both the QL and the MDL.

1 - The lowest criterion for each analyte represents the PAL, presented in Worksheet #15.
2 - Ten times the ORNL RSLs for Chemical Contaminants at Superfund Sites, Residential Soil, July 7, 2008 (available online at http://epa-prgs.ornl.gov/chemicals/index.shtml) is presented. One-tenth values (one-tenth of the ORNL value, times ten) are displayed for noncarcinogenic compounds.
3 - Acenaphthene is used as a surrogate.
4 - Pyrene is used as a surrogate.
5 - Chlordane is used as a surrogate.
6 - Alpha-BHC is used as a surrogate.
7 - Endosulfan is used as a surrogate.
8 - Endrin is used as a surrogate.
9 - Value is for hexavalent chromium.
10 – Saturation concentration is lower than the screening level.
11 - Sources:

TEC = MacDonald, D.D., C.G. Ingersoll and T.A. Berger. 2000. Development and Evaluation of Consensus-Based Sediment Quality Guidelines for Freshwater Ecosystems. Archives of Environmental Contamination and Toxicology. 39, 20-31. January.
OMOE = Persaud, D., R. Jaagumagi, and A. Hayton. 1993. Guidelines for the Protection and Management of Aquatic Sediment Quality in Ontario. Ministry of Environment and Energy. August.
EcoTox = USEPA, 1996. ECO Update, Ecotox Thresholds. U.S. EPA. Office of Solid Waste and Emergency Response. Intermittent Bulletin, Volume 3, Number 2. EPA540/F-95/038. January.
NOAA = Buchman, M.F., 1999. NOAA Screening Quick Reference Tables, NOAA HAZMAT Report 99-1, Seattle, WA, Coastal Protection and Restoration Division, National Oceanic and Atmospheric Administration.
http://response.restoration.noaa.gov/cpr/sediment/squirt/squirt.html

SCV = Jones, D.S., G.W. Suter II, and R.N. Hull. 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Sediment-Associated Biota: 1997 Revision. Oak Ridge National Laboratory. November. ES/ER/TM-95/R4.
12 - Saltwater TEL value.
13 - Saltwater AET value.

14 - LEL for beta-BHC.
15 - SCV for Endosulfan (all isomers).
16 - TEC for Endrin.
17 - Lowest ARCs H. azteca TEL.
18 – The value for vanadium and compounds is presented.

SAP Worksheet #15c -- Reference Limits and Evaluation Table, Groundwater Samples

(UFP-QAPP Manual Section 2.8.1)

Matrix: Groundwater

Analytical Group:

Analyte	CAS Number	PAL[1,2] (µg/L)	Analytical Method	PQL Goal (µg/L)	Laboratory-specific	
					QLs (µg/L)	MDLs (µg/L)
Metals (Total and Dissolved)						
Aluminum	7429-90-5	1430	SW846 6020	200	50	7.21
Antimony	7440-36-0	1.5	SW846 6020	1	1.0	0.12
Arsenic	7440-38-2	0.045	SW846 6020	5	5.0	1.47
Barium	7440-39-3	730	SW846 6010B	200	5.0	0.593
Beryllium	7440-41-7	7.3	SW846 6020	1	1.0	0.0565
Cadmium	7440-43-9	1.8	SW846 6020	1	1.0	0.0925
Calcium	7440-70-2	--	SW846 6010B	5000	50	29.8
Chromium	7440-47-3	11	SW846 6020	3	3.0	0.575
Cobalt	7440-48-4	--	SW846 6020	1	1.0	0.124
Copper	7440-50-8	150	SW846 6010B	30	25	0.754
Iron	7439-89-6	2600	SW846 6010B	100	100	8.96
Lead	7439-92-1	10	SW846 6020	1	1.0	0.112
Magnesium	7439-95-4	--	SW846 6010B	5000	50	4.33
Manganese	7439-96-5	88	SW846 6010B	15	5.0	1.09
Mercury	7439-97-6	*0.063*	SW846 7470A	0.2	0.20	0.061
Nickel	7440-02-0	73	SW846 6020	10	1.0	0.120

Analyte	CAS Number	PAL[1,2] (µg/L)	Analytical Method	PQL Goal (µg/L)	Laboratory-specific	
					QLs (µg/L)	MDLs (µg/L)
Potassium	7440-09-7	--	SW846 6010B	5000	1000	40.9
Selenium	7782-49-2	18	SW846 6020	5	5.0	0.90
Silver	7440-22-4	18	SW846 6020	1	1.0	0.065
Sodium	7440-23-5	20000	SW846 6010B	5000	1000	15.0
Thallium	7440-28-0	0.24	SW846 6020	2	2.0	0.62
Vanadium	7440-62-2	18	SW846 6020	5	5.0	0.58
Zinc	7440-66-6	1100	SW846 6020	20	10	0.905
Anions						
Bromide	--	--	EPA Method 300.0	2000	2000	99.3
Chloride	--	--	EPA Method 300.0	500	500	136

Notes:

1 – The PAL represents the lowest risk-based or regulatory criterion applicable for the project. References (criteria and sources) used to determine PALs are provided below in Table 15a. The lowest criterion for each analyte is shown in **bold font** in Table 15c.

2 - The *shading and italicizing* of a PAL indicates that laboratory-specific QL exceeds the action limit using the analytical methods conventionally available (and specifically the methods cited in this table), but the MDL is less than the PAL. The laboratory will evaluate results down to the MDL, and will report positive detections between the QL and MDL as estimated due to uncertainty of quantitation below the QL. In the case of arsenic, the metal typically occurs naturally at concentrations greater than the indicated QL and PAL, and the arsenic QL is within these background concentrations so the PAL exceedance is not a concern. The shading (without italics) of a PAL indicates that the laboratory-specific QL and MDL exceed the action limit using the analytical methods conventionally available (and specifically the methods cited in this table). Because site investigations at NASB frequently involve the use of analytical methods with quantitation and DLs comparable to those listed in this table, more sensitive methods were not sought for this project. If, however, a specific project would require more sensitive analytical methods, additional background data may need to be collected for that project.

Abbreviations:

-- - Not available

TABLE 15C – DETERMINATION OF PALS, GROUNDWATER SAMPLES

Chemical	CAS Number	ORNL Regional Screening Levels, Tap Water[2] (µg/L)	2006 Maine Maximum Exposure Guidelines for Drinking Water [3] (µg/L)	MEG Status[3]	EPA Volatilization Criteria[4] (µg/L)	Lowest Criterion[1] (µg/L)
Metals (Total and Dissolved)						
Aluminum	7429-90-5	3700	**1430**	Interim	NA	1430
Antimony	7440-36-0	**1.5**	3	Final	NA	1.5
Arsenic	7440-38-2	**0.045**	10	Interim	NA	0.045
Barium	7440-39-3	**730**	2000	Final	NA	730
Beryllium	7440-41-7	**7.3**	NA	NA	NA	7.3
Cadmium	7440-43-9	**1.8**	3.5	Final	NA	1.8
Calcium	7440-70-2	--	NA	NA	NA	NA
Chromium	7440-47-3	**11** [5]	40	Final	NA	11
Cobalt	7440-48-4	**--**	NA	NA	NA	NA
Copper	7440-50-8	**150**	1300	Final	NA	150
Iron	7439-89-6	**2600**	NA	NA	NA	2600
Lead	7439-92-1	**15** [6]	10	Interim	NA	10
Magnesium	7439-95-4	--	NA	NA	NA	NA
Manganese	7439-96-5	**88**	500	Final	NA	88
Mercury	7439-97-6	***0.063***	2	Final	0.68	0.063
Nickel	7440-02-0	**73**	140	Final	NA	73
Potassium	7440-09-7	--	NA	NA	NA	NA

Chemical	CAS Number	ORNL Regional Screening Levels, Tap Water[2] (µg/L)	2006 Maine Maximum Exposure Guidelines for Drinking Water [3] (µg/L)	MEG Status[3]	EPA Volatilization Criteria[4] (µg/L)	Lowest Criterion[1] (µg/L)
Selenium	7782-49-2	**18**	35	Final	NA	18
Silver	7440-22-4	**18**	35	Final	NA	18
Sodium	7440-23-5	--	**20000**	Interim	NA	20000
Thallium	7440-28-0	**0.24**	0.5	Final	NA	0.24
Vanadium	7440-62-2	**18** [7]	NA	NA	NA	18
Zinc	7440-66-6	**1100**	2000	Final	NA	1100

Notes:
TCL = Target Compound List

The specific criterion/standard is shaded and italicized if it is exceed by the QL but not by the MDL. The criterion/standard is shaded (without italics) if it is exceeded by both the QL and the MDL.

1 – The lowest criterion for each analyte represents the PAL presented in Worksheet 15.
2 - ORNL RSLs for Chemical Contaminants at Superfund Sites, Tapwater, July 7, 2008 (available online at http://epa-prgs.ornl.gov/chemicals/index.shtml). One-tenth values are displayed for noncarcinogenic compounds.
3 - Maine CDC, August 7, 2006. Maximum Exposure Guidelines (MEGs) for Drinking Water.
4 - Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils. November 2002. EPA530-F-02-052.
5 - The value for hexavalent chromium is presented.
6 – USEPA Safe Drinking Water Action Level.
7 – The value for vanadium and compounds is presented.

SAP Worksheet #15d -- Reference Limits and Evaluation Table, Surface Water Samples

(UFP-QAPP Manual Section 2.8.1)

Matrix: Surface Water

Analytical Group:

Analyte	CAS Number	PAL[1] (µg/L)	Analytical Method	PQL Goal (µg/L)	Laboratory-specific	
					QLs (µg/L)	MDLs (µg/L)
PAHs						
2-Methylnaphthalene	91-57-6	72.16	SW846 8270C SIM	0.2	0.2	0.035
Acenaphthene	83-32-9	20	SW846 8270C SIM	0.2	0.2	0.028
Acenaphthylene	208-96-8	307	SW846 8270C SIM	0.2	0.2	0.018
Anthracene	120-12-7	20.73	SW846 8270C SIM	0.2	0.2	0.035
Benzo(a)anthracene	56-55-3	0.0038	SW846 8270C SIM	0.2	0.2	0.043
Benzo(a)pyrene	50-32-8	0.0038	SW846 8270C SIM	0.2	0.2	0.08
Benzo(b)fluoranthene	205-99-2	0.0038	SW846 8270C SIM	0.2	0.2	0.078
Benzo(g,h,i)perylene	191-24-2	0.4391	SW846 8270C SIM	0.2	0.2	0.072
Benzo(k)fluoranthene	207-08-9	0.0038	SW846 8270C SIM	0.2	0.2	0.051
Chrysene	218-01-9	0.0038	SW846 8270C SIM	0.2	0.2	0.093
Dibenzo(a,h)anthracene	53-70-3	0.0038	SW846 8270C SIM	0.2	0.2	0.11
Fluoranthene	206-44-0	7.109	SW846 8270C SIM	0.2	0.2	0.071
Fluorene	86-73-7	39.3	SW846 8270C SIM	0.2	0.2	0.033
Indeno(1,2,3-c,d)pyrene	193-39-5	0.0038	SW846 8270C SIM	0.2	0.2	0.085

Analyte	CAS Number	PAL[1] (µg/L)	Analytical Method	PQL Goal (µg/L)	Laboratory-specific	
					QLs (µg/L)	MDLs (µg/L)
Naphthalene	91-20-3	1.4	SW846 8270C SIM	0.2	0.2	0.049
Phenanthrene	85-01-8	19.13	SW846 8270C SIM	0.2	0.2	0.038
Pyrene	129-00-0	10.11	SW846 8270C SIM	0.2	0.2	0.11
Metals (Total and Dissolved)						
Aluminum	7429-90-5	87	SW846 6020	50	50	7.21
Antimony	7440-36-0	5.6	SW846 6020	1	1.0	0.12
Arsenic	7440-38-2	0.018	SW846 6020	5	5.0	1.47
Barium	7440-39-3	4	SW846 6020	1	1.0	0.593
Beryllium	7440-41-7	*0.66*	SW846 6020	1	1.0	0.0565
Cadmium	7440-43-9	*0.25*	SW846 6020	1	1.0	0.0925
Calcium	7440-70-2	--	SW846 6010B	5000	50	29.8
Chromium	7440-47-3	74	SW846 6020	10	3.0	0.575
Cobalt	7440-48-4	23	SW846 6020	1	1.0	0.124
Copper	7440-50-8	9	SW846 6020	1	1.0	0.754
Iron	7439-89-6	300	SW846 6010B	100	100	8.96
Lead	7439-92-1	2.5	SW846 6020	1	1.0	0.112
Magnesium	7439-95-4	--	SW846 6010B	5000	50	4.33
Manganese	7439-96-5	50	SW846 6010B	10	5.0	1.09
Mercury	7439-97-6	0.63	SW846 7470A	0.2	0.2	0.061
Nickel	7440-02-0	52	SW846 6020	10	1.0	0.120
Potassium	7440-09-7	--	SW846 6010B	5000	1000	40.9

Analyte	CAS Number	PAL[1] (µg/L)	Analytical Method	PQL Goal (µg/L)	Laboratory-specific	
					QLs (µg/L)	MDLs (µg/L)
Selenium	7782-49-2	5	SW846 6020	5	5.0	0.90
Silver	7440-22-4	*0.36*	SW846 6020	1	1.0	0.065
Sodium	7440-23-5	--	SW846 6010B	5000	1000	15.0
Thallium	7440-28-0	0.24	SW846 6020	2	2.0	0.62
Vanadium	7440-62-2	20	SW846 6020	5	5.0	0.58
Zinc	7440-66-6	120	SW846 6020	20	10	0.905

Notes:

1 – The PAL represents the lowest risk-based or regulatory criterion applicable for the project. References (criteria and sources) used to determine PALs are provided below in Table 15a. The lowest criterion for each analyte is shown in **bold font** in Table 15d.

2 - The *shading and italicizing* of a PAL indicates that laboratory-specific QL exceeds the action limit using the analytical methods conventionally available (and specifically the methods cited in this table), but the MDL is less than the PAL. The laboratory will evaluate results down to the MDL, and will report positive detections between the QL and MDL as estimated due to uncertainty of quantitation below the QL. In the case of arsenic, the metal typically occurs naturally at concentrations greater than the indicated QL and PAL, and the arsenic QL is within these background concentrations so the PAL exceedance is not a concern. The shading (without italics) of a PAL indicates that the laboratory-specific QL and MDL exceed the action limit using the analytical methods conventionally available (and specifically the methods cited in this table). Because site investigations at NASB frequently involve the use of analytical methods with quantitation and DLs comparable to those listed in this table, more sensitive methods were not sought for this project. If, however, a specific project would require more sensitive analytical methods, additional background data may need to be collected for that project.

Abbreviations:

-- - Not available

TABLE 15D – DETERMINATION OF PALS, SURFACE WATER SAMPLES

Chemical	CAS Number	Human Health Surface Water Screening Level[2] (µg/L)	National Recommended Water Quality Criteria[3] (µg/L)		Freshwater Ecological Screening Level[4] (µg/L)	Source of Ecological Screening Level	Lowest Criterion[1] (µg/L)
			Water and Organisms	Organisms Only			
PAHs							
2-Methylnaphthalene	91-57-6	150	NA	NA	**72.16**	FCV	72.16
Acenaphthene	83-32-9	2200	**20**	20	55.85	FCV	20
Acenaphthylene	208-96-8	2200 [5]	NA	NA	**306.9**	FCV	306.9
Anthracene	120-12-7	11000	8300	40000	**20.73**	FCV	20.73
Benzo(a)anthracene	56-55-3	0.29	**0.0038**	0.018	2.227	FCV	0.0038
Benzo(a)pyrene	50-32-8	0.029	**0.0038**	0.018	0.9573	FCV	0.0038
Benzo(b)fluoranthene	205-99-2	0.29	**0.0038**	0.018	0.6774	FCV	0.0038
Benzo(g,h,i)perylene	191-24-2	1100 [6]	NA	NA	**0.4391**	FCV	0.4391
Benzo(k)fluoranthene	207-08-9	2.9	**0.0038**	0.018	0.6415	FCV	0.0038
Chrysene	218-01-9	29	**0.0038**	0.018	2.042	FCV	0.0038
Dibenzo(a,h)anthracene	53-70-3	0.029	**0.0038**	0.018	0.2825	FCV	0.0038
Fluoranthene	206-44-0	1500	130	140	**7.109**	FCV	7.109
Fluorene	86-73-7	1500	1100	5300	**39.3**	FCV	39.3
Indeno(1,2,3-c,d)pyrene	193-39-5	0.29	**0.0038**	0.018	0.275	FCV	0.0038
Naphthalene	91-20-3	**1.4**	NA	NA	193.5	FCV	1.4
Phenanthrene	85-01-8	1100 [6]	NA	NA	**19.13**	FCV	19.13
Pyrene	129-00-0	1100	830	4000	**10.11**	FCV	10.11
Metals (Total and Dissolved)							
Aluminum	7429-90-5	37000	NA	NA	**87**	NRWQC	87

Chemical	CAS Number	Human Health Surface Water Screening Level[2] (µg/L)	National Recommended Water Quality Criteria[3] (µg/L)		Freshwater Ecological Screening Level[4] (µg/L)	Source of Ecological Screening Level	Lowest Criterion[1] (µg/L)
			Water and Organisms	Organisms Only			
Antimony	7440-36-0	15	**5.6**	640	30	SCV	5.6
Arsenic	7440-38-2	0.45	**0.018**	0.14	150	NRWQC	0.018
Barium	7440-39-3	7300	1000	NA	**4**	SCV	4
Beryllium	7440-41-7	73	NA	NA	***0.66***	SCV	0.66
Cadmium	7440-43-9	18	NA	NA	***0.25***	NRWQC	0.25
Calcium	7440-70-2	--	NA	NA	--	--	--
Chromium	7440-47-3	110 [7]	NA	NA	**74**	NRWQC	74
Cobalt	7440-48-4	--	NA	NA	**23**	SCV	23
Copper	7440-50-8	1500	1000	NA	**9**	NRWQC	9
Iron	7439-89-6	26000	**300**	NA	1000	NRWQC	300
Lead	7439-92-1	15 [8]	NA	NA	**2.5**	NRWQC	2.5
Magnesium	7439-95-4	--	NA	NA	--	--	--
Manganese	7439-96-5	880	**50**	100	120	SCV	50
Mercury	7439-97-6	**0.63**	NA	NA	0.77	NRWQC	0.63
Nickel	7440-02-0	730	610	4600	**52**	NRWQC	52
Potassium	7440-09-7	--	NA	NA	--	--	--
Selenium	7782-49-2	180	170	4200	**5**	NRWQC	5
Silver	7440-22-4	180	NA	NA	***0.36***	SCV	0.36
Sodium	7440-23-5	--	NA	NA	--	--	--
Thallium	7440-28-0	**2.4**	0.24	0.47	4 [10]	NOAA	0.24
Vanadium	7440-62-2	180 [9]	NA	NA	**20**	SCV	20
Zinc	7440-66-6	11000	5000	5000	**120**	NRWQC	120

Notes:

TCL = Target Compound List

PAH = Polycyclic Aromatic Hydrocarbons

The specific criterion/standard is shaded and italicized if it is exceed by the QL but not by the MDL. The criterion/standard is shaded (without italics) if it is exceeded by both the QL and the MDL.

1 - The lowest criterion for each analyte represents the PAL presented in Worksheet 15.
2 - Ten times the ORNL RSLs for Chemical Contaminants at Superfund Sites, Tapwater, July 7, 2008 (available online at http://epa-prgs.ornl.gov/chemicals/index.shtml) is presented. One-tenth values (one-tenth of the ORNL value, times ten) are displayed for noncarcinogenic compounds.

3 - National Recommended Water Quality Criteria. 2006.

4 - The following presents the order of preference that was used for selecting the ecological surface water screening values:

- USEPA (2003) FCV (Final Chronic Value)
- USEPA National Recommended Water Quality Criteria, they are the most current values.
- Suter and Tsao (1996) freshwater values (SCV) (They are commonly used as freshwater screening values).
- Freshwater values in Buchman, 1999 (NOAA) (These were selected last because the basis of their development is not as well documented as the other Suter and Tsao (1996) values).

5 - The value for acenaphthene is presented.

6 - The value for pyrene is presented.
7 - The value for hexavalent chromium is presented.
8 – USEPA Safe Drinking Water Action Level
9 – The value for vanadium and compounds is presented.

10 - Calculated by multiplying the chronic LOAEL by 0.1 to estimate a chronic NOAEL

Sources :
FCV (Final Chronic Value) = USEPA, 2003. Procedures for the Derivation of Equilibrium Partitioning Sediment Benchmarks (ESBs) for the Protection of Benthic Organisms: PAH Mixtures. EPA/600/R-02/013. November.

NRWQC = USEPA (U.S. EPA), 2006. National Recommended Water Quality Criteria. Office of Water. EPA 822-R-02-047.
SCV (Secondary Chronic Value) = Suter, G.W. II and C.L. Tsao. 1996. Toxicological Benchmarks for Screening Potential Constituents of Concern for Effects on Aquatic Biota: 1996 Revision.

Environmental Sciences Oak Ridge National Laboratory. ES/ER/TM-96/R2.
NOAA = Buchman, M.F., 1999. NOAA Screening Quick Reference Tables, NOAA HAZMAT Report 99-1, Seattle, WA, Coastal Protection and Restoration Division, National Oceanic and Atmospheric Administration. http://response.restoration.noaa.gov/cpr/sediment/squirt/squirt.html

SAP Worksheet #16 -- Project Schedule / Timeline Table (optional format)

(UFP-QAPP Manual Section 2.8.2)

TASK NAME	START DATE	END DATE
Procurement and Field Mobilization	7/13/09	8/10/09
Soil Sampling, Well Construction/Development	8/10/09	8/28/09
Event 1: Groundwater, Surface Water/Sediment Sampling	8/31/09	9/11/09
Event 1: Data Validation/Evaluation	9/14/09	10/20/09
Event 2: Groundwater, Surface Water/Sediment Sampling	4/12/10	4/23/10
Event 2: Data Validation/Evaluation	4/26/10	5/31/10
Draft Background Study Report	6/1/10	7/31/10
Responses to comments	8/1/10	8/31/10
Final Background Study Report	9/1/10	9/30/10

SAP Worksheet #17 -- Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)

Overall Sampling Design – The sampling design comprises multiple elements focused on estimating chemical concentrations across different types of background, including naturally occurring and anthropogenic. The sampling plan emphasizes on-base rather than off-base background locations but avoids areas within the influence of industrial operation areas or any NAS Brunswick sites. The plan presents general areas representative of the desired background populations. Within these areas, sample locations will be chosen based on field reconnaissance, topography, and field observations. These areas are identified on Figure 10-2 and described in Table 11-1. An objective statistical approach was used (see Worksheet 11.6.4) to estimate the number of samples required to represent each different background subgroup (e.g., Transitional Unit) within the major background areas.

Sample allocation over the background areas will be judgmentally based on the field reconnaissance and project team agreement. The project team will strive not to allow biases to enter into the selection of sample locations and will strive to spread the samples over as large an area of the selected sampling areas as possible. A purely random sample location was not possible because of expected physical barriers, impediments to sampling, and other constraints. Once general areas are agreed to, then specific sample locations in some cases can be selected using a hybrid random orientation grid. For example, once the suitable locations in Area 1 are selected for soil samples in the Upper Sand, the specific locations within that area could be selected randomly. However, soil samples that may be collected in conjunction with monitoring well installation in Area 5, for example, would be selected judgmentally based on the additional goal of triangulating wells to maximize the value of water level data to interpret groundwater flow directions in those areas.

Sample Location Verification –Background Areas for groundwater, surface soil, sediment, and surface water were inspected by the Navy, EPA, MADEP, MRRA, and BACSE representatives (project team) to determine whether they meet the definition of background material:

- It belongs to one of the two natural geologic materials (Upper Sand, Transitional Unit), not all of which are to be sampled for each environmental matrix (surface or subsurface soil, groundwater, surface water, or sediment).
- The areas were selected based on review of historical data, a site visit, and acceptance by the project team.

Note: Sampling locations may be in areas on-Base and off-Base non-point source anthropogenic impacts.

Matrices to be Sampled – The following matrices will be sampled during this project:

- **Surface Soil (0-1 ft bgs)** – collect one sample from ground surface to 1 foot bgs at each surface soil sample location for all analytes.

- **Subsurface Soil (1-10 ft bgs)** – collect one vertical sample from the targeted soil type between 1 ft bgs and a maximum of 10 ft bgs. Based on review of NAS Brunswick boring logs, it is likely that no more than one or two geologic units are present from 1 to 10 feet bgs in any individual boring. If more than one soil unit is represented, the samples will be separated in to unit-specific groupings prior to homogenization of the targeted soil type.

- **Groundwater** – collect groundwater samples from selected existing wells and newly completed wells. A minimum of two weeks shall elapse after well development before collecting groundwater samples. EPA's Low Flow protocol shall be followed when sampling groundwater.

- **Mere Brook Surface Water** – collect one sample of water above the sediment sample location at several tenths of a foot above the sediment surface to be consistent with the Long-Term surface water monitoring program. Sampling must not occur after a significant storm event. That will be evaluated by the Navy consultant before the time of sampling. The surface water sample must be collected prior to collection of the sediment sample. Collect surface water prior to seidment at each location.

- **Coffins Ice Pond Surface Water** – collect one sample of surface water above the sediment sample location at approximately 60% of the water column consistent with the Picnic Pond surface water samples, using the same type of sampler (Kemmerer or equivalent). Collect surface water prior to seidment at each location.

- **Mere Brook and Coffins Ice Pond Sediment (0-6 inches below the sediment surface)** – collect one sediment sample at each sediment sample location from 0 to 6 inches below the sediment surface. The sediment sample locations will be determined based on site reconnaissance, to sample range of sediment-types, and evaluation of prior surface water sampling records. Sampling must not occur after a significant storm event that will be evaluated by the Navy

consultant before the time of sampling. A Wildco Hand Core Sampler (2-in ID by 20-in long) with disposable acetate liner will be used to collect sediment samples in Coffins Ice Pond, the sediment sampler used in Picnic Pond - an impoundment located on-Base.

Sampling Frequency –Surface and subsurface soil will be sampled once. Groundwater wells will be sampled two times, once after a wet period (e.g., April), and once after a dry period (e.g. September) . Surface water and sediment samples will be collected twice at the same time as groundwater.

Sampling Locations – See Figure 10-2 and Figure 17-1 for location. See Table 11-1 and Tables 17-1, 17-2 and17-3 for justification of the sampling locations.

Number and Type of Samples – The estimated number of samples is summarized on Worksheet 18; the rationale for the required number of samples is based on the criteria for achieving a MDD between site and background as explained in Section 11.6.4. Sample collection should include a minimum of 15 samples collected from each groundwater hydrologic regime (Upper Sand, Lower Sand, Transition Unit, and bedrock) in order to be able to evaluate whether these strata exhibit similar concentration profile; a minimum of 15 surface soil and 15 subsurface soil samples from each of the two soil types: Upper Sand, and Transition Unit;, 15 sediment and 15 surface water samples from Mere Brook; 15 sediment and 15 surface water from Coffins Ice Pond. If the textural classifications and organic matter present differ among the sediment sampling locations, then based on ground truthing, 15 samples of each classification should be collected. During the site walkover (April 28-29, 2009), sediment types observed along Mere Brook varied from fine sand to coarse sand and fine gravel with the coarser materials in the stream bed and the finer materials along the sides of the stream. The sediment varied in color from brown to tan. It is thought that the organic content of the Mere Brook sediments is low in organic carbon. In comparison, sediment in Coffins Ice Pond is expected to contain higher organic content relative to Mere Brook because it is expected to be a deposition area for fine grained materials, such as silt, clay and organic matter similar to on-Base impoundments.

Sampling Methods – The sampling SOPs are listed in Worksheet #21 and are included in Appendix A. The sample log sheets are provided in Appendix B.

Water Level Measurements - Groundwater measurements will be obtained using an electronic water level indicator with a weighted cord that is accurate to 0.01 feet. In addition, an oil/water interface probe will be used to identify the potential presence of non-aqueous phase liquid (NAPL) in each well. Samples shall not be collected from any well exhibiting detectable NAPL and the presence of NAPL shall be thoroughly documented in the field notes. Water level measurements in

wells will be recorded from the surveyed measuring point at the top of the inner well casing. Surface water level measurements will be recorded at any surveyed staff gauges. Detailed procedures are in SOP GH-1.2.

- **Surface Soil Sampling (0-1 ft bgs)** – Surface soil of a single predominant soil type will be collected from the first 1 ft interval of soil borings using a decontaminated stainless steel spoon and/or stainless steel hand auger. Mix the entire sample interval vertically. Surface soil samples will be collected from 0-1 ft bgs using a decontaminated stainless steel trowel or spoon in accordance with the methods described in SOP SA-1.3. A summary of the sampling procedures is provided below.

- **Subsurface Soil Sampling (1-10 ft bgs)** – Subsurface soil samples will be collected in accordance with the procedures described in SOP SA-2.5 using a direct push technology (DPT) rig equipped with a dual-tube sampling system to prevent cross-connections during sampling. Soil samples will be mixed within each geologic unit (Upper Sand, Transition Unit). All soil for the 1 to 10 foot interval will be collected before any homogenizing is performed. If more than one soil unit is represented, the samples from the targeted soil unit will be separated and the target soil unit homogenized. Soil sampling procedures are summarized below.

Soil Sample Selection for Laboratory Analysis

The acetate liner will be opened, visually inspected, and scanned with a photo-ionization detector (PID) portable monitoring instrument according to the instrument manufacturer's instructions. Soil samples will be selected based on soil type not PID measurements. The selected interval will be uniformly hand mixed to form a well-mixed sample and split into aliquots for the analyses. If visual, instrumental, or olfactory evidence of contamination is detected, the FOL will consult with the TtNUS Technical lead to determine whether sampling should continue at a particular location. If a sample is collected from a location that field conditions indicate it could be contaminated, the field geologist shall document thoroughly in the field notes why a sample was collected. Any locations identified with high PID reading will be noted in the boring logs and in the report.

Soil Sampling Procedure

1. Record all required data on the boring log, which will also serve as the soil sample log sheet (Appendix B). Include the sampling equipment, sampling personnel, date, time, depth of sample, and sample analyses. The boring log will also contain soil descriptions, depth of strata changes, and sample depth intervals. The soil will be visually classified using the Unified Soil Classification System [American Society for Testing and Materials (ASTM) D-2488-98].

2. Label appropriate sample jars with the sample location number, sampler's name, date, and analytical fractions.

3. Transfer the soil from the split-barrel sampler or acetate to a decontaminated stainless-steel bowl using only decontaminated stainless steel trowels, and homogenize the sample.

4. If there is insufficient sample volume to fill all the containers for the non-VOC analyses, an equal amount of materials from the intervals immediately above and below the selected sample interval may be used to supplement the composite sample to ensure sufficient sample quantity for all analyses. For the 0 to 1 foot depth interval, if additional soil volume is needed at a location, adjacent soils will be sampled at the 0 to1 foot depth interval until sufficient volume is collected.

5. Remove any large particles such as gravel or artificial fill too large to be sent for analysis. Note the removal of material on the boring log.

6. Fill the appropriate sample containers.

7. For field duplicate samples, after homogenization, fill one set of sample containers for the original sample and fill another set for the field duplicate sample.

8. Ensure that the samples are properly labeled, maintained in coolers with ice, and that the chain-of-custody procedures described in Worksheet #27 are followed. Package and ship the sample coolers to the appropriate laboratory for overnight delivery.

9. Decontaminate the sampling equipment before reuse.

Care should be taken in handling all soil samples to ensure that the exterior of the sample containers are clean and free of soils before shipping. All laboratory analytical samples will be kept on ice in coolers and will be shipped with appropriate QA/QC samples, as described in Worksheet #27.

QA/QC samples will also be collected as part of the soil investigation including field duplicates, rinsate blanks, laboratory QCs, trip blanks, and cooler temperature blanks. Worksheet #18 presents the analytical field program summary. Worksheet #19 presents the analytical methods, sample container types, preservative requirements, and the maximum holding times for the associated analyses. Worksheet #20 presents the quality control sample summary.

All soil samples will be identified in accordance with the sample location identification system presented in Worksheet #27. Soil samples will be handled and delivered in accordance with the chain-of-custody procedures detailed in Worksheet #27. Required data will be recorded on the boring log/sample log sheet, including the sample device used, sampling personnel, date and time of sample, analyses collected, and other information as listed in the boring log (Appendix B).

- **Monitoring Well Completion/Development** – New overburden wells will be installed using direct push technology to maintain consistency in well construction and data comparability within the groundwater data set. Each well will consist of a 2-inch or 1-inch inside diameter (ID) Schedule 40 polyvinyl chloride (PVC) equipped with a 10-foot long well screen (0.010 inch slot openings), surrounded by 20-40 grade silica sand, and bottom plugs. A 5-foot long well screen is acceptable if the geologic unit thickness is less then 11 feet. The well screen length will be 10 feet unless the saturated thickness of the geologic unit is less than 10 feet. All connections will be flush-treaded. Silica sand shall be placed a minimum of 2 feet above the top of the well screen, followed by 2 feet of bentonite rock chips hydrated in place. Bentonite grout will fill the remainder of the annular space to approximately 3.5 feet bgs. A 0.5 foot sand drainage layer shall be placed on top of the bentonite. A protective casing will be centered around the well. Quikrete ™ (or equivalent) will be used to fill the annular space above the sand drainage layer and pad around the well to a radius of 1 foot. The top of the well pad shall be flush with ground surface. Silica sand shall be placed between the well and the protective casing. The protective casing will be painted in a rust-preventative paint and secured with a keyed-alike lock. Wells installed in paved areas and lawns will be flush-mounted and the wells secured with a lockable “J-plug”. Wells installed in other areas will be protected with a “stick-up” steel casing (4-inch diameter by 5-ft long) and protected by bollards. Each new monitoring well will be developed no sooner then 48 hours after well completion. Well development will be performed using a bladder pump or peristaltic pump until the turbidity level reaches 10 Nephelometric Turbidity Units (NTU) or 2 hours of well development elapses (whichever occurs first). See SOP GH-2.8 for additional information.

- **Groundwater Sampling** – All groundwater samples will be collected following EPA's Low Flow (Low Stress) sampling protocol (Appendix A). This method emphasizes the need to minimize water-level drawdown and groundwater pumping rates in order to collect samples with minimal alterations to groundwater chemistry. Submersible bladder pumps with Teflon bladders will be used for low-flow groundwater sampling. Dedicated pumps may be used; non-dedicated pumps will be decontaminated between each well in accordance with this UFP SAP and SOP SA 7.1. The pumping system will be designed to be consistent with the intent of the low-flow groundwater sampling method. Dedicated tubing will be used for each monitoring well to minimize cross-contamination between monitoring wells. During well purging, water level drawdown and flow rate will be recorded on groundwater collection forms. Groundwater will be pumped through a flow-through cell and the pH, conductivity, temperature, dissolved oxygen (DO), and oxidation-reduction potential (ORP) will be measured with a YSI 600 Series water-quality instrument equipped with a flow-through cell. The instrument will be calibrated as described in Worksheet #22. Turbidity will be measured separately with a nephelometer. Every effort will be made to lower the turbidity to less than 5 NTU before sampling. If turbidity below 5 NTU cannot be achieved, samples will still be collected if all parameters are stable and the 2-hour purging limit is reached. Purging is considered complete and sampling may begin when all parameters have stabilized. Stabilization is considered to be achieved when three consecutive readings, taken at 3- to 5-minute intervals, are within the following limits:

  - Turbidity (+ or – 10% if greater than 5 NTU);
  - DO (10% for values greater than 2 mg/L; 0.5 mg/L for values less than 2 mg/L);
  - Specific conductance (3%);
  - Temperature (3%);
  - pH (+ or - 0.5 units);
  - ORP (+ or - 10 millivolts); and
  - Drawdown (no more than 0.3 feet).

If stability criteria are not met after 2 hours of pumping, then document that stabilization was not achieved after 2 hours and collect the groundwater samples.. Care should be taken in handling all groundwater samples to ensure that the exterior of the sample containers are clean and free of liquids before shipping. All laboratory analytical samples will be kept on ice in coolers and will be shipped with appropriate QA/QC samples, as described in Worksheet #27.

QA/QC samples will also be collected as part of the soil investigation including field duplicates, rinsate blanks, laboratory QCs, trip blanks, and temperature blanks. Worksheet #18 presents the analytical field program summary. Worksheet #19 presents the analytical methods, sample container types, preservative requirements, and the maximum holding times for the associated analyses. Worksheet #20 presents the quality control sample summary.

All groundwater samples will be identified in accordance with the sample location identification system presented in Worksheet #27. Groundwater samples will be handled and delivered in accordance with the chain-of-custody procedures detailed in Worksheet #27.

- **Mere Brook Surface Water Sampling** – Surface water sample locations are co-located with the sediment sample locations. Surface water from Mere Brook will be collected within several tenths of a foot above the top of the streambed. Sediment from Mere Brook will be collected from 0 to 6 inches below the sediment surface. The following criterion should be met, if possible, before surface water and sediment can be collected: minimum depth of surface water is 1 foot at each surface water/sediment location. Field notes will document sample collection locations and any necessary deviations from the sampling plan to collect the required number of surface water and sediment samples. The surface water sample will be collected before the sediment sample. Sampling will proceed from downstream to upstream locations. The beaker (or spoon) will be decontaminated prior to use at each sample location. The sampling beaker will be rinsed in the surface water, and the rinse water will be discarded downstream of the sampling station. Surface water samples will then be collected by inverting the beaker and immersing it slowly into the water than slowly inverting the beaker upstream until it is full, then lifting the beaker (full of water) out of the water, without disturbing the stream bottom sediment. The sample will then be poured into the appropriate sample containers. The total depth of the water at each surface water location will be noted on the field forms in addition to the sample depth. After sampling, field parameters (temperature, specific conductivity, DO, pH, turbidity) will be measured by submerging the YSI instrument probe in the flowing surface water and recording the stabilized measurements. Visual observations will also be recorded in the field logbook.

- **Mere Brook Sediment Sampling** –Sediment samples are co-located with surface water samples. Sediment samples from Mere Brook will be collected from 0 to 6 inches below the sediment surface using a stainless steel scoop – the same sampler referenced in the LTMP. Field notes will document sample collection locations and any necessary deviations from the sampling plan to collect the required number of surface water and sediment samples. The surface water sample will be collected before the sediment sample. Sampling will proceed from downstream to upstream locations. The stainless steel scoop will be

decontaminated prior to use. The total depth of the water at each surface water location will be noted on the field forms in addition to the sample depth. Visual observations will also be recorded in the field logbook.

After the water sample is collected and the water quality parameters are recorded, the beaker will be used to collect a sample from the bottom sediment (stainless-steel spoons may be used if field conditions inhibit the use of a beaker). The bottom sediment will be excavated to a depth of no more than 6 inches below the stream bed. A stainless steel scoop will then be filled with sediment, and excess water will be poured from the scoop back into the stream at a point downstream of the sampling location. Stones, leaves, and other non-soil debris will be removed, and then sediment will be homogenized in a stainless steel bowl and transferred directly to the appropriate sample containers. Surface water and sediment sampling stations will be staked with a steel fence post, flagged, labeled, and located using a GPS instrument.

- **Coffins Ice Pond Surface Water Sampling** – Surface water samples will be collected from Coffins Ice Pond using at at Base impoundments (e.g. Picnic Pond) and following TtNUS Standard Operating Procedure (SOP) SA-1.2). The surface water samples at locations will be collected with a Kemmerer sampler due to the depth of surface water. The surface water samples were collected at 60% of the depth of the water column. The total depth of the water at each surface water location will be noted on the field forms in addition to the sample depth. After sampling, field parameters (temperature, specific conductivity, DO, pH, turbidity) will be measured by submerging the YSI instrument probe in the flowing surface water and recording the stabilized measurements. Visual observations will also be recorded in the field logbook. Sample locations will be surveyed using a GPS instrument.

- **Coffins Ice Pond Sediment Sampling** - Sediment samples will be collected utilizing the procedures utilized at Base impoundments following TtNUS Standard Operating Procedure (SOP) SA-1.2. Sediment samples will be collected using a 20-inch by 2-inch Wildco Hand Core Sampler equipped with a dedicated acetate liner. A dedicated basket or acetate line may be used separately if there is insufficient sample recovery. Sediment samples will be collectd from the 0- to 6-inch interval below the sediment surface. Additional sediment cores beside the initial sediment sample location will be collected if there is insufficient sample volume present from the first core.. Samples will be homogenized, decanted if necessary, and transferred into appropriate bottleware.

Field Parameters

- **Groundwater** – The following parameters will be measured: groundwater level, total well depth, pH, temperature, specific conductance, oxidation-reduction potential, and DO. All water quality field parameters, with the exception of turbidity, will be measured using a flow-thru cell. Turbidity will be measured in the discharge water using a turbidity meter.

- **Surface Water** - Same field parameters as groundwater. In addition, sample locations estimated using a GPS.

- **Sediment** - Sample locations estimated using a GPS.

Analytical Parameters – See Table 18-1 for the list of analytical parameters. The rationale for the selection of parameters is discussed in Step 3 in Worksheet #11. The analytical parameters for each sample location are summarized in Worksheet #18.

TABLE 17-1

ENVIRONMENTAL SAMPLING LOCATIONS – SOIL AND GROUNDWATER

Boring/Well [(1)(2)]	Location	Justification
NASB-BG-SB01 through SB05 NASB-BG-MW01 through MW04	Area 1 - Upper Sand	Advance 5 borings in Upper Sand to a depth of 10 ft bgs for collection of 5 surface soil and 5 subsurface soil samples from the Upper Sand. Continue advancing the first 4 boreholes approximately 8 feet into saturated Upper Sand materials to install 4 temporary monitoring wells (1 well in each boring). Develop wells. Collect groundwater samples in 2 events, one after a wet period, and one after a dry period.
NASB BG-EC01 (EC profile only) NASB-BG-MW05	Area 3 (Lower Sand)	Advance electrical conductivity probe through Transition Unit and 5 feet into clay to determine location of Lower Sand. If Lower Sand is present, install 1 temporary monitoring well opposite the Lower Sand unit; otherwise, do not install well. No soil samples for laboratory analysis will be collected. Develop wells. Collect groundwater samples in 2 events, one after a wet period, and one after a dry period.
NASB-BG-SB06 through SB14 NASB-BG-MW06 through MW13 (No NASB-BG-MW14)	Area 4 (Transition Unit)	Advance 9 borings spread across the three "Area 4 subareas" to a maximum depth of 10 feet for collection of 9 surface and 9 subsurface soil samples. Boring locations to be setback a minimum of 200 feet from edge of golf course. Continue advancing the first 8 boreholes approximately 15 feet into saturated Transition Unit and install 8 temporary monitoring wells. Develop wells. Collect groundwater samples in 2 events, one after a wet period, and one after a dry period.
NASB-BG-SB15 through SB17 NASB-BG-MW15 through MW17	Area 5 (Transition Unit)	Advance 3 borings to a depth of 10 feet for collection of 3 surface and 3 subsurface soil samples from the Transition Unit. Continue advancing each borehole approximately 15 feet into saturated Transition Unit materials and install 3 temporary monitoring wells. Develop wells. Collect groundwater samples in 2 events, one after a wet period, and one after a dry period.
NASB-BG-SB18 through SB22 NASB-BG-MW18 through MW21 (No NASB-BG-MW22)	Area 6N (Upper Sand)	Advance 5 borings to a depth of 10 feet for collection of 5 surface and 5 subsurface soil samples from the Upper Sand. Continue advancing the first 4 boreholes approximately 8 feet into saturated Upper Sand materials to install 4 temporary monitoring wells. Develop wells. Collect groundwater samples in 2 events, one after a wet period, and one after a dry period.
NASB-BG-SB23 through SB25 NASB-BG-MW23 through MW25	Area 6S (Transition Unit)	Advance 3 borings to a depth of 10 feet for collection of 3 surface and 3 subsurface soil samples from the Transition Unit. Continue advancing each borehole approximately 15 feet into saturated Transition Unit materials and install 3 temporary monitoring wells. Develop wells. Collect groundwater samples in 2 events, one after a wet period, and one after a dry period. Collect groundwater samples in 2 events, one after a wet period, and one after a dry period.
NASB-BG-SB26 through SB29 (No soil samples in SB28 and SB29) NASB-BG-MW26 through MW29	Area 7 (Off-base-Upper Sand)	Advance 2 borings to a depth of 10 feet for collection of 2 surface and 2 subsurface soil samples from the Upper Sand. Continue advancing each borehole approximately 8 feet into saturated Upper Sand and install 2 temporary monitoring wells. Advance 2 additional borings 8 feet into saturated Upper Sand for installation of 2 temporary monitoring wells. Develop wells. Collect groundwater samples in 2 events, one after a wet period, and one after a dry period. Collect groundwater samples in 2 events, one after a wet period, and one after a dry period.
NASB-BG-SB30 through SB32 NASB-BG-MW30 through MW32	Bowdoin Pines (Off-Base-Upper Sand)	Advance 3 borings in Upper Sand to a depth of 10 ft bgs for collection of 3 surface soil and 3 subsurface soil samples from the Upper Sand. Continue advancing each borehole approximately 8 feet into saturated Upper Sand materials to install 3 temporary monitoring wells. Develop wells. Collect groundwater samples in 2 events, one after

Boring/Well [(1)(2)]	Location	Justification
		a wet period, and one after a dry period.
NASB-BG-EC02 through EC04 (EC profile only) NASB-BG-MW33 through MW35	Area 6N (Lower Sand)	Perform 3 electrical conductivity profiles- through Transition Unit and 5 feet into clay to determine location of Lower Sand. If Lower Sand is present, install 3 temporary monitoring wells opposite the Lower Sand unit; otherwise, do not install well. No soil samples for laboratory analysis will be collected. Develop wells. Collect groundwater samples in 2 events, one after a wet period, and one after a dry period.
NASB-BG-EC05 through EC07 (EC profile only) NASB-BG-MW36 through MW38	Area Northwest of MW-303 (Lower Sand)	Perform 3 electrical conductivity profiles- through Transition Unit and 5 feet into clay to determine location of Lower Sand. If Lower Sand is present, install 3 temporary monitoring wells opposite the Lower Sand unit; otherwise, do not install well. No soil samples for laboratory analysis will be collected. Develop wells. Collect groundwater samples in 2 events, one after a wet period, and one after a dry period.
Existing well: MW-314B	South of Eastern Plume (Transition Unit)	Collect groundwater samples in 2 events, one after a wet period, and one after a dry period. Well located in Weapons Area.
Existing wells: MW-219, MW-302, MW-303, MW-310, MW-314A, MW-315A, MW-231A, MW-335. (8 total)	South and west of Eastern Plume (Lower Sand)	Collect groundwater samples in 2 events, one after a wet period, and one after a dry period. All wells are located in Weapons Area except MW-302 and MW-303 (North of Eastern Plume).
NASB-MW316A, B and MW317A,B	Offbase, east of Eastern Plume	Collect groundwater samples from 2 existing bedrock wells in 2 events that include one event after a wet period and one event after a dry period.

Notes:

(1) Sample locations to be determined in field. Sample names and locations are estimated and may be changed based on field conditions. Once the requisite number samples of each soil type has been collected, sampling will stop. Surface soil and subsurface soil samples do not have to be co-located in the same boring, but may be.

(2) The sample designation that will be used is shown on worksheet 27. Depth will be added as a suffix for soil samples. Date will be added for groundwater samples. For example NASB-BG-SB01-0102 would refer to a soil boring sample collected at a depth between 1 and 2 feet bgs.

(3), 15 monitoring wells targeted for each of the following units: Upper Sand, Transition Unit and Lower Sand.

Abbreviations:
NASB – Naval Air Station Brunswick
BG – Background
SB – Soil boring (surface and subsurface soil sample location).
MW – Monitoring well
EC – Electrical Conductivity
US – Upper Sand
TU– Transition Unit
LS – Lower Sand

TABLE 17-2

ENVIRONMENTAL SAMPLING LOCATIONS – SURFACE WATER AND SEDIMENT LOCATIONS

Surface Water/Sediment Location (co-located)	Location (1)	Justification
NASB-BG-SW01 through SW08	Coffin's Ice Pond. Surface water samples.	Eight surface water sample locations distributed throughout Coffins Pond. Collect surface water samples from each of the 8 locations in 2 events, one after a wet period, and one after a dry period.
NASB-BG-SG01 and NASB-BG-SG02	Coffin's Ice Pond. Sediment samples.	Eight sediment samples locations co-located with Coffins Pond surface water sample locations Collect sediment samples from each of the 8 locations in 2 events, one after a wet period, and one after a dry period.
NASB-BG-SW09 through NASB-BG-SW16	Area 2/Mere Brook - Surface water sample locations to be determined.	Eight surface water sample locations co-located with sediment samples. Sample locations are downstream from Base perimeter road, and downstream of vegetation dam at fence and upstream of first tributary downstream of dam shown on Figure 17-1. Collect surface water samples from each of he 8 locations in 2 events, one after a wet period, and one after a dry period. Determine sediment location based on range of sediment types, then co-locate surface water sample locations with sediment locations.
NASB-BG-SD09 through NASB-BG-SD16	Area 2/Mere Brook- Sediment sample locations co-located with surface water sample locations.	Collect sediment samples from each of the 8 locations that represents a range in sediment types in the stream. Samples to be collected in 2 events, one after a wet period, and one after a dry period. The sediment location for the second event should be the same as the first event.
NASB-BG-SG01 and NASB-BG-SG02	Area 2/Mere Brook - Staff gauge locations to be determined.	Locate NASB-BG-SG01 within 100 feet west of boundary perimeter road, and NASB-BG-SG02 within 100 feet west of culvert beneath runway. Surface water gauges can be used to measure surface water elevations during seasonal sampling events.

Notes:

(1) Sample locations to be determined in field.
(2)The sample designation that will be used is shown on Worksheet 27. Depth will be added as a suffix for sediment samples. Date will be added as a suffix for surface water and sediment samples. For example NASB-BG-SW01-091009 would refer to a NASB background surface water sample 01 collected on September 10, 2009.

Abbreviations:
NASB – Naval Air Station Brunswick
BG – Background
SD – Sediment .
SW –Surface Water
SG – Staff Gauge

SAP Worksheet #18 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Sampling Location / ID Number	Matrix	Depth (units)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]
NASB-BG-SB01-0001 through SB32-0001 except no soil samples for lab analysis at BG-SB28, SB29	Surface Soil	0-1', bgs	PAHs, pesticides, herbicides, TOC, TAL Metals.	30 + 3 FD (See Note 1)	SOP GH-1.3
NASB-BG-SB01-0110 through SB032-0110 except no soil samples for lab analysis at BG-SB28, SB29	Subsurface Soil	1-10' bgs (1)	PAHs, TOC, TAL Metals.	30 + 3 FDs (See Note 2)	SOP SA-1.3
NASB-BG-MW01 through MW38 (except no wells at SB05 and SB14), and 13 existing wells MW-219, MW-302, MW-303, MW-310, MW-231A, MW-314B, MW-314A, MW-315A, MW-335, MW-316A, MW-316B, MW317A, and MW317B.. (15 Upper Sand wells, 15 Transition Unit Wells, and 15 Lower Sand wells sampled 2 times, and 4 bedrock locations sampled 2 times) Sample date added as suffix.	Groundwater	Mid-point of saturated screen interval	TAL Metals (dissolved and total), chloride and bromide (chloride and bromide during first sampling event only).	49+ 5 FD (See Note 4)	EPA Region 1 Low Stress Groundwater Sampling Procedure (Revision 2)
NASB-BG-SD01 through SD08 (Coffins Ice Pond) 8 sediment locations sampled 2 times Sample date added as suffix.	Sediment	0-6 inches below sediment surface	PAHs, pesticides, herbicides, TOC, TAL Metals., ASTM grain size sieve analysis.	8 + 1 FD (See Note 3)	SOP SA-1.2
NASB-BG-SD09 through SD16 (Mere Brook Area 2) 8 sediment locations sampled 2 times Sample date added as suffix.	Sediment	0-6 inches below sediment surface	PAHs, pesticides, herbicides, TOC, TAL Metals., ASTM grain size sieve analysis	8 + 1 FD (See Note 3)	SOP SA-1.2
NASB-BG-SW01 through SW08 (Coffins Ice Pond) 8 surface water locations sampled 2 times. Sample date added as suffix.	Surface Water	6-8 inches below surface water surface	PAHs, TAL Metals (dissolved and Total).	8+ 1 FD (See Note 3)	SOP SA-1.2
NASB-BG-SW09 through SW16 (Mere Brook Area 2) 8 surface water sample locations sampled 2 times, Sample date added as suffix.	Surface Water	6-8 inches below surface water surface	PAHs, TAL Metals (dissolved and Total).	8 + 1 FD (See Note 3)	SOP SA-1.2

Notes:

(1) A surface soil sample will be collected from the 0-1 foot interval bgs. If the surface soil is fill and fill is similar to native materials, then it will be collected; otherwise, it will not be collected.

(2) A vertical mixed sample will be collected from the targeted soil type present between 1 and a maximum of 10 feet bgs.

(3) 8 surface water and 8 sediment sample locations located at Mere Brook; remaining 8 surface water and sediment sample locations distributed in Coffins Ice Pond. All locations to be sampled once after a dry period and once after a wet period.

(4) 15 monitoring wells in each overburden unit (Upper Sand, Transition Unit, Lower Sand) will be sampled. Four bedrock wells will be sampled. All groundwater locations to be sampled once after a dry period and once after a wet period. Chloride and bromide samples to be collected during first sampling event only.

SAP Worksheet #19 -- Analytical SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference[1]	Containers (number, size, and type)	Sample volume (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[2] (preparation / analysis)
Surface Soil	PAHs	3540C/8270 SIM/ CA526/CA213	8 oz glass jar	30 g	Cool to 4° C	14 days to extraction 40 days to analysis
	TCL Pesticides	3550B/8081A/ CA-500/CA-302	8 oz glass jar	30 g	Cool to 4° C	14 days to extraction 40 days to analysis
	Herbicides	8151A/ CA-517/CA-305	8 oz glass jar	30 g	Cool to 4° C	14 days to extraction 40 days to analysis
	TOC	9060A/CA763	4 oz glass jar	1 g	Cool to 4° C	28 days to analysis
	TAL Metals	3050/6010B/6020/7471A/ CA-605/CA-608/CA-627/ CA-611	4 oz glass jar	1 g	Cool to 4° C	180 days to analysis (ICP metals); 28 days (mercury)

Matrix	**Analytical Group**	**Analytical and Preparation Method / SOP Reference[1]**	**Containers** (number, size, and type)	**Sample volume** (units)	**Preservation Requirements** (chemical, temperature, light protected)	**Maximum Holding Time[2]** (preparation / analysis)
Subsurface Soil	PAHs	3540C/8270 SIM/ CA-526/CA-213	8 oz glass jar	30 g	Cool to 4° C	14 days to extraction 40 days to analysis
	TOC	9060A/CA-763	4 oz glass jar	1 g	Cool to 4° C	28 days to analysis
	TAL Metals	3050/6010B/6020/7471A/ CA-605/CA-608/CA-627/ CA-611	4 oz glass jar	1 g	Cool to 4° C	180 days to analysis (ICP metals); 28 days (mercury)
Sediment	PAHs	3540C/8270 SIM/ CA-526/CA-213	8 oz glass jar	30 g	Cool to 4° C	14 days to extraction 40 days to analysis
	TCL Pesticides	3540C/8081A/ CA-524/CA-302	8 oz glass jar	30 g	Cool to 4° C	14 days to extraction 40 days to analysis
	Herbicides	8151A/CA-517/CA-305	8 oz glass jar	30 g	Cool to 4° C	14 days to extraction 40 days to analysis
	TOC	9060A/CA-763	4 oz glass jar	1 g	Cool to 4° C	28 days to analysis

Matrix	**Analytical Group**	**Analytical and Preparation Method / SOP Reference[1]**	**Containers** (number, size, and type)	**Sample volume** (units)	**Preservation Requirements** (chemical, temperature, light protected)	**Maximum Holding Time[2]** (preparation / analysis)
	TAL Metals	3050/6010B/6020/7471A/ CA-605/CA-608/CA-627/ CA-611	4 oz glass jar	1 g	Cool to 4° C	180 days to analysis (ICP metals); 28 days (mercury)
	Grain Size (sieve analysis only)	ASTM D422-63	8 oz glass jar	100 – 200 g	None	None
Groundwater	TAL Metals (Dissolved)	3010/6010B/6020/7470A/ CA-604/CA-608/CA-627/CA-615	500 mL polyethylene	100 ml	HNO_3 to pH < 2, cool to 4° C	180 days to analysis (ICP metals); 28 days (mercury)
	TAL Metals (Total)		500 mL polyethylene	100 ml		
	Chloride and Bromide	EPA Method 300.0/ CA-742	250 mL	1 mL	Cool to 4° C	28 days
Surface Water	PAHs	3510C/8270 SIM/ CA-502/CA-213	2-1 L amber glass jar	1000 ml	Cool to 4° C	7 days to extraction 40 days to analysis
	TAL Metals (Dissolved)	3010/6010B/6020/7470A/ CA-604/CA-608/CA-627/ CA-615	500 mL polyethylene	100 ml	HNO_3 to pH < 2, cool to 4° C	180 days to analysis (ICP metals); 28 days (mercury)
	TAL Metals (total)		500 mL Polyethylene	100 ml		

Matrix	**Analytical Group**	**Analytical and Preparation Method / SOP Reference[1]**	**Containers** (number, size, and type)	**Sample volume** (units)	**Preservation Requirements** (chemical, temperature, light protected)	**Maximum Holding Time[2]** (preparation / analysis)
Blanks	PAHs	3510C/8270 SIM/ CA-502/CA-213	2-1 L amber glass jar	1000 ml	Cool to 4° C	7 days to extraction 40 days to analysis
	TCL Pesticides	3510C/8081A/ CA-515/CA-302	2-1 L amber glass jar	1000 ml	Cool to 4° C	7 days to extraction 40 days to analysis
	Herbicides	8151A/ CA-516/CA-305	2-1 L amber glass jar	1000 ml	Cool to 4° C	7 days to extraction 40 days to analysis
	TAL Metals (Dissolved)	3010/6010B/6020/7470A/ CA-604/CA-608/CA-627/ CA-615	500 mL polyethylene	100 ml	HNO_3 to pH < 2, cool to 4° C	180 days to analysis (ICP metals); 28 days (mercury)
	TAL Metals (Total)			100 ml		
	Chloride and Bromide	EPA Method 300.0/ CA-742	250 mL	1 mL	Cool to 4° C	28 days

SAP Worksheet #20 -- Field Quality Control Sample Summary Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates[1]	No. of MS/MSDs[2]	No. of Field Blanks[3]	No. of Equip. Blanks[4]	No. of Proficiency Testing (PT) Samples	Total No. of Samples to Lab[5]
Surface soil	PAHs	30	3	2	1	2	0	36
	TCL Pesticides	30	3	2	1	2	0	36
	Herbicides	30	3	2	1	2	0	36
	TOC	30	3	2	0	0	0	33
	TAL Metals	30	3	2	1	2	0	36
Subsurface soil	PAHs	30	3	2	0	2	0	35
	TOC	30	3	2	0	0	0	33
	TAL Metals	30	3	2	0	2	0	35
Groundwater – Event 1	Metals (Dissolved)	49	5	3	0	3	0	57
	Metals (Total)	49	5	3	1	3	0	58
	Chloride and Bromide	49	5	3	1	3	0	58
Groundwater - Event 2	Metals (Dissolved)	49	5	3	0	3	0	57
	Metals (Total)	49	5	3	1	3	0	58
Surface Water - Event 1 (Note 6)	PAHs	16	2	1	1	1	0	20
	Metals (Dissolved)	16	2	1	0	1	0	19

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates[1]	No. of MS/MSDs[2]	No. of Field Blanks[3]	No. of Equip. Blanks[4]	No. of Proficiency Testing (PT) Samples	Total No. of Samples to Lab[5]
	Metals (Total)	16	2	1	1	1	0	20
Surface Water - Event 2 (Note 6)	PAHs	16	2	1	1	1	0	20
	Metals (Dissolved)	16	2	1	0	1	0	19
	Metals (Total)	16	2	1	1	1	0	20
Sediment - Event 1 (Note 6)	PAHs	16	2	1	1	1	0	20
	TCL Pesticides	16	2	1	1	1	0	20
	Herbicides	16	2	1	1	1	0	20
	TOC	16	2	1	0	0	0	18
	TAL Metals	16	2	1	1	1	0	20
	Grain Size (sieve analysis only)	16	2	0	0	0	0	18
Sediment – Event 2 (Note 6)	PAHs	16	2	1	1	1	0	20
	TCL Pesticides	16	2	1	1	1	0	20
	Herbicides	16	2	1	1	1	0	20
	TOC	16	2	1	0	0	0	18
	TAL Metals	16	2	1	1	1	0	20
	Grain Size (sieve analysis only)	16	2	0	0	0	0	18

1. Collect 1 field duplicate per 10 field samples.
2. Assign 1 Lab QC sample per 20 samples for MS/MSD for organic analyses (collect triple volume of sediment, groundwater, and surface water for these analyses) and 1 Lab QC for MS/laboratory duplicate for inorganics analyses (collect double volume of sediment, groundwater, and surface water for these analyses)
3. Collect 1 field blank per water source (e.g., decontamination rinse water)

4. Collect 1 rinsate blank per 20 field samples.
5. Total number of samples does not include the MS/MSD.
6. Co-located surface water and sediment samples to be collected from Mere Brook (8 samples/media) and Coffins Ice Pond (8 samples/media) during each of the two sampling events.

Following is a description of the QC sample types.

- Field QC Samples

Field duplicates are used to assess the combined field and laboratory precision. Field duplicates will be collected by alternately filling sample containers for a given analytical group from the source being sampled. Field duplicate samples will be shipped blind to the laboratory.

Equipment rinsate blanks are used to assess the effectiveness of decontamination procedures. Rinsate blanks are obtained under representative field conditions by running analyte-free deionized/distilled water (provided by the laboratory) through sample collection equipment after decontamination and prior to use, and placing it in the appropriate sample containers for analysis.

Field blanks are used to determine the quality of the source water used for decontamination. These samples will consist of the contaminant-free deionized water used in the last step of decontamination.

- Laboratory QC Samples

Laboratory MS provide information about the effect of the sample matrix on the preparation and measurement methodology and **laboratory duplicates** provide information about laboratory precision. MS for organic analyses are performed in duplicate for assessment of laboratory precision. A single MS analysis and a laboratory duplicate analysis will be performed for metals analysis, and a laboratory duplicate analysis for TOC. The laboratory will perform the MS/MSD and laboratory duplicate analyses at a frequency of one for every 20 or fewer investigative samples per sample matrix. The field sampler will assign samples for MS/MSD analysis at this frequency on the chain-of-custody form and sample log sheet. No extra volume for soil samples and triple volume for aqueous and for sediment samples (due to high percent moisture content) should be collected.

SAP Worksheet #21 -- Project Sampling SOP References Table

(UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date and / or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
S1	CT-04 - Sample Nomenclature; Revision 2, 2003	TtNUS	Not applicable	N	SOP is included in Appendix A.
S2	CT-05 - Database Records and Quality Assurance; Revision 2, 2001	TtNUS	Not applicable	N	SOP is included in Appendix A.
S3	GH-1.1 - Site Reconnaissance; Revision 1, 1999	TtNUS	Not applicable	N	SOP is included in Appendix A.
S4	GH-1.2 - Evaluation of Existing Monitoring Wells and Water Level Measurement; Revision 2, 2003	TtNUS	Electronic water level indicator	N	SOP is included in Appendix A.
S5	GH-1.3 - Soil and Rock Drilling Methods; Revision 1, 1999	TtNUS	Drilling rig and accessories	N	SOP is included in Appendix A.
S6	GH-1.5 - Borehole and Sample Logging; Revision 1, 1999	TtNUS	Not applicable	N	SOP is included in Appendix A.
S7	GH-2.5 – Groundwater Contour Maps and Flow Determination; Revision 1, 1999	TtNUS	Not applicable	N	SOP is included in Appendix A.
S8	GH-2.8 - Groundwater Monitoring Well Installation; Revision 3, 2003	TtNUS	Drilling rig, accessories, and well supplies	N	SOP is included in Appendix A.
S9	HS-1.0 - Utility Locating and Excavation Clearance; Revision 2, 2003	TtNUS	Remote subsurface sensing, magnetometer, GPR, etc.	N	SOP is included in Appendix A.
S10	SA-1.2 – Surface Water and Sediment Sampling; Revision 5, 2008	TtNUS		N	SOP is included in Appendix A.
S11	SA-1.3 - Soil Sampling; Revision 8, 2008	TtNUS	Sampling supplies	N	SOP is included in Appendix A.
S12	SA-2.5 - Direct Push Technology; Revision 3, 2003	TtNUS	Drilling rig and accessories	N	SOP is included in Appendix A.
S13	SA-6.1 - Non-Radiological Sample Handling ; Revision 3, 2004	TtNUS	Not applicable	N	SOP is included in Appendix A.
S14	SA-6.3 - Field Documentation; Revision 2, 2003	TtNUS	Not applicable	N	SOP is included in Appendix A.
S15	SA-7.1 - Decontamination of Field Equipment ; Revision 4, 2008	TtNUS	Not applicable	N	SOP is included in Appendix A.
S16	GW 0001 - Low Stress (Low Flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells; Revision 2, 2001	EPA Region 1/GW-001	Submersible pump, Horiba U-22 multiparameter meter, turbidimeter	N	SOP is included in Appendix A.

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

(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Activity[1]	Frequency	Acceptance Criteria	CA	Resp. Person	SOP Reference	Comments
YSI 600 Series Water Quality Meter	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's guidance	Operator correction or Replacement	FOL	GW-0001	Rental field equipment will be used
Turbidity Meter	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's guidance	Operator correction or Replacement	FOL	GW-0001	
Water Level Indicator	Visual Inspection Field checks as per manufacturer	Daily Once upon receiving from vendor	0.01 foot accuracy	Operator correction or Replacement	FOL	GH-1.2	
Photo-Ionization Detector	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's guidance	Operator correction or Replacement	FOL	Operation according to manufacturer's instructions	

1. Rental equipment and instruments will be used in the field. The rental firms will be responsible for the proper care, maintenance, and repair of these items, and for tracking and documenting equipment and instrument maintenance and repairs.

SAP Worksheet #23 -- Analytical SOP References Table

(UFP-QAPP Manual Section 3.2.1)

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
CA-101	"Equipment Maintenance" SOP No. CA-101, Revision 6, 04/07.	Definitive	Various	Various	Katahdin Analytical Services	No
CA-213	"Analysis of Semivolatile Organic Compounds By: SW 846 Method 8270 – Modified For Selected Ion Monitoring (SIM)", SOP No. CA213, Revision 5, 02/08	Definitive	Aqueous and Soil/PAHs	GC/MS-SIM	Katahdin Analytical Services	No
CA-302	"Analysis of Pesticides By Gas Chromatography/Electron Capture Detector (GC/ECD): SW-846 Method 8081", SOP No. CA-302, Revision 8, 01/08.	Definitive	Aqueous and Soil/TCL Pesticides	GC/Electron Capture Detector (ECD)	Katahdin Analytical Services	No
CA-305	"Analysis Of Chlorinated Herbicides By GC Using Methylation Derivatization: Sw-846 Method 8151", SOP No. CA-305, Revision 6, 01/08.	Definitive	Aqueous and Soil/Herbicides	GC/ECD	Katahdin Analytical Services	No
CA-500	"Preparation of Sediment/Soil Samples by Sonication using Method 3550 for subsequent PCB Analysis", SOP No. CA-500, Revision 4, 09/07.	Definitive	Soil/Pesticides	Ultrasonic Extractor	Katahdin Analytical Services	No
CA-502	"Preparation Of Aqueous Samples For Extractable Semivolatile Analysis", SOP No. CA-502, Revision 4, 09/07.	Definitive	Aqueous/PAHs	Separatory Funnel/Continuous Liquid-Liquid Extractor (CLLE)	Katahdin Analytical Services	No
CA-515	"Preparation of Aqueous Samples for Pesticides/PCBs Analysis", SOP No. CA-515, Revision 4, 09/07.	Definitive	Aqueous/Pesticides	Separatory Funnel/CLLE	Katahdin Analytical Services	No

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-516	"Preparation Of Aqueous Samples For Herbicide Analysis By Method 8151" SOP No. CA-516, Revision 3, 04/06.	Definitive	Aqueous/Herbicides	Separatory Funnel	Katahdin Analytical Services	No
CA-517	"Preparation Of Solid Samples For Herbicide Analysis By Method 8151", SOP No. CA-517, Revision 01, 04/06	Definitive	Soil/Herbicides	Ultrasonic Extractions	Katahdin Analytical Services	No
CA-524	"Preparation Of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For Pesticide/PCB Analysis". SOP No. CA524, Revision 4, 09/07.	Definitive	Soil/Pesticides	Soxhlet Extractor	Katahdin Analytical Services	No
CA-526	"Preparation Of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For Subsequent Extractable Semivolatile Analysis", SOP No. CA-526, Revision 4, 09/07.	Definitive	Soil//PAHs	Soxhlet Extractor	Katahdin Analytical Services	No
CA-604	"Acid Digestion of Aqueous Samples by EPA Method3010 for ICP Analysis of Total or Dissolved Metals", SOP No. CA-604, Revision 3, 04/06.	Definitive	Aqueous/ICP Metals	Acid Digestion Apparatus	Katahdin Analytical Services	No
CA-605	"Acid Digestion of Solid Samples by USEPA Method 3050 for Metals by ICP-AES and GF AA", 3050B, SOP No. CA-605, Revision 3, 01/08.	Definitive	Soil/ICP Metals	Acid Digestion Apparatus	Katahdin Analytical Services	No
CA-608	"Trace Metals Analysis By ICP-AES Using EPA Method 6010", SOP No. CA-608, Revision 6, 09/07.	Definitive	Aqueous and Soil/Metals	ICP	Katahdin Analytical Services	No

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-611	"Digestion And Analysis Of Solid Samples For Mercury By USEPA Method 7471", SOP No. CA-611, Revision 3, 03/06	Definitive	Soil/Mercury Analysis	Mercury Analyzer	Katahdin Analytical Services	No
CA-615	"Digestion And Analysis Of Aqueous Samples For Mercury By USEPA Method 7470", CA-615, Revision 1, 04/06.	Definitive	Aqueous/mercury Analysis	Mercury Analyzer	Katahdin Analytical Services	No
CA-627	"Trace Metals Analysis By ICP-MS Using USEPA Method 6020", SOP No. CA-627, Revision 3, 0707	Definitive	Aqueous and Soil/Metals	ICPMS	Katahdin Analytical Services	No
CA-763	"Analysis of TOC, DOC, and TIC in Aqueous Samples using the Shimadzu Carbon Analyzer: EPA Method 415.1, SW846 9060 and SM5310B", SOP No. CA-763, Revision 2, 03/07	Definitive	SoilWet Chemistry	TOC Analyzer	Katahdin Analytical Services	No
CA-742	Anions by Ion Chromatography (IC) – Method 300.0, 02/09, Revision 6.	Definitive	Chloride and Bromide	Ion Chromatagraph	Katahdin Analytical Services	No
SD-902	"Sample Receipt and Internal Control", SOP No. SD-902, Revision 7, 02/07.	Definitive	Various	NA	Katahdin Analytical Services	No
SD-903	"Sample Disposal", SOP No. SD-903, Revision 2, 04/06.	Definitive	Various	NA	Katahdin Analytical Services	No
BR-GT-006	Particle Size Analysis, ASTMD422-63, Revision 5, 1/1/08	Definitive	Sediment/Grain Size	Sieves	Test America Burlington	No

1 If yes, then specify the modification that has been made. Note that any analytical SOP modification made relative to project specific needs must be reviewed and approved by the Navy QAO.

SAP Worksheet #24 -- Analytical Instrument Calibration Table

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA[2]	SOP Reference
GCMS-PAHs - SIM	Initial Calibration	ICAL – Instrument receipt, instrument change (new column, source cleaning, etc.), when CCV is out of criteria. Six-point initial calibration for all analytes	RSD ≤30 for RFs of the CCCs; Average %RSD < 15% for all compounds	Repeat calibration if criterion is not met	Analyst, Supervisor	CA-213
	Continuing Calibration	CCV – at the beginning of each 12 hour shift immediately after DFTPP tune.	CCCs ≤ 20%D; SPCCs RF ≥0.050	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification	Analyst, Supervisor	
	Tune				Analyst, Supervisor	
ICP	Initial calibration	At the beginning of each day or if QC is out of criteria.	One point calibration per manufacturer's guidelines	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Supervisor	CA-608
	Continuing calibration	At the beginning and end of each run sequence and every 10 samples	90-110% of True Values	Check problem, recalibrate and reanalyze any samples not bracketed by passing CCVs.	Analyst, Supervisor	
ICP-MS	Tune	Daily prior to calibration	Mass calibration within 0.1 atomic mass units (amu) of true value, Resolution < 0.9 amu at 10% peak height	Perform necessary equipment maintenance	Analyst, Supervisor	Ca-627

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA[2]	SOP Reference
	Initial calibration	Daily prior to sample analysis.	4 point calibration plus blank – correlation coefficient ≥ 0.995.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Supervisor	
	Continuing calibration	At the beginning and end of each run sequence and every 10 samples	90-110% of True Values	Check problem, recalibrate and reanalyze any samples not bracketed by passing CCVs.	Analyst, Supervisor	
Mercury analyzer	Initial calibration	IC-instrument receipt, major instrument change, at the start of each day	Correlation coefficient ≥ 0.995.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Supervisor	CA-611, CA-615
	continuing calibration	CCV-at beginning and end of each run sequence and every 10 samples	80-120% of True Value	Check problem, recalibrate and reanalyze any samples not bracketed by passing CCVs.	Analyst, Supervisor	
GC-Pesticides	Initial calibration	IC-instrument receipt, major instrument change, when CC does not meet criteria	6 pt calibration of all pesticides except for toxaphene and chlordane - coefficient of determination ≥ 0.990. Mid-point cal of toxaphene and chlordane.	Repeat Initial calibration and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Supervisor	CA-302
	Continuing calibration	After every 10 samples; If calibration curve previously analyzed, analyze daily before samples.	%D ≤ 15 for both the quantitation and confirmation columns	Evaluate the samples: If the %D >+15% and sample results are <PQL, narrate. If %D >±15% only on one channel, narrate. If %D >±15% for closing CV, and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples back to last acceptable CV.	Analyst, Supervisor	
GC-Herbicides	Initial calibration	IC-instrument receipt, major instrument change, when CC does not meet criteria	6 pt calibration of all herbicides	Repeat Initial calibration and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Supervisor	CA-305

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	CA	Person Responsible for CA[2]	SOP Reference
	Continuing calibration	After every 10 samples; If calibration curve previously analyzed, analyze daily before samples.	%D ≤ 15 for both the quantitation and confirmation columns	Evaluate the samples: If the %D >+15% and sample results are <PQL, narrate. If %D >±15% only on one channel, narrate. If %D >±15% for closing CV, and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples back to last acceptable CV.	Analyst, Supervisor	
TOC analyzer	Initial calibration	Initial Calibration- initially, when the daily CCV does not pass, but no longer than every 3 months.	Correlation coefficient ≤ 0.995	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Supervisor	CA-763
	Continuing calibration	CCV-every 10 samples and at the end of the run	80-120%	If the CCV fails high, report samples that are <PQL. Recalibrate and/or reanalyze samples back to last acceptable CCV recovery.	Analyst, Supervisor	
Ion Chromatograph (Chloride and Bromide)	Initial Instrument Calibration (Blank + 5 standards, lowest standard at or below PQL)	Every 6 months or with each change in instrument operating conditions or equipment	(1) Correlation coefficient ≥ 0.995 (2) Recovery of lowest standard within 50%-150%	Correct problem and recalibrate	Analyst, Supervisor	CA-742
	Initial calibration verification (ICV) (second source)	One per prep/analysis batch of 20 or fewer samples, prepared from a separate source than calibration standards	90%-110% Recovery	(1) If the ICV fails high, report samples that are <PQL. (2) Recalibrate and/or reanalyze other samples.	Analyst, Supervisor	
	Midrange continuing calibration verification (CCV)	At beginning of run, after every 10 samples, and at end of run	90%-110% Recovery	(1) If the CCV fails high, report samples that are <PQL. (2) Recalibrate and/or reanalyze samples back to last acceptable CCV recovery	Analyst, Supervisor	

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

(UFP-QAPP Manual Section 3.2.3)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person[2]	SOP Reference
GCMS- PAH	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	QC standards	Ion source, injector liner, column, column flow	Prior to initial calibration and/or as necessary	Refer to SOP	Refer to SOP	Analyst, Department Manager	CA-204, CA-213
ICP	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, replace peristaltic pump tubing as needed. Other maintenance specified in lab Equipment Maintenance SOP.	QC standards	Torch, nebulizer chamber, pump, pump tubing	Prior to initial calibration and as necessary	Refer to SOP	Refer to SOP	Analyst, Department Manager	CA-608
ICPMS	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, replace peristaltic pump tubing as needed. Other maintenance specified in lab Equipment Maintenance SOP.	QC standards	Torch, nebulizer, spray chamber, pump tubing	Prior to initial calibration and as necessary	Refer to SOP	Refer to SOP	Analyst, Department Manager	CA-627

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person[2]	SOP Reference
Mercury Analyzer	Replace peristaltic pump tubing, replace mercury lamp, replace drying tube, clean optical cell and/or clean liquid/gas separator as needed. Other maintenance specified in lab Equipment Maintenance SOP.	QC standards	Tubing, sample probe, optical cell	Prior to initial calibration and as necessary	Refer to SOP	Refer to SOP	Analyst, Department Manager	CA-611, CA-615
TOC Combustion Analyzer	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 necessary.	QC standards	Tubing, sample boat, syringe, humidifier, rinse Reservoir, phosphoric acid vessel, oxygen pressure	Prior to initial calibration and as necessary	Refer to SOP	Refer to SOP	Analyst, Department Manager	CA-763
GC/ECD-Pesticides	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	QC standards	Injector liner, septa, column, column flow.	Prior to initial calibration and/or as necessary.	Refer to SOP	Refer to SOP	Analyst, Department Manager	CA-302
GC/ECD-Herbicides	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	QC standards	Injector liner, septa, column, column flow.	Prior to initial calibration and/or as necessary.	Refer to SOP	Refer to SOP	Analyst, Department Manager	CA-305

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person[2]	SOP Reference
GC/MS (680)	Check pressure and gas supply daily. Change septa as needed, change liner as needed, cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP (AN53)	Physical check	Check pressure and gas supply daily. Change septa as needed, change liner as needed, cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP (AN53)	As needed	Refer to SOP	Refer to SOP	Analyst, Supervisor	AN53
Ion Chromatograph (Chloride and Bromide)	Check regenerate pump tubing and replace as needed. Clean or regenerate column as needed. Replace analytical column or guard column as needed. Change suppressor as needed.	QC Standards	Tubing, column, suppressor.	Prior to initial calibration and/or as necessary.	Passing ICAL or CCV.	Correct problem and repeat calibration or CCV.	Analyst, Department Manager	CA-742

SAP Worksheet #26 -- Sample Handling System

(UFP-QAPP Manual Appendix A)

SAMPLE HANDLING SYSTEM

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): FOL, Tetra Tech NUS, Inc.
Sample Packaging (Personnel/Organization): FOL, Tetra Tech NUS, Inc.
Coordination of Shipment (Personnel/Organization): FOL, Tetra Tech NUS, Inc.
Type of Shipment/Carrier: Hand carrier or Federal Express
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Receipt Personnel/Katahdin Analytical Services, Inc. ; Sample Receipt Personnel/Sample Custodian/TestAmerica
Sample Custody and Storage (Personnel/Organization): Sample Receipt Personnel/Katahdin Analytical Services, Inc.; Sample Custodian/TestAmerica
Sample Preparation (Personnel/Organization): Extractions Personnel/Katahdin Analytical Services, Inc. ; Sample Preparation Group/TestAmerica
Sample Determinative Analysis (Personnel/Organization): Analytical Personnel/Katahdin Analytical Services, Inc; Bench Chemist/Test America
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): samples will be shipped or delivered to the laboratory 1 or 2 days from collection
Sample Extract/Digestate Storage (No. of days from extraction/digestion): Katahdin Analytical Services, Inc. 1 year; TestAmerica Savannah: 30 days from invoice date; TestAmerica Sacramento: 120 days
Biological Sample Storage (No. of days from sample collection): N/A

SAMPLE DISPOSAL
Personnel/Organization: Environmental Health and Safety Officer\ Katahdin Analytical Services, Inc.; Sample Custodian/TestAmerica
Number of Days from Analysis: Katahdin Analytical Services, Inc. : Samples may be disposed of 90 days after report mail date; TestAmerica: 30 days from invoice date

SAP Worksheet #27 – Sample Custody Requirements Table

(UFP-QAPP Manual Section 3.3.3)

Sample Designation and Tracking System

Each sample collected will be assigned a unique sample tracking number used to catalog the results. The sample tracking number for all samples except groundwater samples from existing wells will consist of alphanumeric characters identifying the Site, sample medium, location, and depth or date. Any other pertinent information regarding sample identification will be recorded on the sample logsheets or in the field logbooks. The alphanumeric (A-N) coding to be used in the sample system is described below.

AA	-	AA	-	AANN	-	(NNNN)
(Site ID)	-	(Medium)		(Location)	-	(Depth or Date)

Site identifier: NASB-BG

Medium identifier: "SB" for soil boring samples (surface and subsurface),
"SD" for sediment samples,
"SW" for surface water samples, and
"GW" for groundwater samples,
.

Sample location identifier: The sample location numbers for soil and groundwater samples are identified in Table 17-2. The location numbers for surface water and sediment samples are listed in Table 17-3.

Depth/Date: For soil samples, this portion of the sample tracking number will represent the depth in feet bgs from which the sample was collected; e.g., for soil samples collected from 1 to 3 feet bgs, this portion of the sample tracking number will be "0103".

For surface water, sediment and for groundwater samples from new wells, this portion of the sample tracking number will represent the date of the sample collection, "MMYY".

For example, a groundwater sample from well MW1302 collected in September of 2008 will be identified as: NASB-GW-MW1302-0908.

For groundwater samples from existing wells, the sample tracking number will be the same as that used previously, except for the addition of the date suffix.

Quality Control (QC) Samples collected during the background investigation will use the same coding system as for the environmental samples. Field QC designations will conform with the following formats:

- Field Duplicates: Blind field duplicate samples will be designated such that the location designation will be replaced with "DUP" followed by a sequential value (the nth duplicate sample collected during that sampling event) and the date (MMDDYY). The sample log sheet will note which sample location the duplicate was collected from. The first groundwater field duplicate sample collected September 14, 2008 will be labeled NASB-GW-DUP01-091408 and the second field duplicate collected on September 15, 2008 will be NASB-GW-DUP02-091508.

- Field Blanks: Field blank sample identifiers will consist of the site identifier, the medium, the "FB" label, and the date (MMDDYY). Example: NASB-GW-FB01-091408.

- Rinsate Blanks: Rinsate blank sample identifiers will consist of the site identifier, the medium, the "RB" label, and the date (MMDDYY). Example: NASB-GW-RB01-091408.

- Laboratory QC samples (MS and laboratory duplicate samples) have no separate sample identifier codes, but are noted on the chain-of-custody record and sample log sheet.

Sample Collection Documentation

A project-specific field logbook will be used to keep daily records of significant events, observations, and measurements during field investigations. The field logbook also will be used to document all sampling activities. Logbook entries will be made with indelible ink to provide a permanent record and any errors found in the logbook will be verified, crossed-through, and initialed by the person discovering the error. Field logbooks will be maintained according to TtNUS SOP No. SA-6.3 (Appendix A). The field logbooks are intended to provide sufficient data and observations to reconstruct events that occurred during field activities. Field logbooks should be permanently bound and pre-paginated; designated forms should be used whenever possible to ensure that field records are complete. The following items are examples of information that may be included in a field logbook:

- Name, date, and time of entry
- Names and responsibilities of field crew members
- Name and titles of any site visitors
- Descriptions of field procedures, and problems encountered
- Samples taken at each location

- Details of sampling location, including sampling coordinates
- Sample identification numbers of all samples collected
- Date and time of collection
- Sample collector
- Sample collection method
- Decontamination procedures
- Field instrument calibration and maintenance
- Field measurements (e.g., DO, ORP, temperature, pH, specific conductance) and general observations.
- Weather conditions
- Site observations
- Site sketches
- Health & Safety issues including Personal Protective Equipment (PPE)
- Log of photographs

Field sample log sheets will be used to document sample collection details, while other observations and activities will be recorded in the field logbook. Instrument calibration logs will be used to record the daily instrument calibration. The following sections outline the information that will be documented in the field according to the media to be sampled and the activities to be performed. Examples of these forms can be found in Appendix B. Groundwater level measurement and “Low Flow” log sheets will be used to document the groundwater elevations and the samples collected.

Field Sample Handling and Chain-of Custody Procedures

Custody of samples must be maintained and documented at all times to ensure the integrity of a sample from collection through analysis. An accurate written record is necessary to trace the possession and handling of the sample, this documentation is referred to as the "chain of custody" form. Chain of custody begins when samples are collected in the field, and is maintained by storing the samples in secure areas until custody can be passed on. All samples will be delivered to the laboratory accompanied by a chain-of-custody form that will describe the sample identifiers, the analytical parameters, and the persons who are responsible for the sample integrity.

Following collection, samples will be placed on ice in a secure cooler and attended by TtNUS personnel or placed in locked vehicles or designated storage areas until analysis or shipment to an off-site laboratory. Chain of custody procedures are described in further detail in the following TtNUS SOPs: SA-6.3, Field Documentation and SA-6.1, Non-Radiological Sample Handling (Appendix A).

The samples will be shipped to the laboratory in coolers packed with ice and bubble wrap, or equivalent packing material, to cushion the samples to prevent breakage and to maintain the required temperature for the samples. A container filled with water and labeled "temperature blank" will be included in each cooler. The temperature of this blank will be measured by the laboratory upon sample receipt to verify acceptable sample preservation temperature. The coolers will be taped and sealed with a signed custody seal to ensure the chain of custody is maintained. Samples will be shipped to the laboratory by an overnight courier to ensure that maximum sample holding times are not exceeded. The maximum allowable sample holding times for each analytical group is presented in Worksheet #19. This worksheet also lists the sample containers, chemical preservatives, and temperature condition requirements to maintain the integrity of the sample.

Each sample collected will be assigned a unique sampling tracking number, as described above. The sample number, sample collection date and time, person collecting the sample and a list of the sample analyses to be performed will be recorded on each container, and also on the chain-of-custody form. The chain-of-custody form is a two-part form: the original accompanies the samples to the analytical laboratory, and the copy will be archived in the project files. The following Information will be recorded on the chain-of-custody form:

- Project name and number
- Sample matrix
- Sample collector's name
- Dates/times of sample collection
- Sample identification numbers
- Number and type of containers for each sample aliquot
- Type of preservation
- QC sample designation
- Analysis method
- Special handling instructions
- Destination of samples
- Name, date, time, and signature of each individual releasing the shipping container

The field crew will attempt to identify any potentially high concentration samples on the chain-of-custody form.

Laboratory Custody Procedures

The laboratory's sample receipt and chain of custody procedures are included in Appendix D. The laboratory sample custodian will inspect the integrity of the cooler custody seals and measure the temperature of the samples received using the "Temperature Blank" container included in each cooler. The samples will be checked against the chain-of-custody form for holding times, sample identification, and integrity. The samples will be logged into the laboratory management system. Custody of the samples will be maintained and recorded in the laboratory from receipt to analysis and this record will be included with the data package deliverables.

The laboratory SOPs detailing procedures for disposal of the environmental samples are included in Appendix D.

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SAP Worksheet #28a -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Note: Katahdin Analytical Services' "statistically-derived limits" in the following worksheets are presented following Worksheet 28l in Table 28a.

Matrix	Aqueous (SW)
Analytical Group	TCL PAHs (SIM)
Analytical Method/ SOP Reference	SW846 8270 SIM/ CA-213

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch of twenty or fewer samples of similar matrix	No analytes detected > PQL.	(1) Investigate source of contamination (2) Reprep and analyze method blank and all samples processed with the contaminated blank	Analyst, Supervisor, QA Manager	Accuracy/bias-Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
Reagent Blank	1 per Lot	No analyte detected > QL	(1) Investigate source of contamination (2) If required replace Lot	Analyst, Supervisor, QA Manager	Accuracy/bias-Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
MS	One per prep batch of twenty or fewer samples of similar matrix	Katahdin Analytical Services statistically derived limits	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, reprep and analyze the samples and QC.	Analyst, Supervisor, QA Manager	Accuracy/bias	Katahdin Analytical Services statistically derived limits
MSD	One per prep batch of twenty or fewer samples of similar matrix	Katahdin Analytical Services statistically derived limits, Precision RPD ≤ 50%.	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, reprep and analyze the samples and QC.	Analyst, Supervisor, QA Manager	Accuracy/bias & Precision	Katahdin Analytical Services statistically derived limits, Precision RPD ≤ 50%
LCS	One per prep batch of twenty or fewer samples of similar matrix.	Katahdin Analytical Services statistically derived acceptance limits.	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCS Duplicate (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.	Analyst, Supervisor, QA Manager	Accuracy/bias	Katahdin Analytical Services statistically derived limits

Matrix	Aqueous (SW)
Analytical Group	TCL PAHs (SIM)
Analytical Method/ SOP Reference	SW846 8270 SIM/ CA-213

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
IS	Every sample, control, standard, and method blank	Retention time (RT) ± 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. If reanalysis confirms matrix interference, report sample and narrate.	Analyst, Supervisor, QA Manager	Accuracy	RT ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS
Surrogates	Every sample, control, standard, and method blank	Statistically derived acceptance 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	Analyst, Supervisor, QA Manager	Accuracy/bias	Statistically derived acceptance limits.
Temperature Blank	One per cooler	4° C $\pm$ 2° C	Laboratory will notify TtNUS Project Manager (PM) of temperatures outside criteria. TtNUS PM will respond whether to proceed with analysis.	Laboratory Quality Manager and TtNUS Project Manager.	Accuracy/bias/ representativeness	4° C $\pm$ 2° C

SAP Worksheet #28b -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Note: Katahdin Analytical Services' "statistically-derived limits" in the following worksheets are presented following Worksheet 28l in Table 28a.

Matrix	Soil/Sediment
Analytical Group	PAHs (SIM)
Analytical Method/ SOP Reference	SW846 8270 SIM/ CA-213

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch of twenty or fewer samples of similar matrix	No analytes detected > PQL.	(1) Investigate source of contamination (2) Reprep and analyze method blank and all samples processed with the contaminated blank	Analyst, Supervisor, QA Manager	Accuracy/bias-Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
Reagent Blank	1 per Lot	No analyte detected > QL	(1) Investigate source of contamination (2) If required replace Lot	Analyst, Supervisor, QA Manager	Accuracy/bias-Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
MS	One per prep batch of twenty or fewer samples of similar matrix	Katahdin Analytical Services statistically derived limits	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, reprep and analyze the samples and QC.	Analyst, Supervisor, QA Manager	Accuracy/bias	Katahdin Analytical Services statistically derived limits
MSD	One per prep batch of twenty or fewer samples of similar matrix	Katahdin Analytical Services statistically derived limits, Precision RPD ≤ 50%.	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, reprep and analyze the samples and QC.	Analyst, Supervisor, QA Manager	Accuracy/bias & Precision	Katahdin Analytical Services statistically derived limits, Precision RPD ≤ 50%
LCS	One per prep batch of twenty or fewer samples of similar matrix.	Katahdin Analytical Services statistically derived 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 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 and analyze them.	Analyst, Supervisor, QA Manager	Accuracy/bias	Katahdin Analytical Services statistically derived limits

Matrix	Soil/Sediment
Analytical Group	PAHs (SIM)
Analytical Method/ SOP Reference	SW846 8270 SIM/ CA-213

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
IS	Every sample, control, standard, and method blank	RT ± 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. If reanalysis confirms matrix interference, report sample and narrate.	Analyst, Supervisor, QA Manager	Accuracy	RT ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS
Surrogates	Every sample, control, standard, and method blank	Statistically derived acceptance 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	Analyst, Supervisor, QA Manager	Accuracy/bias	Statistically derived acceptance limits.
Temperature Blank	One per cooler	4° C $\pm$ 2° C	Laboratory will notify TtNUS Project Manager (PM) of temperatures outside criteria. TtNUS PM will respond whether to proceed with analysis.	Laboratory Quality Manager and TtNUS Project Manager.	Accuracy/bias/ representativeness	4° C $\pm$ 2° C

SAP Worksheet #28c -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Note: Katahdin Analytical Services' "statistically-derived limits" in the following worksheets are presented following Worksheet 28l in Table 28a.

Matrix	Soil/Sediment
Analytical Group	TCL Pesticides
Analytical Method/ SOP Reference	SW846 8081A/ CA-302

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch of twenty or fewer samples of similar matrix	No analyte detected > QL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the QL, report sample results which are <QL or > 10X the blank concentration. Otherwise, reprep a blank and samples >QL and <10XQL and analyze them.	Analyst, Supervisor, QA Manager	Accuracy/bias-Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
Reagent Blank	1 per Lot	No analyte detected > QL	(1) Investigate source of contamination (2) If required replace Lot	Analyst, Supervisor, QA Manager	Accuracy/bias-Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
MS	One per prep batch of twenty or fewer samples of similar matrix	Katahdin Analytical Services statistically derived limits	(1) Evaluate the samples and associated QC (2) If the LCS results are acceptable, narrate (3) If both the LCS and MS/MSD are unacceptable, reprep and analyze the samples and QC.	Analyst, Supervisor, QA Manager	Accuracy/bias	Katahdin Analytical Services statistically derived limits
MSD	One per prep batch of twenty or fewer samples of similar matrix	Katahdin Analytical Services statistically derived limits, Precision RPD ≤ 50%	(1) See above	Analyst, Supervisor, QA Manager	Accuracy/bias & Precision	Katahdin Analytical Services statistically derived limits, Precision RPD ≤ 50%
LCS	One per prep batch of twenty or fewer samples of similar matrix	Katahdin Analytical Services statistically derived limits	(1) If an MS/MSD was performed and acceptable, narrate. (2) If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. (3) If the LCS recovery is high but the sample results are <QL, narrate. Otherwise, re-extract blank and affected sample batch.	Analyst, Supervisor, QA Manager	Accuracy/bias	Katahdin Analytical Services statistically derived limits

Matrix	Soil/Sediment
Analytical Group	TCL Pesticides
Analytical Method/ SOP Reference	SW846 8081A/ CA-302

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogates	2 per Sample	Statistically derived, Tetrachloro-m-xylene: 41-116 Decachlorobiphenyl: 52-119	(1) No CA will be taken when one surrogate is within criteria. (2) If surrogates are outside high and sample is <QL no CA taken. (3) If surrogates are outside low the affected samples are re-extracted and reanalyzed.	Analyst, Supervisor, QA Manager	Accuracy/bias	Statistically derived, Tetrachloro-m-xylene: 41-116 Decachlorobiphenyl: 52-119
Temperature Blank	One per cooler	4° C $\pm$ 2° C	Laboratory will notify TtNUS Project Manager (PM) of temperatures outside criteria. TtNUS PM will respond whether to proceed with analysis.	Laboratory Quality Manager and TtNUS Project Manager.	Accuracy/bias/ representativeness	4° C $\pm$ 2° C

SAP Worksheet #28d -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Soil/Sediment
Analytical Group	Herbicides
Analytical Method/ SOP Reference	SW846 8151A/ CA-305

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch; no greater than 1/20	No analyte detected > QL	Investigate source of contamination Evaluate the samples and associated QC: i.e. If the blank results are above the QL, report sample results which are <QL or > 10X the blank concentration. Otherwise, reprep a blank and samples >QL and <10X the blank and analyze them.	Analyst, Supervisor, QA Manager	Accuracy/bias-Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
Reagent Blank	1 per Lot	No analyte detected > QL	Investigate source of contamination If required replace Lot	Analyst, Supervisor, QA Manager	Accuracy/bias-Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
MS	One per prep batch of twenty or fewer samples of similar matrix	Nominal limits 30-120% Recovery	Evaluate the samples and associated QC If the LCS results are acceptable, narrate If both the LCS and MS/MSD are unacceptable, reprep and analyze the samples and QC.	Analyst, Supervisor, QA Manager	Accuracy/bias	30-120% Recovery
MSD	One per prep batch of twenty or fewer samples of similar matrix	Nominal limits 30-120% Recovery, ≤ 50% RPD	See above	Analyst, Supervisor, QA Manager	Accuracy/bias & Precision	Recovery 30-120%, RPD ≤ 50%
LCS	One per prep batch; no greater than 1/20	Nominal limits 30-120% Recovery	If an MS/MSD was performed and acceptable, narrate. If the LCS recovery is high but the sample results are <QL, narrate. Otherwise, re-extract blank and affected sample batch.	Analyst, Supervisor, QA Manager	Accuracy/bias	30-120% Recovery
Surrogates	1 per Sample	2,4-Dichlorophenylacetic acid: 29-116%	No CA will be taken when one surrogate is within criteria. If surrogates are outside high and sample is <QL no CA taken. If surrogates are outside low the affected samples are re-extracted and reanalyzed.	Analyst, Supervisor, QA Manager	Accuracy/bias	2,4-Dichlorophenylacetic acid: 29-116%

Matrix	Soil/Sediment
Analytical Group	Herbicides
Analytical Method/ SOP Reference	SW846 8151A/ CA-305

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Temperature Blank	One per cooler	4° C $\pm$ 2° C	Laboratory will notify TtNUS Project Manager (PM) of temperatures outside criteria. TtNUS PM will respond whether to proceed with analysis.	Laboratory Quality Manager and TtNUS Project Manager.	Accuracy/bias/ representativeness	4° C $\pm$ 2° C

SAP Worksheet #28e -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Soil/Sediment
Analytical Group	Total Organic Carbon
Analytical Method/ SOP Reference	SW846 9060/CA-763

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch of 20 or fewer samples	No TOC detected ≥ QL	(1) Investigate source of contamination (2) Re-analyze all affected samples.	Analyst, Supervisor, QA Manager	Accuracy/bias-Contamination	No TOC > ½ QL
Laboratory Duplicate	One per prep batch of 20 or fewer samples	Precision ≤ 20% RPD	(1) Narrate any results that are outside control limits	Analyst, Supervisor, QA Manager	Precision	RPD ≤ 20%
LCS	One per prep batch of 20 or fewer samples	80-120%	(1) Re-analyze associated samples (2) If sample is within holding time, re-analyze affected sample batch. (3) If the LCS recovery is high but the sample results are <QL, narrate. Otherwise, re-prep blank and affected sample batch.	Analyst, Supervisor, QA Manager	Accuracy/bias	80-120%
Temperature Blank	One per cooler	4° C ± 2° C	Laboratory will notify TtNUS Project Manager (PM) of temperatures outside criteria. TtNUS PM will respond whether to proceed with analysis.	Laboratory Quality Manager and TtNUS Project Manager.	Accuracy/bias/ representativeness	4° C ± 2° C

SAP Worksheet #28f -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Aqueous (SW, GW)
Analytical Group	Metals (ICP-AES)
Analytical Method/ SOP Reference	SW846 6010B/ CA-608

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
ICV	Immediately after calibration	± 10 %	Correct problem, recalibrate and reanalyze ICV	Analyst, Supervisor, QA Manager	Accuracy/bias	± 10 %
ICB	Immediately after the ICV	≤ PQL	Correct problem, recalibrate and reanalyze ICV and ICB	Analyst, Supervisor, QA Manager	Accuracy/bias, Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
PQL Standard for ICP (PQL)	At the beginning of a sample run, after every 20 samples and at the end of the run	80-120 % recovery.	Reanalyze immediately for failing elements only. Terminate analysis, correct problem, recalibrate and reanalyze all analytical samples analyzed since last good PQL Std.	Analyst, Supervisor, QA Manager	Sensitivity	70-130 % recovery .
Preparation Blank (PBW)	One per prep batch of twenty or fewer samples of similar matrix	Absolute value < PQL.	If blank value > PQL report sample results if < PQL or > 10 x the blank value; otherwise redigest and analyze. If blank value is less than negative PQL, report sample results if > 10x the absolute value of the blank result, otherwise redigest and analyze.	Analyst, Supervisor, QA Manager	Accuracy/bias, Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
ICS (ICP only)	At beginning and end of run	ICS-A: For Al, Ca, Fe, and Mg, recovery within ± 20% of true value. For analytes not spiked, ±PQL, or, if PQL ≤ 0.01 mg/L, ± 2x PQL. ICS-AB: Recovery of each analyte within ± 20% of true value.	Do not use results for failing elements. Investigate and correct problem.	Analyst, Supervisor, QA Manager	Accuracy/Bias	ICS-A: Unspiked analytes ≤ 2x MDL, if interferent concentrations in sample > 50% interferent concentration in ICS-A solution. ICS-AB: Recovery of each analyte within ± 20% of true value.

Matrix	Aqueous (SW, GW)
Analytical Group	Metals (ICP-AES)
Analytical Method/ SOP Reference	SW846 6010B/ CA-608

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Serial Dilution (L)	One per prep batch of twenty or fewer samples of similar matrix	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag results for affected analytes for all associated samples with "E".	Analyst, Supervisor, QA Manager	Accuracy/Bias, Precision	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 15% of the original result.
LCSW	One per prep batch of twenty or fewer samples of similar matrix	Recovery within ± 20% of true value.	Redigest and reanalyze all associated samples for affected analyte.	Analyst, Supervisor, QA Manager	Accuracy/Bias	80-120 % recovery.
Laboratory Duplicate	One per prep batch of twenty or fewer samples of similar matrix	≤ 20% RPD	Narrate any results that are outside control limits	Analyst, Supervisor, QA Manager	Precision	Values ≥ 5xPQL: RPD ± 20% Values < 5xPQL: Absolute Difference ≤ PQL
MS	One per prep batch of twenty or fewer samples of similar matrix	Recovery ± 25 % of true value if sample < 4x spike value	Flag results for affected analytes for all associated samples with "N".	Analyst, Supervisor, QA Manager	Accuracy/Bias	Recovery ± 25 % of true value if sample < 4x spike value
Post-Digestion Spike	One per prep batch of 20 or fewer samples	Recovery within 75-125 % expected	"J" flag all sample results for specific analyte for all samples associated with the post digestion spike addition.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Recovery ± 25 % of true value
Temperature Blank	One per cooler	4° C ± 2° C	Laboratory will notify TtNUS Project Manager (PM) of temperatures outside criteria. TtNUS PM will respond whether to proceed with analysis.	Laboratory Quality Manager and TtNUS Project Manager.	Accuracy/bias/ representativeness	4° C ± 2° C

SAP Worksheet #28g -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Soil/Sediment
Analytical Group	Metals (ICP-AES)
Analytical Method/ SOP Reference	SW846 6010B/ CA-608

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
ICV	Immediately after calibration	± 10 %	Correct problem, recalibrate and reanalyze ICV	Analyst, Supervisor, QA Manager	Accuracy/bias	± 10 %
ICB	Immediately after the ICV	≤ PQL	Correct problem, recalibrate and reanalyze ICV and ICB	Analyst, Supervisor, QA Manager	Accuracy/bias, Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
PQL Standard for ICP (PQL)	At the beginning of a sample run, after every 20 samples and at the end of the run	80-120 % recovery.	Reanalyze immediately for failing elements only. Terminate analysis, correct problem, recalibrate and reanalyze all analytical samples analyzed since last good PQL Std.	Analyst, Supervisor, QA Manager	Sensitivity	70-130 % recovery .
PBW	One per prep batch of twenty or fewer samples of similar matrix	Absolute value < PQL.	If blank value > PQL report sample results if < PQL or > 10 x the blank value; otherwise redigest and analyze. If blank value is less than negative PQL, report sample results if > 10x the absolute value of the blank result, otherwise redigest and analyze.	Analyst, Supervisor, QA Manager	Accuracy/bias, Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
ICS (ICP only)	At beginning and end of run	ICS-A: For Al, Ca, Fe, and Mg, recovery within ± 20% of true value. For analytes not spiked, ± PQL, or, if PQL ≤ 0.01 mg/L, ± 2x PQL. ICS-AB: Recovery of each analyte within ± 20% of true value.	Do not use results for failing elements. Investigate and correct problem.	Analyst, Supervisor, QA Manager	Accuracy/Bias	ICS-A: Unspiked analytes ≤ 2x MDL, if interferent concentrations in sample > 50% interferent concentration in ICS-A solution. ICS-AB: Recovery of each analyte within ± 20% of true value.
Serial Dilution (L)	One per prep batch of twenty or fewer samples of similar matrix	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag results for affected analytes for all associated samples with "E".	Analyst, Supervisor, QA Manager	Accuracy/Bias, Precision	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 15% of the original result.

Matrix	Soil/Sediment
Analytical Group	Metals (ICP-AES)
Analytical Method/ SOP Reference	SW846 6010B/ CA-608

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	One per prep batch of twenty or fewer samples of similar matrix	Recovery within vendor supplied limits	Redigest and reanalyze all associated samples for affected analyte.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Recovery within vendor supplied limits
Laboratory Duplicate	One per prep batch of twenty or fewer samples of similar matrix	≤ 20% RPD	Narrate any results that are outside control limits	Analyst, Supervisor, QA Manager	Precision	Values ≥ 5xPQL: RPD ± 35% Values < 5xPQL: Absolute Difference ≤ 2x PQL
MS Sample	One per prep batch of twenty or fewer samples of similar matrix	Recovery ± 25 % of true value if sample < 4x spike value	Flag results for affected analytes for all associated samples with "N",	Analyst, Supervisor, QA Manager	Accuracy/Bias	Recovery ± 25 % of true value if sample < 4x spike value
Post-Digestion Spike	One per prep batch of twenty or fewer samples of similar matrix	Recovery within 75-125 % expected	"J" flag all sample results for specific analyte for all samples associated with the post digestion spike addition.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Recovery ± 25 % of true value
Temperature Blank	One per cooler	4° C ± 2° C	Laboratory will notify TtNUS Project Manager (PM) of temperatures outside criteria. TtNUS PM will respond whether to proceed with analysis.	Laboratory Quality Manager and TtNUS Project Manager.	Accuracy/bias/ representativeness	4° C ± 2° C

SAP Worksheet #28h -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Aqueous (SW, GW)
Analytical Group	Metals (ICP-MS)
Analytical Method/ SOP Reference	SW846 6020/ CA-627

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
ICV	Immediately after calibration	± 10 %	Correct problem, recalibrate and reanalyze ICV	Analyst, Supervisor, QA Manager	Accuracy/bias	± 10 %
ICB	Immediately after the ICV	≤ PQL	Correct problem, recalibrate and reanalyze ICV and ICB	Analyst, Supervisor, QA Manager	Accuracy/bias, Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
PQL Standard for ICPMS (PQL)	At the beginning of a sample run, after every 20 samples and at the end of the run	80-120 % recovery.	(1) Reanalyze immediately for failing elements only. (2) Terminate analysis, correct problem, recalibrate and reanalyze all analytical samples analyzed since last good PQL Std.	Analyst, Supervisor, QA Manager	Sensitivity	70-130 % recovery .
PBW	One per prep batch of twenty or fewer samples of similar matrix	Absolute value < PQL.	(1) If blank value > PQL report sample results if < PQL or > 10 x the blank value; otherwise redigest and analyze. (2) If blank value is less than negative PQL, report sample results if > 10x the absolute value of the blank result, otherwise redigest and analyze.	Analyst, Supervisor, QA Manager	Accuracy/bias, Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
ICS (ICP only)	CS-A: Before beginning a sample run, and every 12 hours during a run. ICS-AB: Before beginning a sample run, and every 12 hours during a run.	ICS-A: ± PQL unless otherwise specified by client. ICS-AB: ± 20% of true value	None established.	Analyst, Supervisor, QA Manager	Accuracy/Bias	ICS-A: Unspiked analytes ≤ 2x MDL, if interferent concentrations in sample > 50% interferent concentration in ICS-A solution. ICS-AB: Recovery of each analyte within ± 20% of true value.

Matrix	Aqueous (SW, GW)
Analytical Group	Metals (ICP-MS)
Analytical Method/ SOP Reference	SW846 6020/ CA-627

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Serial Dilution (L)	One per prep batch of twenty or fewer samples of similar matrix	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag results for affected analytes for all associated samples with "E".	Analyst, Supervisor, QA Manager	Accuracy/Bias, Precision	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 15% of the original result.
LCSW	One per prep batch of twenty or fewer samples of similar matrix	Recovery within ± 20% of true value.	Redigest and reanalyze all associated samples for affected analyte.	Analyst, Supervisor, QA Manager	Accuracy/Bias	80-120 % recovery.
Laboratory Duplicate	One per prep batch of twenty or fewer samples of similar matrix	≤ 20% RPD	Narrate any results that are outside control limits	Analyst, Supervisor, QA Manager	Precision	Values ≥ 5xPQL: RPD ± 20% Values < 5xPQL: Absolute Difference ≤ PQL
MS Sample	One per prep batch of twenty or fewer samples of similar matrix	Recovery ± 25 % of true value if sample < 4x spike value	Flag results for affected analytes for all associated samples with "N".	Analyst, Supervisor, QA Manager	Accuracy/Bias	Recovery ± 25 % of true value if sample < 4x spike value
Post-Digestion Spike	When serial dilution fails or analyte concentration for all samples < 100 x MDL.	Recovery ± 25% of true value, if sample < 4x spike added.	"J" flag all sample results for specific analyte for all samples associated with the post digestion spike addition.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Recovery ± 25 % of true value
IS	IS Appropriate IS required for all analytes in all samples. Mass of IS must be <50 amu different from that of analyte	For each sample, IS intensity within 30%-120% of that of initial calib. standard. For ICV, ICB, CCV, and CCB, IS intensity within 80%-120% of that in initial calib. standard.	Reanalyze affected samples	Analyst, Supervisor, QA Manager	Accuracy/bias	For each field sample, IS intensity within 30%-120% of that of initial calib. standard.
Temperature Blank	One per cooler	4° C ± 2° C	Laboratory will notify TtNUS Project Manager (PM) of temperatures outside criteria. TtNUS PM will respond whether to proceed with analysis.	Laboratory Quality Manager and TtNUS Project Manager.	Accuracy/bias/ representativeness	4° C ± 2° C

SAP Worksheet #28i -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Soil/Sediment
Analytical Group	Metals (ICP-MS)
Analytical Method/ SOP Reference	SW846 6020/ CA-627

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
ICV	Immediately after calibration	± 10 %	Correct problem, recalibrate and reanalyze ICV	Analyst, Supervisor, QA Manager	Accuracy/bias	± 10 %
ICB	Immediately after the ICV	≤ PQL	Correct problem, recalibrate and reanalyze ICV and ICB	Analyst, Supervisor, QA Manager	Accuracy/bias, Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
PQL Standard for ICPMS (PQL)	At the beginning of a sample run, after every 20 samples and at the end of the run	80-120 % recovery.	Reanalyze immediately for failing elements only. Terminate analysis, correct problem, recalibrate and reanalyze all analytical samples analyzed since last good PQL Std.	Analyst, Supervisor, QA Manager	Sensitivity	70-130 % recovery .
PBW	One per prep batch of twenty or fewer samples of similar matrix	Absolute value < PQL.	If blank value > PQL report sample results if < PQL or > 10 x the blank value; otherwise redigest. If blank value is less than negative PQL, report sample results if > 10x the absolute value of the blank result, otherwise redigest.	Analyst, Supervisor, QA Manager	Accuracy/bias, Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
ICS (ICP only)	ICS-A: Before beginning a sample run, and every 12 hours during a run. ICS-AB: Before beginning a sample run, and every 12 hours during a run.	ICS-A: ± PQL unless otherwise specified by client. ICS-AB: ± 20% of true value	None established.	Analyst, Supervisor, QA Manager	Accuracy/Bias	ICS-A: Unspiked analytes ≤ 2x MDL, if interferent concentrations in sample > 50% interferent concentration in ICS-A solution. ICS-AB: Recovery of each analyte within ± 20% of true value.
Serial Dilution (L)	One per prep batch of twenty or fewer samples of similar matrix	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag results for affected analytes for all associated samples with “E”.	Analyst, Supervisor, QA Manager	Accuracy/Bias, Precision	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 15% of the original result.

Matrix	Soil/Sediment
Analytical Group	Metals (ICP-MS)
Analytical Method/ SOP Reference	SW846 6020/ CA-627

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCSW	One per prep batch of twenty or fewer samples of similar matrix	Recovery within vendor supplied limits	Redigest and reanalyze all associated samples for affected analyte.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Recovery within vendor supplied limits
Laboratory Duplicate	One per prep batch of twenty or fewer samples of similar matrix	≤ 20% RPD	Narrate any results that are outside control limits	Analyst, Supervisor, QA Manager	Precision	Values ≥ 5xPQL: RPD ± 35% Values < 5xPQL: Absolute Difference ≤ 2x PQL
MS Sample	One per prep batch of twenty or fewer samples of similar matrix	Recovery ± 25 % of true value if sample < 4x spike value	Flag results for affected analytes for all associated samples with "N".	Analyst, Supervisor, QA Manager	Accuracy/Bias	Recovery ± 25 % of true value if sample < 4x spike value
Post-Digestion Spike	When serial dilution fails or analyte concentration for all samples < 100 x MDL.	Recovery ± 25% of true value, if sample < 4x spike added.	"J" flag all sample results for specific analyte for all samples associated with the post digestion spike addition.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Recovery ± 25 % of true value
IS	Appropriate IS required for all analytes in all samples. Mass of IS must be <50 amu different from that of analyte	For each sample, IS intensity within 30%-120% of that of initial calib. standard. For ICV, ICB, CCV, and CCB, IS intensity within 80%-120% of that in initial calib. standard.	Reanalyze affected samples	Analyst, Supervisor, QA Manager	Accuracy/bias	For each field sample, IS intensity within 30%-120% of that of initial calib. standard.
Temperature Blank	One per cooler	4° C ± 2° C	Laboratory will notify TtNUS Project Manager (PM) of temperatures outside criteria. TtNUS PM will respond whether to proceed with analysis.	Laboratory Quality Manager and TtNUS Project Manager.	Accuracy/bias/ representativeness	4° C ± 2° C

SAP Worksheet #28j -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Aqueous (SW, GW)
Analytical Group	Metals (Mercury)
Analytical Method/ SOP Reference	7470A/CA-615

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
ICV	Immediately after calibration	± 10 %	Correct problem, recalibrate and reanalyze ICV	Analyst/Supervisor, QA Manager	Accuracy/bias	± 10 %
ICB	Immediately after the ICV	≤ PQL	Correct problem, recalibrate and reanalyze ICV and ICB	Analyst/Supervisor, QA Manager	Accuracy/bias, Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
PQL Standard (PQL)	At the beginning of a sample run, after every 20 samples and at the end of the run	Recovery within 50% - 150 % of true value.	Terminate analysis, correct problem, recalibrate and reanalyze all analytical samples analyzed since last good PQL Std.	Analyst/Supervisor, QA Manager	Sensitivity	70-130 % recovery .
PBW	One per prep batch of twenty or fewer samples of similar matrix	Absolute value < PQL.	If blank value > PQL report sample results if < PQL or > 10 x the blank value; otherwise redigest and analyze. 2. If blank value is less than negative PQL, report sample results if > 10x the absolute value of the blank result, otherwise redigest and analyze.	Analyst/Supervisor, QA Manager	Accuracy/bias, Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
Serial Dilution (L)	One per prep batch of twenty or fewer samples of similar matrix	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag results for affected analytes for all associated samples with “E”.	Analyst/Supervisor, QA Manager	Accuracy/Bias, Precision	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 15% of the original result.
LCSW	One per prep batch of twenty or fewer samples of similar matrix	Recovery within ± 20% of true value.	Redigest and reanalyze all associated samples.	Analyst/Supervisor, QA Manager	Accuracy/Bias	80-120 % recovery.
Laboratory Duplicate	One per prep batch of twenty or fewer samples of similar matrix	RPD ± 20%	Flag results for affected analytes for all associated samples with “*”.	Analyst/Supervisor, QA Manager	Precision	Values ≥ 5xPQL: RPD ± 20% Values < 5xPQL: Absolute Difference ≤ PQL
MS (S)	One per prep batch of twenty or fewer samples of similar matrix	Recovery ± 25 % of true value if sample < 4x spike value	Flag results for affected analytes for all associated samples with ”N”,	Analyst/Supervisor, QA Manager	Accuracy/Bias	Recovery ± 25 % of true value if sample < 4x spike value
Temperature Blank	One per cooler	4° C ± 2° C	Laboratory will notify TtNUS Project Manager (PM) of temperatures outside criteria. TtNUS PM will respond whether to proceed with analysis.	Laboratory Quality Manager and TtNUS Project Manager.	Accuracy/bias/ representativeness	4° C ± 2° C

SAP Worksheet #28k -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Soil/Sediment
Analytical Group	Metals (Mercury)
Analytical Method/ SOP Reference	7471A/CA-611

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
ICV	Immediately after calibration	± 10 %	Correct problem, recalibrate and reanalyze ICV	Analyst/Supervisor, QA Manager	Accuracy/bias	± 10 %
ICB	Immediately after the ICV	≤ PQL	Correct problem, recalibrate and reanalyze ICV and ICB	Analyst/Supervisor, QA Manager	Accuracy/bias, Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
PQL Standard (PQL)	At the beginning of a sample run, after every 20 samples and at the end of the run	Recovery within 50% - 150 % of true value.	Terminate analysis, correct problem, recalibrate and reanalyze all analytical samples analyzed since last good PQL Std.	Analyst/Supervisor, QA Manager	Sensitivity	70-130 % recovery .
PBW	One per prep batch of twenty or fewer samples of similar matrix	Absolute value < PQL.	If blank value > PQL report sample results if < PQL or > 10 x the blank value; otherwise redigest and analyze. 2. If blank value is less than negative PQL, report sample results if > 10x the absolute value of the blank result, otherwise redigest and analyze.	Analyst/Supervisor, QA Manager	Accuracy/bias, Contamination	No target analytes > ½ QL. Exception: for common laboratory contaminants, no target analytes > QL.
Serial Dilution (L)	One per prep batch of twenty or fewer samples of similar matrix	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag results for affected analytes for all associated samples with "E".	Analyst/Supervisor, QA Manager	Accuracy/Bias, Precision	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 15% of the original result.
LCSS	One per prep batch of twenty or fewer samples of similar matrix	Recovery within vendor supplied limits	Redigest and reanalyze all associated samples for affected analyte (except Ag and Sb)	Analyst/Supervisor, QA Manager	Accuracy/Bias	Recovery within vendor supplied limits
Laboratory Duplicate	One per prep batch of twenty or fewer samples of similar matrix	RPD ± 20%	Flag results for affected analytes for all associated samples with "*".	Analyst/Supervisor, QA Manager	Precision	Values ≥ 5xPQL: RPD ± 35% Values < 5xPQL: Absolute Difference ≤ 2x PQL
MS (S)	One per prep batch of twenty or fewer samples of similar matrix	Recovery ± 25 % of true value if sample < 4x spike value	Flag results for affected analytes for all associated samples with "N",	Analyst/Supervisor, QA Manager	Accuracy/Bias	Recovery ± 25 % of true value if sample < 4x spike value
Temperature Blank	One per cooler	4° C ± 2° C	Laboratory will notify TtNUS Project Manager (PM) of temperatures outside criteria. TtNUS PM will respond whether to proceed with analysis.	Laboratory Quality Manager and TtNUS Project Manager.	Accuracy/bias/ representativenes s	4° C ± 2° C

SAP Worksheet #28I -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Groundwater
Analytical Group	Chloride and Bromide
Analytical Method/ SOP Reference	EPA Method 300.0/CA-742

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep/analysis batch of 20 or fewer samples	No analyte detected ≥PQL	(1) Investigate source of contamination (2) Report all sample results <PQL. (3) Report sample results >10X the blank result and flag results with a "B". (4) Reanalyze all other samples associated with the failing blank.	Analyst, Department Manager	Accuracy/bias, Contamination	No analyte detected ≥PQL
LCS	One per prep/analysis batch of 20 or fewer samples, prepared from a separate source than calibration standards	90%-110% Recovery	(1) If the LCS fails high, report samples that are <PQL. (2) Recalibrate and/or reanalyze other samples.	Analyst, Department Manager	Accuracy/Bias	80%-120% Recovery
MS	One for every set of 20 samples.	90%-110% Recovery	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. (3) Notate sample result in raw data if matrix interference suspected.	Analyst, Department Manager	Accuracy/Bias	80%-120% Recovery
MSD	One per 20 samples	90%-110% Recovery RPD ≤20%	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD still out, report original result with notation or narration.	Analyst, Department Manager	Accuracy/Bias Precision	80%-120% Recovery RPD ≤20%
Temperature Blank	One per cooler	4° C ± 2° C	Laboratory will notify TtNUS Project Manager (PM) of temperatures outside criteria. TtNUS PM will respond whether to proceed with analysis.	Laboratory Quality Manager and TtNUS Project Manager.	Accuracy/bias/ representativeness	4° C ± 2° C

TABLE 28A -- KATAHDIN ANALYTICAL SERVICES ACCURACY AND PRECISION LIMITS

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)
Pesticides					
4,4'-DDD	8081	61-127	60-120	0-50	0-30
4,4'-DDE	8081	69-120	63-124	0-50	0-30
4,4'-DDT	8081	64-133	68-127	0-50	0-30
Aldrin	8081	61-117	59-116	0-50	0-30
Alpha-BHC	8081	61-108	62-116	0-50	0-30
Alpha-Chlordane	8081	64-121	66-124	0-50	0-30
Beta-BHC	8081	68-124	75-122	0-50	0-30
Delta-BHC	8081	53-145	66-142	0-50	0-30
Dieldrin	8081	66-122	66-126	0-50	0-30
Endosulfan I	8081	58-108	56-118	0-50	0-30
Endosulfan II	8081	63-116	64-118	0-50	0-30
Endosulfan Sulfate	8081	58-144	65-138	0-50	0-30
Endrin	8081	63-118	67-119	0-50	0-30
Endrin Aldehyde	8081	54-105	68-120	0-50	0-30
Endrin Ketone	8081	71-132	71-131	0-50	0-30
Gamma-BHC(Lindane)	8081	67-116	65-124	0-50	0-30
Gamma-Chlordane	8081	68-121	68-126	0-50	0-30
Heptachlor	8081	59-118	60-114	0-50	0-30
Heptachlor Epoxide	8081	64-119	64-123	0-50	0-30
Methoxychlor	8081	68-129	64-129	0-50	0-30
Toxaphene	8081	---	---	0-50	0-30
Surrogates:					
Tetrachloro-m-xylene	8081	42-104	48-112	---	---
Decachlorobiphenyl	8081	51-119	41-131	---	---

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)
Chlorinated Herbicides					
2,4-D	8151	30-120	82-141	0-50	0-30
2,4-DB	8151	30-120	72-126	0-50	0-30
2,4,5-TP (Silvex)	8151	30-120	77-125	0-50	0-30
2,4,5-T	8151	30-120	64-145	0-50	0-30
Dalapon	8151	30-120	42-117	0-50	0-30
Dicamba	8151	30-120	75-115	0-50	0-30
Dichloroprop	8151	30-120	78-124	0-50	0-30
Dinoseb	8151	30-120	10-142	0-50	0-30
MCPA	8151	30-120	77-123	0-50	0-30
MCPP	8151	30-120	84-128	0-50	0-30
Surrogates:					
2,4-Dichlorophenylacetic acid	8151	44-119	35-126	---	---
Semivolatile Organic Compounds					
2-Methylnaphthalene	8270C	42-122	43-112	0-50	0-30
Acenaphthene	8270C	64-106	60-111	0-50	0-30
Acenaphthylene	8270C	59-108	47-118	0-50	0-30
Anthracene	8270C	56-114	58-118	0-50	0-30
Benzo(a)anthracene	8270C	65-111	64-122	0-50	0-30
Benzo(a)pyrene	8270C	62-114	61-124	0-50	0-30
Benzo(b)fluoranthene	8270C	47-121	54-121	0-50	0-30
Benzo(g,h,i)perylene	8270C	53-126	55-144	0-50	0-30
Benzo(k)fluoranthene	8270C	37-130	50-126	0-50	0-30
Chysene	8270C	66-107	61-119	0-50	0-30
Dibenzo(a,h)anthracene	8270C	54-133	60-149	0-50	0-30
Fluoranthene	8270C	70-115	11-104	0-50	0-30
Fluorene	8270C	57-108	61-112	0-50	0-30

Target List	EPA Method	Soil Accuracy % Recovery	Aqueous Accuracy % Recovery	Soil Precision RPD (1)	Aqueous Precision RPD (1)
Indeno(1,2,3-cd)pyrene	8270C	55-128	45-153	0-50	0-30
Naphthalene	8270C	55-99	40-99	0-50	0-30
Phenanthrene	8270C	61-117	60-120	0-50	0-30
Pyrene	8270C	54-131	63-123	0-50	0-30
Surrogates:					
2-Fluorophenol	8270C	43-99	12-62	---	---
Phenol-d6	8270C	53-98	10-42	---	---
Nitrobenzene-d5	8270C	47-100	50-88	---	---
2-Fluorobiphenyl	8270C	49-114	44-109	---	---
2,4,6-Tribromophenol	8270C	44-111	32-109	---	---
Terphenyl-d14	8270C	58-140	61-116	---	---
Semivolatile Organic SIM Compounds					
Benzo(a)anthracene	8270C	50-138	48-120	0-50	0-30
Benzo(a)pyrene	8270C	52-113	63-113	0-50	0-30
Benzo(b)fluoranthene	8270C	44-119	49-104	0-50	0-30
Dibenzo(a,h)anthracene	8270C	30-134	30-136	0-50	0-30
Indeno(1,2,3-cd)pyrene	8270C	32-134	30-140	0-50	0-30
Phenanthrene	8270C	58-122	57-119	0-50	0-30
Pyrene	8270C	33-133	64-121	0-50	0-30
Surrogates:					
2-Methylnaphthalene-D10	8270C	33-125	34-110	---	---
Fluorene-D10	8270C	53-136	46-122	---	---
Pyrene-D10	8270C	16-162	34-134	---	---

SAP Worksheet #29 -- Project Documents and Records Table

(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
Sample Collection Documents and Records: Sampling and Analysis Plan Field Sampling SOPs Field logbook (and sampling notes) Field sample forms (e.g. boring logs, sample logsheets, etc.) Chain of custody records Sample shipment airbills Equipment calibration logs Photographs Field Task Modification Forms	TtNUS project file, results will be discussed in subject document.
Laboratory Documents and Records Sample receiving records Sample preparation logs Extraction/clean-up records Standard traceability logs Equipment Calibration logs Sample analysis run logs Equipment maintenance, testing , and inspection logs CA forms Reported field sample results Reported results for standards, quality control checks, and quality control samples Data completeness checklists Sample storage and disposal records Telephone logs Raw data	TtNUS project file, long term data package storage

Document	Where Maintained
Data Assessment Documents and Records Field Sampling Audit Checklist (if an audit is conducted) Analytical Audit Checklist (if an audit is conducted) CA response records (if needed) Data Validation Memoranda	TtNUS project file, results will be discussed in subject document.

Note: See next page for a description of data handling, management, tracking, and control.

Data Handling, Management, Tracking, and Control

- 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 Study, 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 Project Manager (or designee) is responsible for the overall tracking and control of data generated for the project.

  - Data Tracking. Data are tracked from generation to archiving in the TtNUS project-specific files. The 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.

SAP Worksheet #30 -- Analytical Services Table

(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Sample Locations/ ID Number	Analytical Method	Data Package Turnaround Time (days)	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
Surface Soil/ Subsurface Soil/ Sediment/ Surface water	PAHs	See Worksheet #18	SW-846 Method 8270C	21	Katahdin Analytical Services Andrea Colby 207-874-2400	Not Applicable
Surface Soil/ Sediment	TCL Pesticides	See Worksheet #18	SW-846 Method 8081A	21	Katahdin Analytical Services Andrea Colby 207-874-2400	Not Applicable
Surface Soil/ Sediment	Herbicides	See Worksheet #18	SW-845 Method 8151A	21	Katahdin Analytical Services Andrea Colby 207-874-2400	Not Applicable
Surface Soil/ Subsurface Soil/ Sediment	TOC	See Worksheet #18	SW-845 Method 9060A	21	Katahdin Analytical Services Andrea Colby 207-874-2400	Not Applicable
Sediment	Grain Size	See Worksheet #18	ASTM D422-63	21	Katahdin Analytical Services[1] Andrea Colby 207-874-2400	Not Applicable
Surface Soil/ Subsurface Soil/ Sediment	TAL Metals	See Worksheet #18	SW-845 Methods 6010B and 6020	21	Katahdin Analytical Services Andrea Colby 207-874-2400	Not Applicable
Groundwater/ Surface water	TAL Metals (Total and Dissolved)	See Worksheet #18	SW-845 Methods 6010B and 6020	21	Katahdin Analytical Services Andrea Colby 207-874-2400	Not Applicable
Groundwater	Chloride and Bromide	See Worksheet #18	EPA Method 300.0	21	Katahdin Analytical Services Andrea Colby 207-874-2400	Not Applicable

Notes:

1 – Katahdin Analytical Services will subcontract for these analytical services with TestAmerica Burlington, 30 Community Drive, Suite 11, South Burlington, VT 05403

Data package deliverables are detailed in the Analytical Technical Specification included in Appendix C. Data packages will be provided as both hardcopy and portable document format (.PDF). Katahdin Analytical Services will provide a Naval Installation Restoration Information Solutions (NIRIS) compatible electronic data deliverable (EDD). Data packages will be contract laboratory program (CLP)-equivalent (i.e., they will contain CLP-equivalent summary forms and raw data). Data will be stored by the analytical laboratory for five years.

SAP Worksheet #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 CAs (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Field Supervision	Daily during sampling events	Internal	TtNUS	TtNUS FOL	TtNUS FOL	TtNUS FOL and Field Crew	TtNUS FOL, SM, PM, QA Manager
Project Supervision	Every sampling	Internal	TtNUS	PM (Jeff Orient), TtNUS	TtNUS FOL	PM (Jeff Orient) TtNUS FOL	PM (Jeff Orient) TtNUS FOL
Field Sampling System Audit	1 per contract year	Internal	TtNUS	TBD	PM (Jeff Orient) TtNUS FOL	Auditor, SM, and PM	TtNUS QA Manager (Tom Johnston)
Laboratory System Audit	Every 18 months	External	NFESC	NFESC	Laboratory QA Manager or Laboratory Manager	Laboratory Manager and QA Manager	NFESC

System audits will be performed as appropriate to ensure that the work is being implemented in accordance with the approved project SOPs and in an overall satisfactory manner.

- The TtNUS FOL will supervise the field operations. The FOL will perform a daily check to ensure that the field instruments are calibrated, equipment is properly decontaminated, samples are collected and handled properly, and the fieldwork is accurately and neatly documented. CAs will be implemented immediately if any con-compliance is detected.
- System audits for the laboratory will be performed regularly and in accordance with NFESC guidance and Department of Defense Quality Systems Manual for Environmental Laboratory (DoD QSM) (DoD, 2006), as provided in the Laboratory QA Plan (LQAP).
- The data validator(s) will review the data to ensure that the analytical results were obtained through the approved methodology, and that the appropriate levels of QC were followed. The data review effort will be supervised by the TtNUS Project Chemist.

The Project Manager will oversee the FOL and data reviewer, and check that management of the acquired data proceeds in an organized and expeditious manner.

An independent performance audit of field activities may be conducted at the discretion of and under the direction of the CLEAN QA officer. If a formal field audit is conducted, the QA officer will check that sample collection, handling, and shipping protocols, as well as equipment decontamination and field documentation procedures, are being performed in accordance with the approved project planning documents and SOPs. These audits and laboratory systems audits will identify the following:

- The assessed entity (e.g., field crew, office personnel, etc. and the associated project, field event, office, etc.)
- Whether the audit is internal or external
- Location and date(s) of assessment
- Assessment team members
- Type of assessment
- Scope of assessment
- Documents to be reviewed
- Notification dates
- Proposed assessment schedule
- Assessment number
- Contract number

Performance audits of laboratory are coordinated through NFESC and are conducted every 18 months by NFESC's independent quality assurance contractor.

Assessment findings that require CA initiate a sequence of events that include documentation of deficiencies, notification of findings, request for CA, implementation of CA, and follow-up assessment of the CA effectiveness. The procedures for handling any QAPP deviations and project deficiencies that are identified through the planned project assessments are summarized in Worksheet #32.

Potential problems may involve nonconformance with the SOPs and/or analytical procedures established for the project or other unforeseen difficulties. Any person identifying a condition adverse to project quality will notify the PM. The PM, with the assistance of the QA officer, will be responsible for developing and initiating appropriate CA. If the identified deficiencies involve field work, this will be done through the FOL; if the deficiencies involve the laboratory, this will be done through the laboratory project manager. The CAs will require follow through to the point of verifying that the CA has been effective. CAs may include re-sampling and/or reanalyzing samples or amending or adjusting project procedures. If warranted by the severity of the problem (for example, if a change in the approved plan is required), the Navy will be notified in writing and the Navy's approval will be obtained before any change is implemented. Minor changes will be documented for the main file by the TtNUS PM. Additional work that depends on a nonconforming activity will not be performed until the problem has been eliminated. The overall CA responsibility for system audits will reside with the PM. The overall CA responsibility for field audits will reside with the QA officer.

SAP Worksheet #32 -- Assessment Findings and CA 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 CA Response Documentation	Individual(s) Receiving CA Response (name, title, organization)	Timeframe for Response
Field Supervision	Site Log Book and sample collection logs	TtNUS PM (Jeff Orient) and FOL	Immediately	Entry in site log book	PM (Jeff Orient), TtNUS, FOL	24 hours
Project Supervision	Written report	Program Manager (John Trepanowsky), TtNUS Deputy Program Manager (Garth Glenn), TtNUS	Monthly	Written memo	Program Manager (John Trepanowsky), TtNUS Deputy Program Manager (Garth Glenn), TtNUS	Within a week of notification
Field Sampling System Audit (if performed)	Audit checklist and written audit finding summary	PM (Jeff Orient), TtNUS FOL TtNUS, and Program management TtNUS	Dependant on the finding, if major a stop work may be issue immediately, however if minor within 1 week of audit	Written memo	QAM (Tom Johnston),TtNUS Auditor TtNUS Program Manager TtNUS	Within 48 hours of notification
Laboratory System Audit (if performed)	Written audit report	Laboratory Manager, Lab QA Manager	Not specified by NFESC	Letter	NFESC	Specified by NEFSC

SAP Worksheet #33 -- QA Management Reports Table

(UFP QAPP Manual Section 4.2)

Type of Report	**Frequency** (daily, weekly monthly, quarterly, annually, etc.)	**Projected Delivery Date(s)**	**Person(s) Responsible for Report Preparation** (title and organizational affiliation)	**Report Recipient(s)** (title and organizational affiliation)
Data validation report	Per Sample Delivery Group (SDG)	Within 3 weeks of receipt of laboratory data	Project Chemist (Lucy Guzman), TtNUS	PM (Jeff Orient), TtNUS Project file
Major analysis problem identification (Internal Memorandum)	When persistent analysis problems are detected	Immediately upon detection of problem	QA Manager (TtNUS)	PM (TtNUS), QAM (TtNUS), Program Manager (TtNUS), project file
Project monthly progress report	Monthly for duration of the project	Monthly	PM (Jeff Orient), TtNUS	Navy, project file
Field progress reports	Daily, oral, during the course of sampling	Every day that field sampling is occurring	FOL (TtNUS)	PM (Jeff Orient), TtNUS
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately upon detection of problem	Subcontracted laboratory	TtNUS PM, project file

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

(UFP-QAPP Manual Section 5.2.1)

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-custody forms	Chain-of-custody forms will be checked against the samples collected to verify complete and accurate information. 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. See SOP SA-6.3	Internal	TtNUS sampler and FOL
SAP Sample tables	Verify that all proposed samples listed in the SAP tables have been collected	Internal	TtNUS, FOL or designee
Sample log sheets	Verify that information recorded in the log sheets is accurate and complete	Internal	TtNUS, FOL or designee
Sample coordinates	Verify that sample locations are correct and in accordance with the SAP proposed locations.	Internal	TtNUS, FOL or designee
Field QC samples	Check that field QC samples listed in Worksheet 20 were collected as required	Internal	TtNUS, FOL or designee
Chain-of-custody forms	The TtNUS FOL or designee will review and sign the chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory. The laboratory sample custodian will review that the sample shipment is complete and all samples are received and 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.	Internal/ External	1 - TtNUS FOL or designee 2 - Laboratory sample custodian 3 - TtNUS, data validators
Analytical data package	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The laboratory QA Manager will sign the case narrative for each data package.	External	Katahdin QAM
Analytical data package	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	Internal	TtNUS, data validators
Electronic data deliverables	The electronic data will be verified against the chain-of-custody and hard copy data package for accuracy and completeness	Internal	TtNUS, data validators

Verification includes field data verification and laboratory data verification. Verification inputs as per SAP Worksheet 34 will be checked.

SAP Worksheet #35 -- Validation (Steps IIa and IIb) Process Table

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

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Field SOPs	Ensure that all sampling SOPs were followed and any field deviation were documented	TtNUS PM, FOL, or designee
IIa	Analytical SOPs	Ensure that the laboratory followed the analytical SOPs cited in the SAP and any method deviation were approved by TtNUS and documented in the case narrative	TtNUS project chemist or data validators
IIa	Chain-of-custody	Ensure that the custody and integrity of the samples was maintained from collection to analysis and the custody records are complete and any deviations are recorded	TtNUS project chemist or data validators
IIa	Holding times	Verify that the samples were shipped and store at the required temperature and sample pH for chemically-preserved samples meet the requirements listed in Worksheet 19. Verify that the analyses were performed within the holding times listed in Worksheet 19	TtNUS project chemist or data validators
IIa	Data results	Verify the summary form results against the raw data. Check calculations for accuracy	TtNUS project chemist or data validators
IIa	Standards	Ensure that the standards used at the field and laboratory are traceable and meet the contract, method, or procedural requirements	TtNUS project chemist or data validators
IIa/IIb	Laboratory data results for accuracy	Ensure that the laboratory QC samples listed in Worksheet 28 were analyzed and that the measurement performance criteria listed in Worksheet 12 were met for all field samples and QC analyses. Verify that specified field QC samples were collected and analyzed and that the analytical quality control criteria set up for this project were met.	TtNUS project chemist or data validators
IIa/IIb	Field and laboratory duplicate analyses for precision	Verify the field sampling precision by checking the RPD for field duplicate samples. Verify the laboratory precision by checking the RPD or percent difference values from laboratory duplicate analyses; MS/MSD; and laboratory control sample/laboratory control sample duplicate. Ensure compliance with the methods and project Method Performance Criteria (MPC) accuracy goals listed in Worksheets 12	TtNUS project chemist or data validators
IIa/IIb	Sample results for representativeness	Verify that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from sample collection to analysis	TtNUS project chemist or data validators
IIb	PQLs for sensitivity	Verify that the PQLs listed in Worksheet #15 were achieved	TtNUS project chemist or data validators

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation (name, organization)
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 no-detected. Document this usability issue and inform the project manager.	TtNUS project chemist or data validators
IIb	Analytical data Deviations	Determine the impact of any deviation from sampling or analytical methods and SOPs requirements and matrix interferences effect on the analytical results.	TtNUS project chemist or data validators
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 data base 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	TtNUS project chemist or data validators

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

(UFP-QAPP Manual Section 5.2.2.1)

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Surface Soil/ Subsurface Soil/ Sediment/ Surface water	PAHs	Project specific criteria for SW-846 Method 8270C-SIM are listed in Worksheets #12, 15, 28. Tier II data validation by EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, December 1996 (U.S. EPA, 1996) will be applied using these criteria.	TtNUS, Project Chemist (L. Guzman and staff chemists)
IIa and IIb	Surface Soil/ Sediment	TCL Pesticides	Project specific criteria for SW-846 Method 8081 are listed in Worksheets #12, 15, 28. Tier II data validation by EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, February 2004 (U.S. EPA, 2004) will be applied using these criteria.	TtNUS, Project Chemist (L. Guzman and staff chemists)
IIa and IIb	Surface Soil/ Sediment	Herbicides	Project specific criteria for SW-846 Method 8151 are listed in Worksheets #12, 15, 28. Tier II data validation by EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, February 2004 (U.S. EPA, 2004) will be applied using these criteria.	TtNUS, Project Chemist (L. Guzman and staff chemists)
IIa and IIb	Surface Soil/ Subsurface Soil/ Sediment	TOC	Project specific criteria for SW-846 Method 9060 are listed in Worksheets #12, 15, 28. Tier II data validation by EPA-NE Data Validation Functional Guidelines for Evaluating Inorganic Analyses, 1989 (U.S. EPA, 1989) will be applied using these criteria.	TtNUS, Project Chemist (L. Guzman and staff chemists)
IIa and IIb	Surface Soil/ Subsurface Soil/ Sediment	TAL Metals	Project specific criteria for SW-846 Method 6010B/6020/7471A are listed in Worksheets #12, 15, 28. Tier II data validation by EPA-NE Data Validation Functional Guidelines for Evaluating Inorganic Analyses, 1989 (U.S. EPA, 1989) will be applied using these criteria.	TtNUS, Project Chemist (L. Guzman and staff chemists)
IIa and IIb	Groundwater/ Surface water/	TAL Metals (Total and Dissolved)	Project specific criteria for SW-846 Method 6010B/6020/7470A are listed in Worksheets #12, 15, 28. Tier II data validation by EPA-NE Data Validation Functional Guidelines for Evaluating Inorganic Analyses, 1989 (U.S. EPA, 1989) will be applied using these criteria.	TtNUS, Project Chemist (L. Guzman and staff chemists)
IIa and IIb	Groundwater	Chloride and Bromide	Project specific criteria for EPA Method 300.0 are listed in Worksheets #12, 15, 28. Tier II data validation by EPA-NE Data Validation Functional Guidelines for Evaluating Inorganic Analyses, 1989 (U.S. EPA, 1989) will be applied using these criteria.	TtNUS, Project Chemist (L. Guzman and staff chemists)

SAP Worksheet #37 -- Usability Assessment

(UFP-QAPP Manual Section 5.2.3)

USABILITY ASSESSMENT
The Project Manager will be responsible for the overall usability assessment. The Project Manager will assemble a team of professionals that will evaluate the overall data usability or specific portions of the data usability. The following items will be evaluated: • Evaluate whether the precision goals for field duplicate, and laboratory duplicate analyses were met. Specify the limitation on the use of the data when precision goals were exceed for limited or overall project data. • Evaluate whether the accuracy/bias goals were met for project data. This include a discussion of field and laboratory contamination, instrument calibration variability, recovery for surrogates, MS, laboratory control samples, dual column analysis for the pesticide identification, ICP interferences, and ICP serial dilution results. Specify data limitations and bias of the qualified data results; discuss the impact of these non-compliances on a specific data package or on the overall project data. • Discuss the representativeness of the data by assessing variation at the sampling point, a process condition, or and environmental condition. The usability report will discuss the representativeness of the data for each matrix, analysis, and concentration level performed. • Evaluate the comparability of the data generated under this project to other historical data generated by different methods and for samples collected using different procedures and under different weather of seasonal conditions. Discuss and compare overall comparability between data sets for each matrix, analytical group, and concentration level. This may include a comparison of field and laboratory precision if such as comparison is viewed by the TtNUS PM to be useful. • Evaluate whether the project sensitivity goals listed in Worksheet #15 were achieved for non-detected site contaminants. If sensitivity goals are exceeded, the limitations on the data use for this project will be evaluated. Discuss and compare the overall sensitivity and quantitation limits from multiple data sets for each matrix, analysis, and concentration level. • The usability report will discuss the overall completeness of multiple data sets collected for the project for each matrix, analysis, and concentration level. The report will discuss limitations on the use of the data if the goal for completeness are not achieved for the overall project or when the incompleteness is limited to a group of samples, analysis, matrix, or concentration level. • If verification and validation are not acceptable the data are qualified, the data are estimated (J, UJ) for minor QC deviations or the data are rejected for major QC. The usability assessment will discuss the impact of these data restrictions on the attainment of project objectives. Re-sampling may be necessary in cases of serious deficiencies. The use of estimated data will be discussed in the project report and the project team will determine whether resampling is necessary.

- For statistical comparisons and mathematical manipulations, non-detect values will be carefully handled as discussed in Section 11.6.5. For box plots, scatter plots, and preliminary probability plots used to display qualitative trends, non-detects will be denoted with a different symbol from detected values and represented by a concentration equal to one-half the sample-specific DL. Duplicate results (original and duplicate) will not be averaged for the purpose of representing the range of concentrations. However, the average of the original and duplicate will be used to represent the concentration at that sample location.

- Statistical calculation of the mean and lowest and highest detected concentration will be presented in summary tables in the report.

- Statistical tests will be conducted to identify potential outliers. Potential outliers will be removed if a review of the field and laboratory documentation indicates that the results are true outliers.

- Evaluate project assumptions that form the basis of the QAPP.

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

After completion of the data validation and review of the DQIs, the data will be reconciled with the DQOs and MPC to determine whether sufficient data of acceptable quality are available for compiling representative background data sets. A series of inspections and statistical analyses will be performed to estimate the data set characteristics. The statistical evaluations to be performed are described below in sections 37.1 – 37.6. The project team members identified by the project manager 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 attain project objectives. The data also will be evaluated to determine whether missing or rejected data can be compensated by other data.

Identify the personnel responsible for performing the usability assessment:

TtNUS Project Manager, Project Chemist, Project Hydrogeologist, Risk Assessors and others as designated by the Project Manager.

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, data qualifications such as estimation (J, UJ) or rejection(R) will be applied. 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 if necessary to fill the data gaps. The usability assessment will be presented to the NAVFAC and regulatory project managers for review and concurrence.

37.0 STATISTICAL DATA ASSESSMENT

37.1 Outlier Tests

All background data subsets will be evaluated for potential outliers before conducting any statistical comparisons between data groups. Candidate outliers will be investigated for systematic errors, reporting errors, and analytical bias and then located on a site map to check for any pattern of localized, anthropogenic contamination that is not representative of the background area as a whole. Dixon's outlier test will be applied to each background data subgroup (for example, different soil types) for those analytes displaying a normal distributional shape and having less than 25 samples (EPA, 2006a). Outliers will be screened by successively deleting the largest, second largest, and third largest data points from the set, computing the distributional fit and, if data are normally distributed, then performing Dixon's outlier test on the remaining data points versus the excluded data point. For cases where the analytical data for a substance do not fit a normal distribution, Tukey's nonparametric outlier test will be conducted by comparing each sample concentration to a critical value represented by the 75th quantile plus 3 times the interquartile range (IQR). All candidate outliers from either Dixon's test or Tukey's test will be labeled with the sample ID and highlighted on univariate box plots. Probability (Q-Q) plots will be used along with the Shapiro Wilk W-test to evaluate how well the data distribution matches a normal or lognormal shape, and whether any suspected outlier falls above the predicted line. A point of departure at the upper end of the probability plot may indicate that the candidate outlier does not belong to the same population distribution as the rest of the data.

After candidate outliers are identified based on statistical outlier tests, a scientific review and evaluation of the plausible reasons for outliers will be performed for each candidate. Candidate outliers will be located on a map to determine if elevated concentrations were co-located and/or possibly affected by localized anthropogenic contamination unrelated to any region-wide source. Correlation of elevated concentrations of analyte and other components/minerals (for example, iron, clay content, etc.) can identify that a suspected outlier actually represents an expected background pattern. The final decision to retain candidate extreme values will be based upon considerations whether the observed levels are plausibly consistent with natural conditions (within the range of naturally occurring metals concentrations) or whether associated samples are beyond the proximal range of influence from NAS Brunswick site activities and do not represent evidence of any localized source that is uniquely found within the background area and not plausible to consider as part of a region-wide source or pattern. EPA guidance QA-G9 (EPA, 2006a) further elaborates on the rationale for handling outliers: "Outliers may also represent true extreme values of a distribution (for instance, hot spots) and indicate more variability in the population than was expected." In addition, it is noted, "These [outlier] tests should only be used to identify

data points that require further investigation. The tests alone cannot determine whether a statistical outlier should be discarded or corrected within a data set; this decision should be based on judgmental or scientific grounds."

After completing the outlier tests, it will be necessary to further process the data to determine whether different data sets can be combined into fewer data sets that are larger and more robust for background comparisons. Although these evaluations are not strictly focused on the evaluation of data usability, documentation of the tests is important to ensure that the proper data evaluations are conducted. Therefore, the remainder of this worksheet is devoted to describing those tests and the documentation to be produced upon completion of the tests and identification of the background data sets. Which data analyses to conduct and which data presentations to prepare will be left largely to the professional judgment of the statistician with oversight by the TtNUS PM, NAVFAC RPM and regulatory agencies. The intent will be to thoroughly analyze the data in a cost effective manner to:

- determine the best method of data presentation
- develop the fewest defensibly representative data sets characteristic of uniquely different environmental background media groups in support of the widest array of environmental investigations to be conducted at potentially contaminated NAS Brunswick sites.

Because of the number of potentially different combinations of chemical parameter and background medium, it is not cost-effective to speculate on the process that will be universally applicable to all media and analyte groups. It is possible, however, to briefly describe the process for comparing site and background data sets and this is presented in Section 37.7.

37.2 Assessment of Distributional Shape

For each data subset (for example, soil type category), probability (Q-Q) plots and the Shapiro Wilk W-test will be used to evaluate how well the data distribution matches either a normal or lognormal shape, and whether any region of data points represented on the graph curves away from the predicted line. A non-linear bend in the Q-Q probability plot, a point of departure from linearity at one end of the probability plot, or a W-test score that is below the critical W-Table value all suggest that the observed data are not normally distributed, which can indicate that the data belong to a population having some other distributional form, the presence of multiple populations within the same data set, or the presence of outliers.

Normally distributed data exhibit a characteristic "bell-shape" curve that is symmetrical, whereas lognormal data have a skewed shape with a longer tail at the high-concentration end. The W-test will be performed twice – first using the original data, and then using the logarithms of the data. A five percent level of significance will be used to determine whether or not the data deviate from either hypothesized distribution. Data sets that conform to a normal distribution will be evaluated with parametric statistical tests whenever possible. Data that are non-normally distributed will be evaluated solely with non-parametric tests.

37.3 Graphical Exploratory Data Evaluation

Graphical presentations of the data (e.g., simple scatter plots, box plots, histograms, probability plots, or posting plots) may identify potential outliers, indicate the presence of more than one data population in a data set (population analysis), suggest that data gaps exist, and/or that there are spatial or temporal trends in the dataset. Each of these has the potential to confound data evaluations and statistical analysis designed to determine if site concentrations exceed background concentrations.

A multi-purpose plot consists of side-by-side univariate box plots for comparing each subcategory of data (for example, simultaneous box plots for different soil types). These plots will be shaded to highlight the interquartile range (25th to 75th percentile), annotated with the concentration of the median, include a vertical line (“whiskers”) that terminate at the maximum and minimum values, labeled with the sample IDs of any candidate outliers, and populated with distinct symbols for individual data points representing detects versus non-detects. This type of plot can reveal whether surface soil and subsurface soil are similar, if two soil types are similar, the extent and magnitude of candidate outliers, and whether a significant portion of the data set is non-detected and the range of DLs.

Probability (Q-Q) plots, as described above, augment the identification of outliers and identify any data points or data ranges that contribute to a poor fit to a normal distributional shape.

Histograms also depict the distributional shape, showing the mode, the relative frequency of detection at various concentration ranges, and the spread of data points near the upper end. These may be used in conjunction with probability plots to determine which data points contribute anomalies relative to the assumption of a normal bell curve shape.

Posting plots represent geographical base maps superimposed with sample locations and analytical results. These can illustrate contaminant transport pathways across an area, such as surficial drainage/flow paths or groundwater hydrological gradients. Examination of locations of hot spots relative to nearby samples can reveal whether an elevated concentration occurs over a contiguous area or if the hot spot is isolated.

Scatter plots visualize the relationship between two metals within a data subset. In some cases, a scatter plot can help explain the occurrence of seemingly anomalous elevated concentrations of one analyte, which might turn out to be related to a natural property of preferential accumulation associated with higher concentrations of another correlating substance or component. For example, enhanced absorption of arsenic may occur in minerals containing higher levels of iron, or absorption of certain metals and hydrophobic organics may be associated with soils containing a higher clay content.

37.4 Hypothesis Testing to Compare Background Data Groups

To test whether background data group concentrations are significantly different from each other, the site-wide hypothesis tests discussed in Section 11.5.3 and 11.5.4 will be applied given the input assumptions for alpha and beta listed in Sections 11.6.1 and 11.6.2. If the hypothesis is not rejected, then the subgroups of background data for a given substance can be combined. Given an appropriate outcome, it will be desirable to merge statistically similar data sets, because the use of a larger set of composite data will result in a lower chance of overlooking any significant difference that might exist in future comparisons between site and background populations (i.e. better statistical power).

Quantitative statistical tests that will be used to compare selected pairs of background data subgroups to each other including: three quantitative tests that look for overall differences between two populations of data values; a quantitative test that essentially looks for hot spots in one population relative to the other; and two tests that examine only the frequency of detection (proportion of detected versus non-detected values in one data set versus the other), but not the magnitude of values. Thus, measured statistical differences between populations can reflect either differences in central tendency, upper ranks, or frequency of detection.

As noted in Section 11.5.3, when comparing background data groups to each other, the hypothesis test form I will be used with a two-sided decision-making P-level of 0.05, and the overall conclusion (whether either soil data set's concentrations are greater than the other) will be assumed to be "yes" if any one of the quantitative tests that use concentrations conclude that data are elevated.

As noted in Section 11.5.4, if there are more than two background data groups to be compared, for normally distributed data, a parametric ANOVA test will be employed to determine if there is a difference somewhere among the means of the groups. If the Fisher F-statistic exceeds the critical value, then the ANOVA test concludes that at least one of the means among groups is different. To determine which of the group means are different, post-hoc comparisons will be performed using several types of tests having different degrees of conservatism in balancing the goals of controlling the individual test error rate and the overall family-wise error rate. Post-hoc tests may include t-tests (with or without the Bonferonni adjustment), Tukey's Honestly Significant Difference (HSD) test, and the Ryan-Einot-Gabriel-Welsch (REGW) test, the latter of which in many situations has more power than other techniques which control family-wise error.

When the background data groups do not meet the assumptions for a parametric ANOVA test, then the rank sums among groups will be compared using the Kruskal Wallis ANOVA test to determine if there is a difference between groups. If differences are found, then post-hoc nonparametric comparisons will be performed with and without Bonferonni adjustments to the alpha for each test.

37.5 Use of Final Background Data Sets for Evaluating Site Data

As discussed in Sections 11.5.5, 11.6.1, and 11.6.2, comparison of site-wide data sets to background will involve use of hypothesis test form II, with an alpha level and a beta level both set equal to 0.05 and a MDD of 2 standard deviations. Site to background statistical comparisons will not be employed in a manner inconsistent with EPA or Navy policy with regards to site decision making steps. In particular, the EPA process for selecting COPCs will be performed first, without regards to background, wherever this is required on risk assessments or other projects.

If site-wide tests cannot be used because the individual site wells do not belong to one homogeneous population, or because there are too few samples to achieve the desired statistical power, then hypothesis test form I will be applied using individual comparison tests. BTVs will be based upon UPLs so that a single sample can be compared to background. However, there may be a need to examine additional lines of evidence in order to reach a confident decision on background.

37.6 Geochemical Predictions

As discussed in Section 11.5.5, soil background data will be evaluated by means of scatter plots and correlation coefficients to assess possible geochemical correlations that may be utilized to predict trace metal concentrations based on an observed linear dependence on another mineral component. In some cases, a

factor analysis may provide a more accurate representation of geochemical predictions than a single element regression. The upper 95 percent confidence limits from geochemical regressions will be tabulated and used to select the optimal prediction equation and predictor metal(s), the most appropriate weighting factors to achieve good coverage and accurate and precise upper prediction limits, and limited probability of outliers among the various background soil types included in the regression.

Factor analysis can be interpreted as a mathematical method that attempts to reproduce an entire data set of sample results by extracting certain common factors, each of which is represented by a linear combination of the different variables (in this case metals concentrations) (Malinowski, 1991, and Sharaf, 1986). This is analogous to each sample being composed of a combination of various amounts from each mineral (factor) component, although the extracted factors may or may not actually represent pure mineral components because other properties such as cation exchange capacity of clays or organic carbon content may simultaneously enhance the concentration of several metals to give the appearance of co-varying levels with constant relative proportions. The metals that are detected in all samples will be included in the factor analysis for all combined soil types. In the factor analysis, the correlation matrix should demonstrate high positive correlation coefficients between metals that contribute to a given factor. This indicates that among the samples in a given data set, groups of metals that comprise one factor tend to have concentrations in a fixed proportion, given the absence of any other factors affecting the same metals.

While a conventional geochemical analysis employs linear regression plots for metal pairs within background soils to predict the range of naturally occurring or expected concentrations, a further extension of this technique which uses even more of the available information is to define the background data sets by means of simultaneous multi-element correlation analysis. Multiple correlations may be characterized by factor analysis to provide a means to describe the overall pattern of covarying minerals in the background data sets, which may then be used to facilitate prediction of upper limits of background ranges for trace metals in site soils considering a sample's overall mineral pattern. Analogous to single element prediction methods, frequently the regression plots based on factor analysis of multiple metals can provide a more accurate estimate of the linearity of the correlation and a narrower prediction window from which to evaluate background soil concentrations. There are a number of ways to optimize factor analysis, and the method is very sensitive to variable outputs of derived factor patterns depending on the specific modeling options, so multiple trial simulations and comparison of overall prediction errors among simulations is needed. During exploratory analysis, the potential reliability of predictions from any candidate factor analysis model will be confirmed by a procedure which omits the influence of the metal in question when performing a set of analyte-of-interest predictions across the entire background data set, and then tabulating the overall residuals of observed versus factor analysis-derived analyte-of-interest concentrations across all samples in that data set. The relative percent prediction errors, expressed as observed

minus predicted analyte-of-interest concentration values divided by the observed values times 100, will be tabulated for all individual background samples. Also, the number of data points with relative errors above a certain absolute value will be noted for each background soil type as supporting data.

For a particular analyte of interest, the most appropriate factor analysis model will be selected, which consists of a factor pattern matrix (FPM), a factor pattern coefficient matrix (FPC), and mean-centered and variance normalizing constants for each metal in the background data set. In each background sample, a rearrangement of the factor model equations will be applied in order to generate an abscissa axis value that represents a back-predicted concentration for the analyte of interest, while the ordinate axis will represent the concentration value actually observed for that analyte. On this scatter plot, a prediction limit envelope will be calculated to encompass approximately 95 percent of the background data based on linear regression statistics. The method for determining the back-predicted analyte concentrations from factor analysis involves modifying the factor pattern coefficients (FPC) matrix by replacement with zeros across the row containing the metal of interest, which prevents the invalid use the metal of interest as an input to predict the concentration for the same metal of interest. However, so that the factor analysis formulas remain unbiased, each column of coefficients in the FPC matrix must be adjusted by multiplication by a scalar constant equal to the original sum of column coefficients divided by the sum of column coefficients minus the coefficient from the row containing the metal to be predicted. Comparison of back-predictions done with and without the influence of the metal in question will be employed to demonstrate the adequateness of predictive accuracy.

To document the uncertainty and prediction errors associated with factor analysis geochemical prediction envelopes, several measures will be taken. For an analyte of interest, the residual deviation of each background data point minus the regression value will be plotted on the ordinate axis against the factor analysis derived value as the abscissa value. The residual deviations, normalized to the weighted variance, should display a fairly consistent spread throughout the entire domain covered by the regression. Also, probability (Q-Q) plots of the residuals will be presented, which will verify the extent to which residuals are approximately normally distributed given any particular variance weighting employed.

As a further check to ensure lack of transformation bias, the practical coverage of the fitted prediction envelope for each background population will be tabulated in several ways. The number of background samples that fall outside the prediction envelope will be listed for each soil type and for all soil types considered together. Because approximately 100 or more data points will be employed in one regression, there will be sufficient data to assume that all of the background population is adequately represented and prediction envelope coverage of approximately 95 % of the background data can be verified directly on the scatter plot.

In addition, a tabulation of the relative width of the prediction interval, as expressed in untransformed analyte concentration units, will be listed for four separate regions of each regression: 25th percentile, median, 75th percentile, and 90th percentile. For example, at an analyte concentration equal to the 90th percentile of the background data set, the prediction interval envelope might be expressed as an upper and lower limit of plus or minus 42 percent from the regression line's predicted value. At the 25th percentile, the upper and lower limits might be somewhat different, for example, plus or minus 32 percent from the regression line's predicted value. Thus, the quality of fit for a geochemical prediction envelope can be compared by looking at the narrowness of the width of the prediction interval. For example, a 95 percent upper prediction limit equal to the predicted value plus 20 percent is a tighter fit than an upper prediction limit equal to the predicted value plus 30 percent.

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FIGURES

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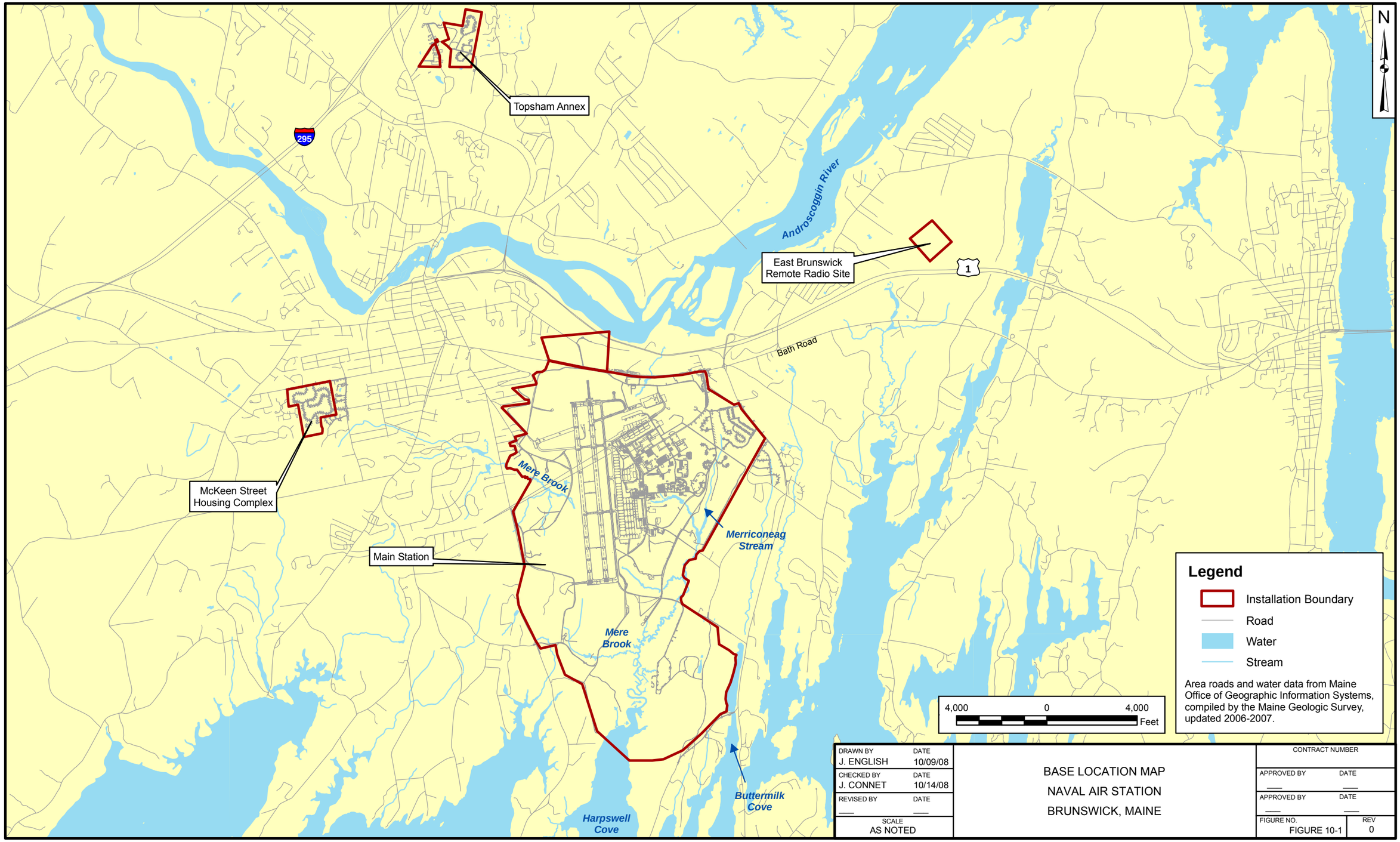
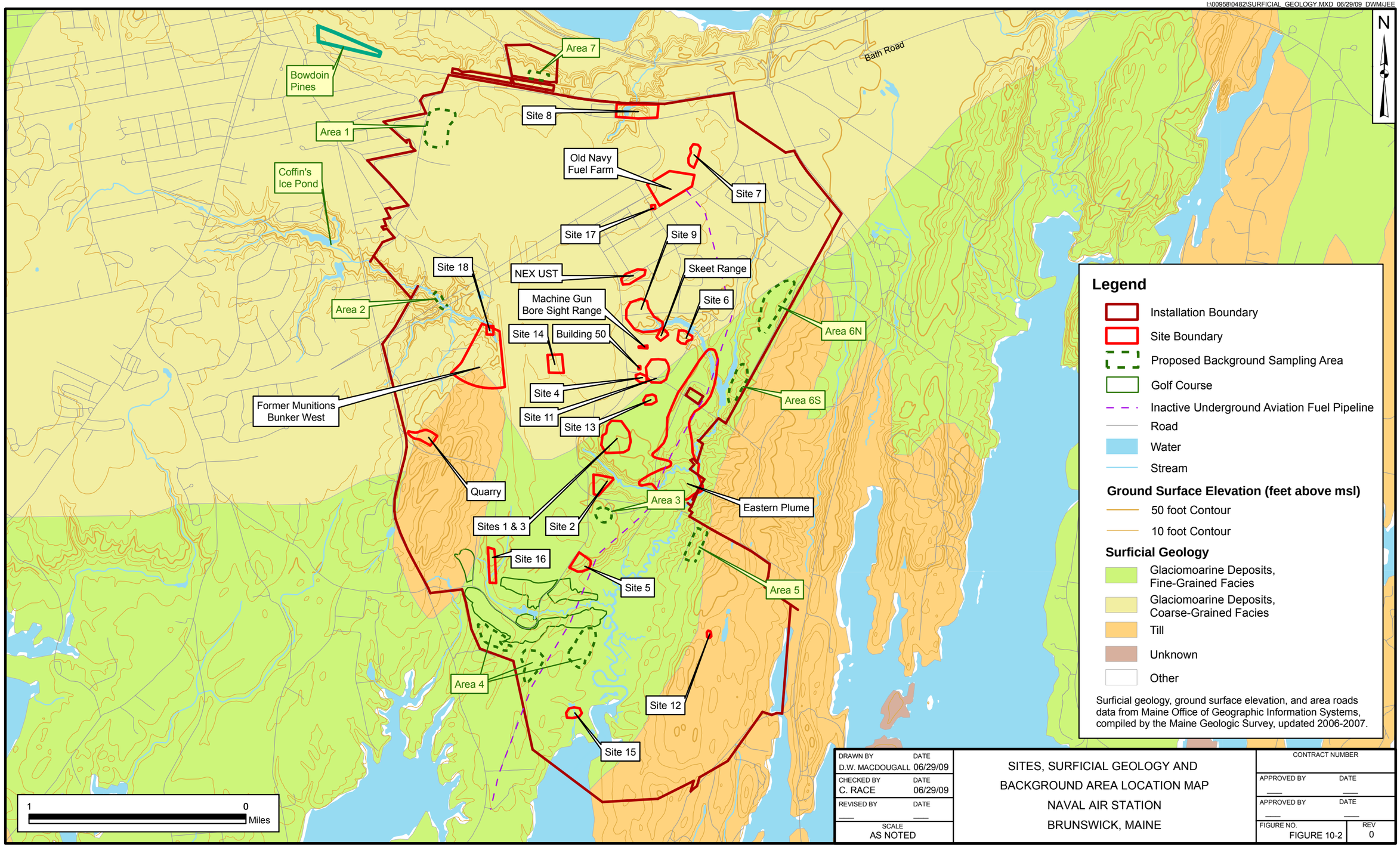


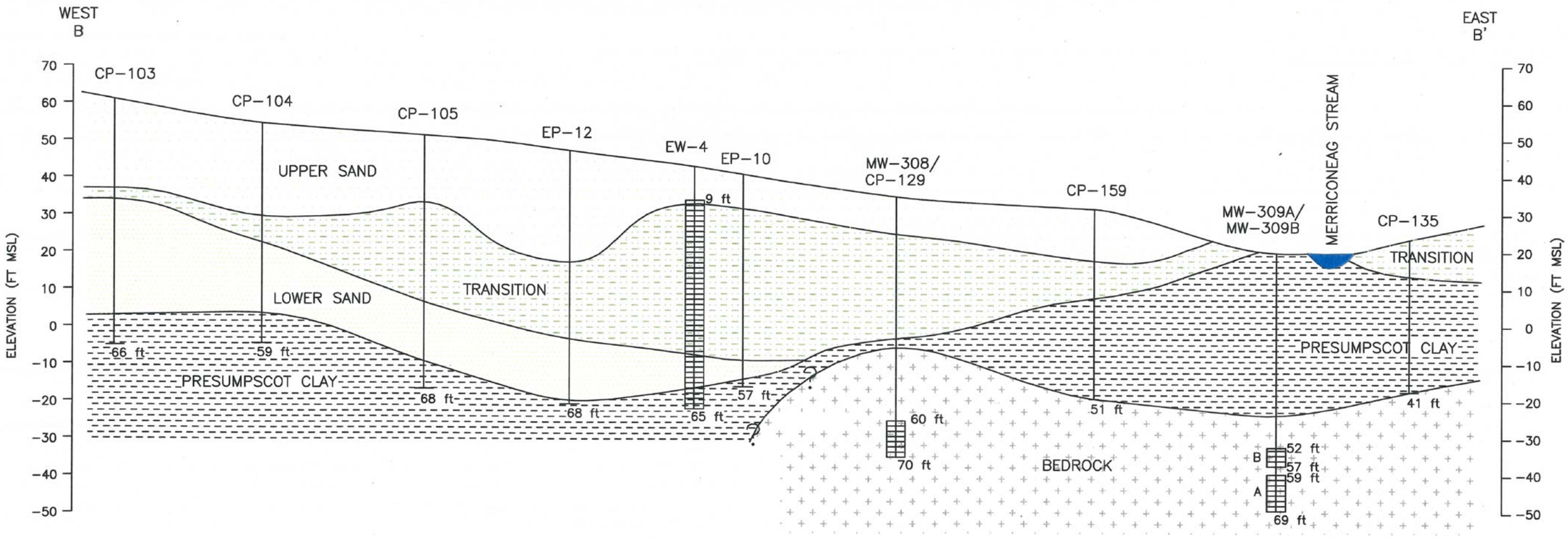
DRAWN BY J. ENGLISH	DATE 10/09/08	BASE LOCATION MAP NAVAL AIR STATION BRUNSWICK, MAINE	CONTRACT NUMBER
CHECKED BY J. CONNET	DATE 10/14/08		APPROVED BY ___ DATE ___
REVISED BY ___	DATE ___		APPROVED BY ___ DATE ___
SCALE AS NOTED			FIGURE NO. FIGURE 10-1 / REV 0

I:\00958\0482\SURFICIAL_GEOLOGY.MXD 06/29/09 DWM/JEE



DRAWN BY	DATE	SITES, SURFICIAL GEOLOGY AND BACKGROUND AREA LOCATION MAP NAVAL AIR STATION BRUNSWICK, MAINE	CONTRACT NUMBER	
D.W. MACDOUGALL	06/29/09			
CHECKED BY	DATE		APPROVED BY	DATE
C. RACE	06/29/09		___	___
REVISED BY	DATE		APPROVED BY	DATE
___	___		___	___
SCALE			FIGURE NO.	REV
AS NOTED			FIGURE 10-2	0

ACAD: \00645\1002\X-SECTION.DWG 10/10/08 DWM



LEGEND

MW-308 — Location ID (Monitoring wells or borings in bold were not used in cross-section from 1990/1990 RI)

Ground surface

60 ft — Depth to top of screen well interval

70 ft — Total Depth (ft below ground surface)

SOIL DESCRIPTION

UPPER SAND: Tan to light brown fine sand, trace to little medium sand and silt, poorly graded, loose to moderately dense, slightly stratified with depth

TRANSITION: Brown to gray fine sand and silty fine sand, trace clay, stratified with silt and clay lenses, poorly graded, slightly plastic and soft

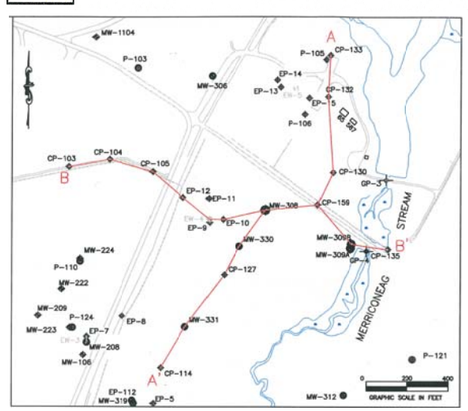
LOWER SAND: Gray to orange brown fine or very fine sand (likely to be sandy interval within transition unit)

PRESUMPSCOT CLAY: Gray silty clay to clayey silt (rock flour), medium to high plasticity, very soft

BEDROCK: Weathered micaceous schist

NOTE: FIGURE BASED ON EA FIGURE: WEST-EAST CROSS-SECTION OF THE EASTERN PLUM NEAR MW-308, 10 NOV 2000, W-E X-SECTION.DWG

NOTES:
1. SOIL AND BEDROCK CONTACTS ARE INFERRED BELOW AND BETWEEN LOCATIONS
2. CONE PENETOMETER LOCATIONS SHOWN ON INDEX MAP ARE APPROXIMATE



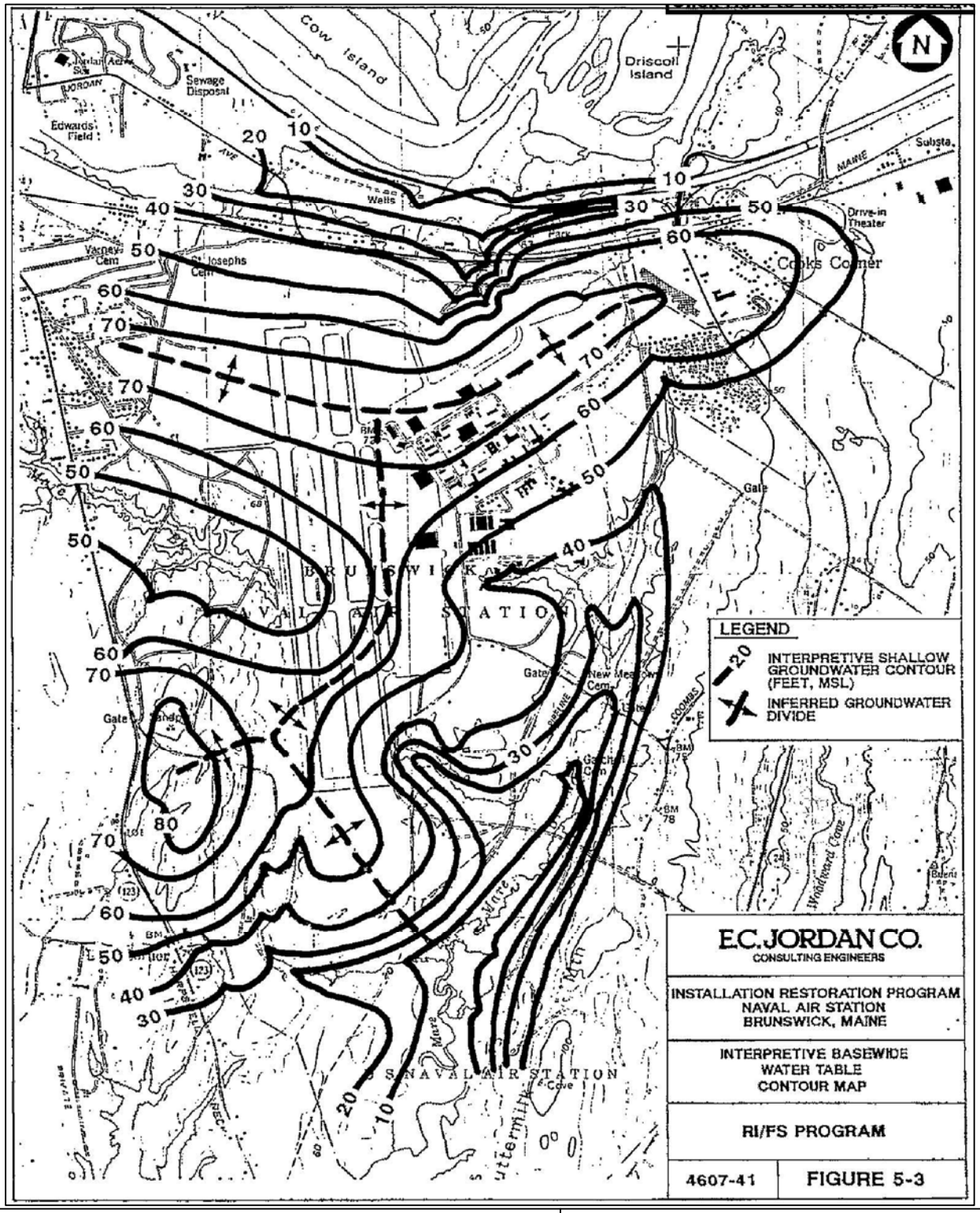
0 100 200 400

GRAPHIC SCALE IN FEET
VERTICAL EXAGGERATION = 5X

TETRA TECH NUS, INC.

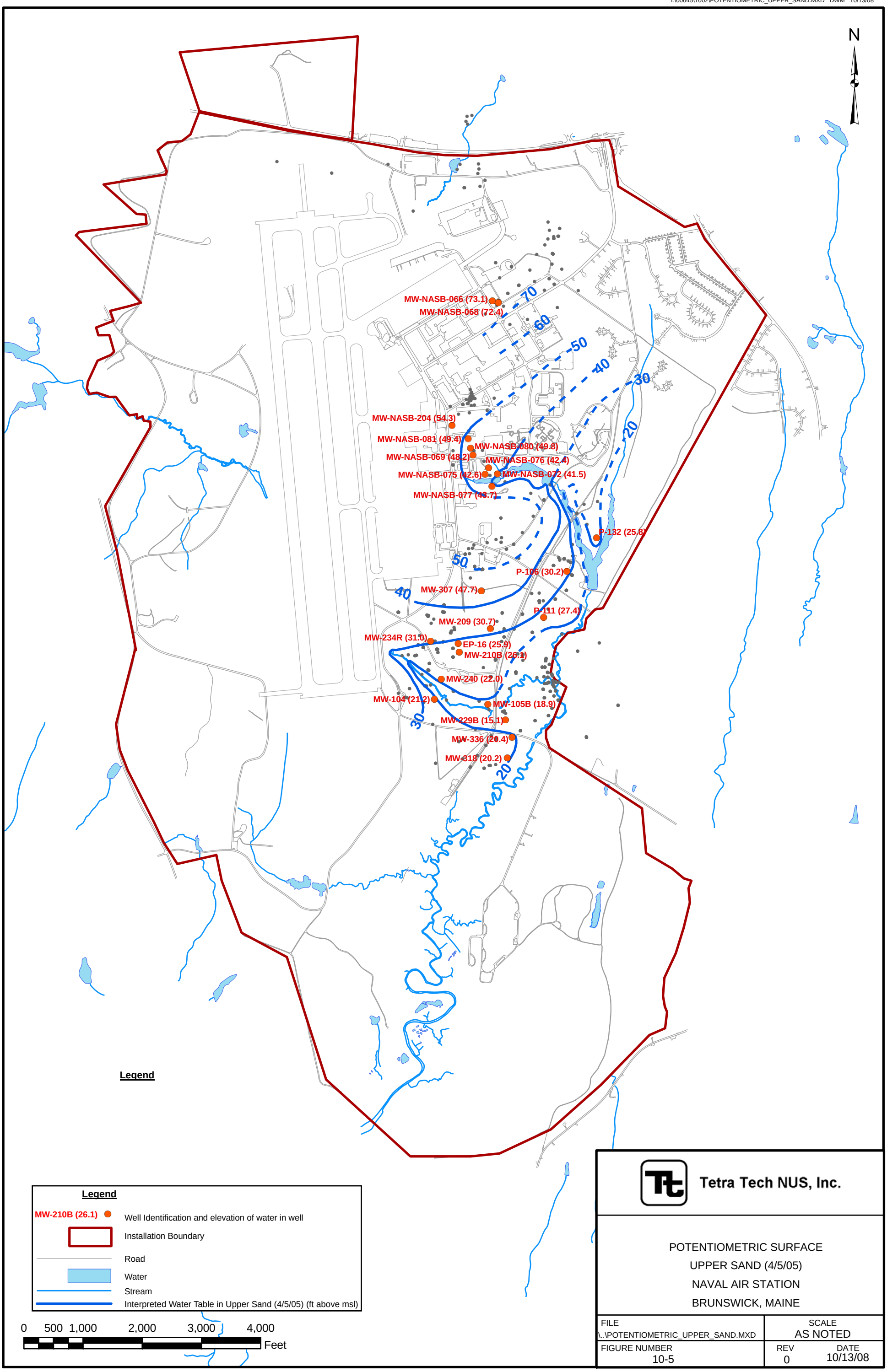
WEST-EAST CROSS-SECTION OF THE EASTERN PLUME NEAR ME-308
NAVAL AIR STATION
BRUSNWICK, MAINE

FILE	SCALE
\..\X-SECTION.DWG	AS NOTED
FIGURE NUMBER 10-3	REV 0 / DATE 10/10/08



INTERPRETIVE BASEWIDE WATER TABLE CONTOUR MAP (1990)	FIGURE 10–4
NAVAL AIR STATION	TETRA TECH NUS, INC.
BRUNSWICK, MAINE	
DRAWN BY: D.W. MACDOUGALL / REV.: 0	
CHECKED BY: J. CONNETT / DATE: OCTOBER 10, 2008	55 Jonspin Road Wilmington, MA 01887 (978)658–7899
SCALE: NOT GIVEN / ACAD NAME: \00645\1002\GW_CONTOUR.DWG	

I:\00645\1002\POTENTIOMETRIC_UPPER_SAND.MXD DWM 10/13/08

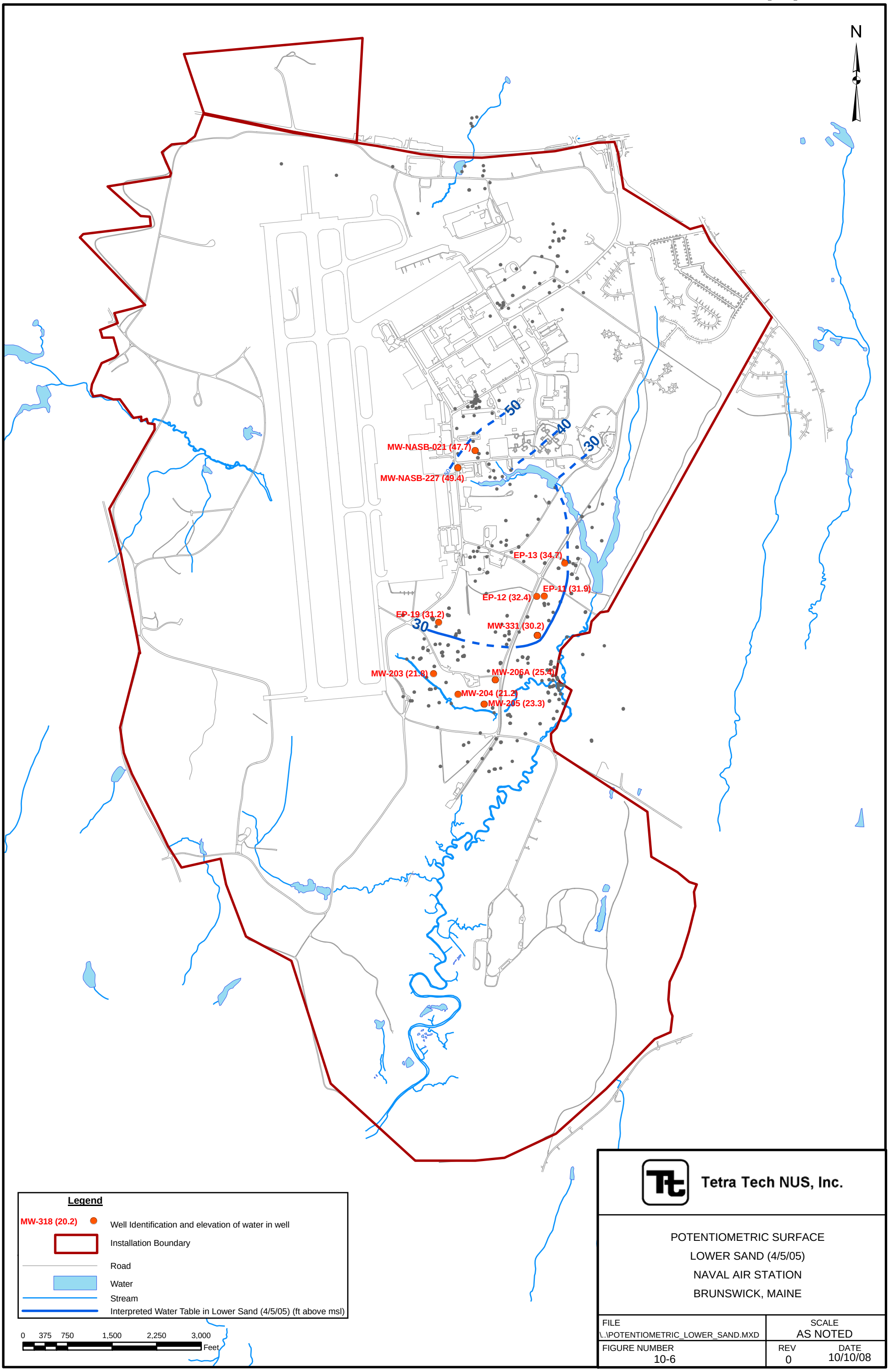


Tetra Tech NUS, Inc.

POTENTIOMETRIC SURFACE
UPPER SAND (4/5/05)
NAVAL AIR STATION
BRUNSWICK, MAINE

FILE	SCALE
\..\POTENTIOMETRIC_UPPER_SAND.MXD	AS NOTED
FIGURE NUMBER 10-5	REV 0 / DATE 10/13/08

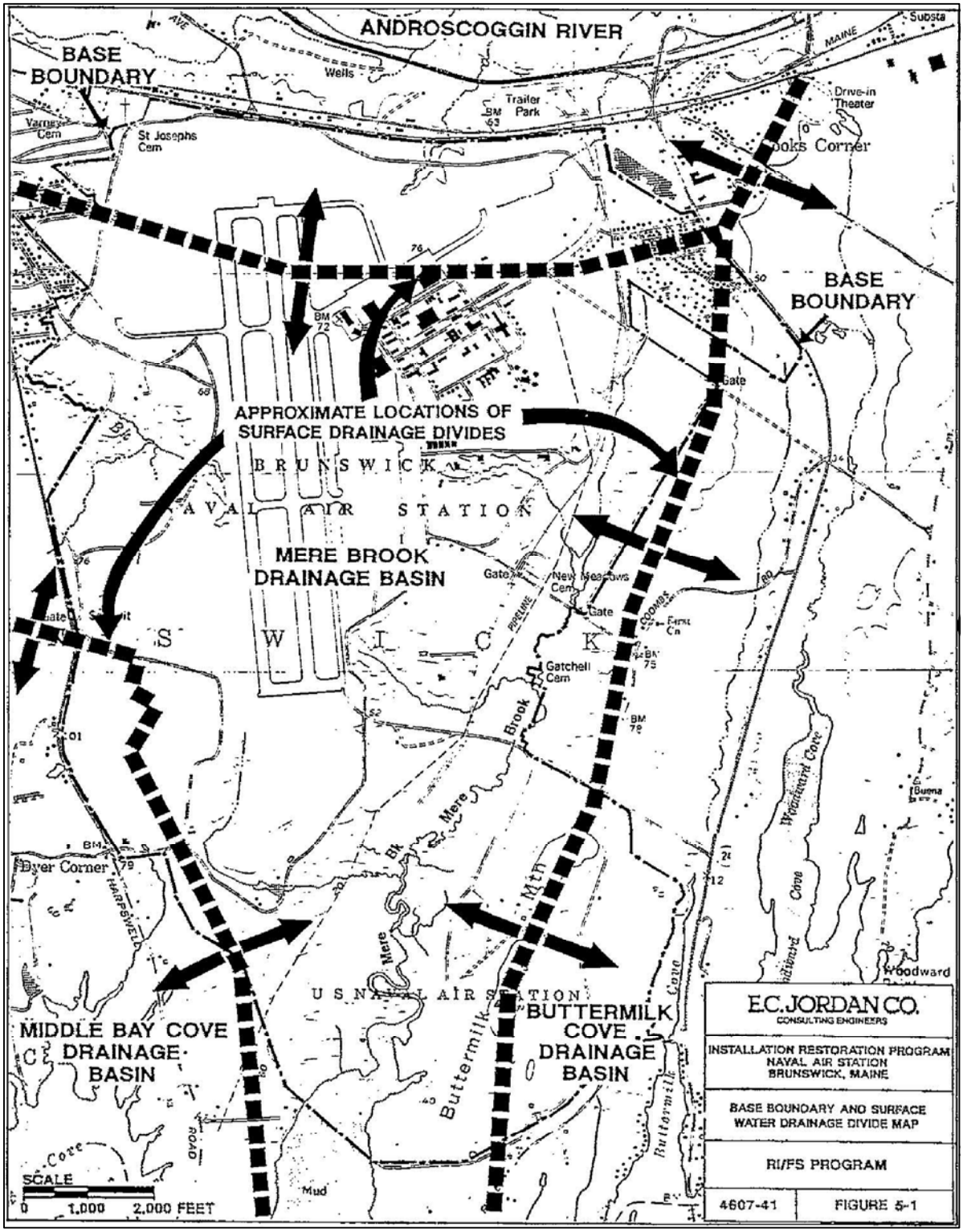
I:\00645\1002\POTENTIOMETRIC_LOWER_SAND.MXD DWM 10/10/08



Tetra Tech NUS, Inc.

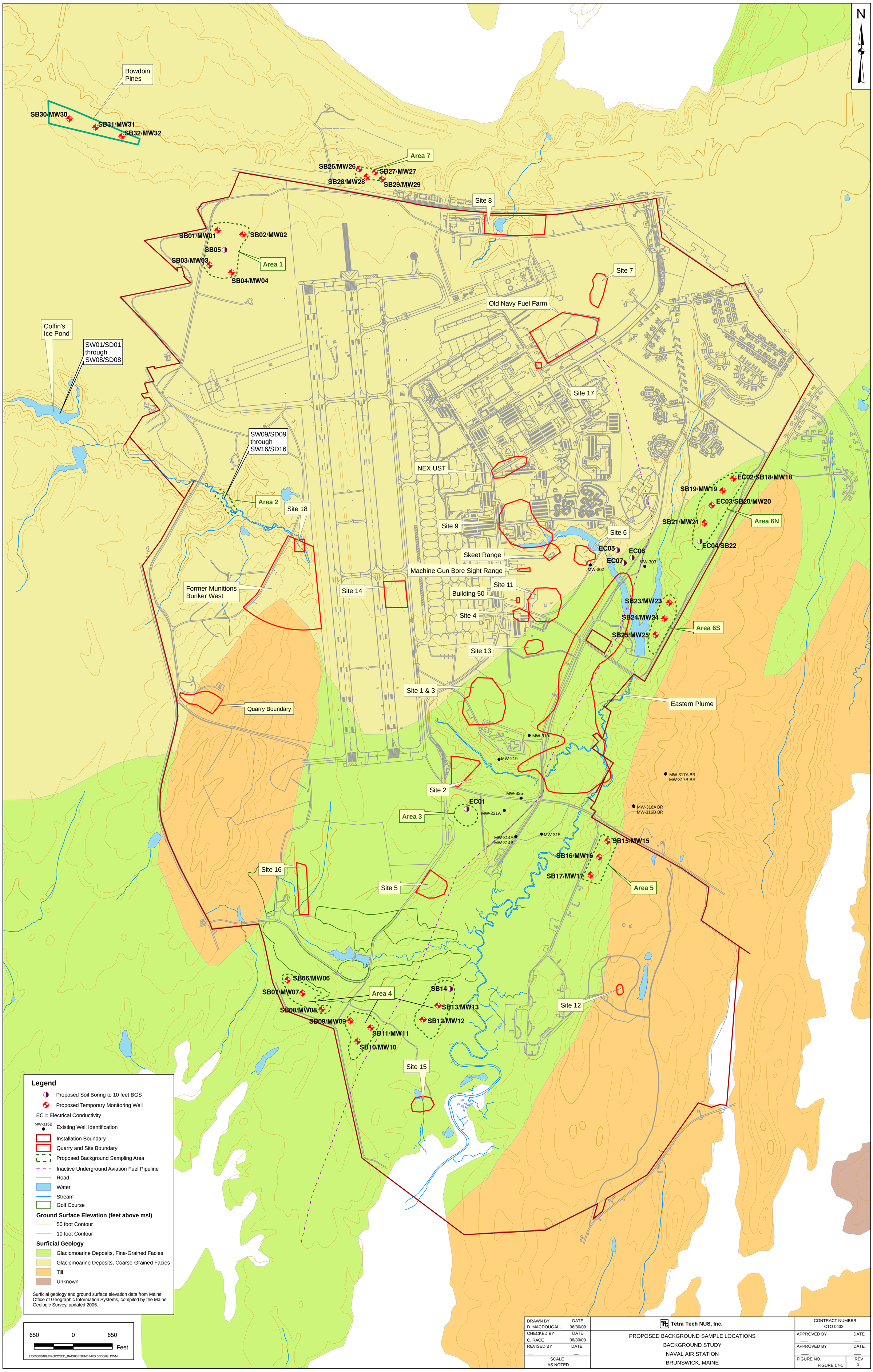
POTENTIOMETRIC SURFACE
LOWER SAND (4/5/05)
NAVAL AIR STATION
BRUNSWICK, MAINE

FILE	SCALE
\..\POTENTIOMETRIC_LOWER_SAND.MXD	AS NOTED
FIGURE NUMBER 10-6	REV 0 / DATE 10/10/08



SURFACE WATER DRAINAGE MAP	FIGURE 10–7
NAVAL AIR STATION	TETRA TECH NUS, INC.
BRUNSWICK, MAINE	55 Jonspin Road Wilmington, MA 01887 (978)658–7899

DRAWN BY:	D.W. MACDOUGALL	REV.:	0
CHECKED BY:	J. CONNETT	DATE:	OCTOBER 10, 2008
SCALE:	AS NOTED	ACAD NAME:	\00645\1002\SURF_WATER_DRAIN.DWG


N
Bowdoin Pines
SB30/MW30
SB31/MW31
SB32/MW32
Area 7
SB26/MW26
SB27/MW27
SB28/MW28
SB29/MW29
Site 8
SB01/MW01
SB02/MW02
SB05
SB03/MW03
Area 1
SB04/MW04
Site 7
Old Navy Fuel Farm
Coffin's Ice Pond
SW01/SD01 through SW08/SD08
Site 17
SW09/SD09 through SW16/SD16
NEX UST
EC02/SB18/MW18
SB19/MW19
EC03/SB20/MW20
Area 6N
Area 2
Site 18
SB21/MW21
Site 9
Site 6
EC04/SB22
EC05
EC06
EC07
Skeet Range
MW-303
MW-302
Machine Gun Bore Sight Range
Former Munitions Bunker West
Site 14
Site 11
Building 50
SB23/MW23
SB24/MW24
Site 4
Area 6S
SB25/MW25
Site 13
Site 1 & 3
Eastern Plume
Quarry Boundary
MW-310
MW-219
MW-317A BR
MW-317B BR
Site 2
MW-335
EC01
MW-231A
MW-316A BR
MW-316B BR
Area 3
MW-314A
MW-314B
MW-315
SB15/MW15
SB16/MW16
Site 16
SB17/MW17
Site 5
Area 5
SB06/MW06
SB07/MW07
Area 4
SB14
SB13/MW13
Site 12
SB08/MW08
SB09/MW09
SB12/MW12
SB11/MW11
SB10/MW10
Site 15
Legend
Proposed Soil Boring to 10 feet BGS
Proposed Temporary Monitoring Well
EC = Electrical Conductivity
MW-316B
Existing Well Identification
Installation Boundary
Quarry and Site Boundary
Proposed Background Sampling Area
Inactive Underground Aviation Fuel Pipeline
Road
Water
Stream
Golf Course
Ground Surface Elevation (feet above msl)
50 foot Contour
10 foot Contour
Surficial Geology
Glaciomoarine Deposits, Fine-Grained Facies
Glaciomoarine Deposits, Coarse-Grained Facies
Till
Unknown
Surficial geology and ground surface elevation data from Maine Office of Geographic Information Systems, compiled by the Maine Geologic Survey, updated 2006.
650 0 650
Feet
I:/00958/0482/PROPOSED_BACKGROUND.MXD 06/30/09 DWM
DRAWN BY D. MACDOUGALL DATE 06/30/09
CHECKED BY C. RACE DATE 06/30/09
REVISED BY — DATE —
SCALE AS NOTED
Tetra Tech NUS, Inc.
PROPOSED BACKGROUND SAMPLE LOCATIONS
BACKGROUND STUDY
NAVAL AIR STATION
BRUNSWICK, MAINE
CONTRACT NUMBER CTO 0432
APPROVED BY — DATE —
APPROVED BY — DATE —
FIGURE NO. FIGURE 17-1
REV 1

APPENDIX A

SAMPLING AND RELATED SOPs

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5.3 EXAMPLE SAMPLE FIELD DESIGNATIONS 4
5.4 EXAMPLES OF SAMPLE NOMENCLATURE 5
5.5 FIELD QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) SAMPLE NOMENCLATURE) 6
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6.0 DEVIATIONS **6**

1.0 PURPOSE

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

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

2.0 SCOPE

The methods described in this procedure shall be used consistently for all projects requiring electronic data.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

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

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

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

5.0 PROCEDURES

5.1 Introduction

The sample identification (ID) system can consist of as few as 8 but not more than 20 distinct alpha-numeric characters. The sample ID will be provided to the laboratory on the sample labels and chain-of-custody forms. The basic sample ID provided to the lab has three segments and shall be as follows where "A" indicates "alpha," and "N" indicates "numeric":

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

Additional segments may be added as needed. For example:

(1) Soil and Sediment Sample ID

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

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

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	NN 2-Characters	-A
Site Identifier	Sample type	Sample Location	Round Number	Filtered Sample only

(3) Biota Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	AA 2-Characters	NNN 3-Characters
Site Identifier	Sample Type	Sample Location	Species Identifier	Sample Group Number

5.2 Sample Identification Field Requirements

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

- Site Identifier
- Sample Type
- Sample Location
- Sample Depth
- Sampling Round Number
- Filtered
- Species Identifier
- Sample Group Number

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

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

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

The sample depth field is used to note the depth below ground surface (bgs) at which a soil or sediment 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. If the sample depth is equal to or greater than 100, then only the top interval would be represented and the sampling depth would be truncated to

three-characters. 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.

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

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

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

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

5.3 Example Sample Field Designations

Examples of each of the fields are as follows:

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

A01 - Area of Concern Number 1
125 - Solid Waste Management Unit Number 125
000 - Base or Facility Wide Sample (e.g., upgradient well)
BBG - Base Background

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

Sample Type - Examples of sample types are as follows:

AH - Ash Sample
AS - Air Sample
BM - Building Material Sample
BSB - Biota Sample Full Body
BSF - Biota Sample Fillet
CP - Composite Sample
CS - Chip Sample
DS - Drum Sample
DU - Dust Sample
FP - Free Product
IDW - Investigation Derived Waste Sample
LT - Leachate Sample
MW - Monitoring Well Groundwater Sample
OF - Outfall Sample
RW - Residential Well Sample
SB - Soil Boring Sample
SD - Sediment Sample
SC - Scrape Sample

SG - Soil Gas Sample
SL - Sludge Sample
SP - Seep Sample
SS - Surface Soil Sample
ST - Storm Sewer Water Sample
SW - Surface Water Sample
TP - Test Pit Sample
TW - Temporary Well Sample
WC - Well Construction Material Sample
WP - Wipe Sample
WS - Waste/Solid Sample
WW - Wastewater Sample

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

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

Species Identifier - Examples of species identifier are as follows:

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

5.4 Examples of Sample Nomenclature

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

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

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

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

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

A sediment sample collected at SWMU 19 from 0 to 6 inches at location 14 would be designated as 019SD0140001. The sample data sheet would reflect the precise depth at which this sample was collected.

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

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

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

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

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

The QC types are identified as:

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

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

5.6 Examples of Field QA/QC Sample Nomenclature

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

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

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

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

6.0 DEVIATIONS

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

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5.2 FILE ESTABLISHMENT 4
5.3 ELECTRONIC DELIVERABLES 4
5.4 SAMPLE TRACKING FORMS 5
5.5 CHAIN-OF-CUSTODY FORMS 5
5.6 DATA VALIDATION LETTERS 5
5.7 HISTORICAL DATA 5

6.0 RECORDS **6**

ATTACHMENTS

A MIS REQUEST FORM **7**

1.0 PURPOSE

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

2.0 SCOPE

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

3.0 GLOSSARY

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

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

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

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

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

4.0 RESPONSIBILITIES

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

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

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

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

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

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

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

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

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

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

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

5.0 PROCEDURES

5.1 Introduction

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

5.2 File Establishment

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

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

5.3 Electronic Deliverables

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

5.4 Sample Tracking Forms

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

5.5 Chain-of-Custody Forms

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

5.6 Data Validation Letters

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

5.7 Historical Data

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

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

6.0 RECORDS

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

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

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

ATTACHMENT A

MIS REQUEST FORM

Tetra Tech NUS, Inc.

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

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

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

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

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

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

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

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A SITE RECONNAISSANCE CHECKLIST 6

1.0 PURPOSE

The purpose of a site reconnaissance is to collect both general and technical information which will support the scoping, scheduling, implementing project activities, and writing reports for an environmental investigation. This procedure is not intended as a guide for Phase I investigations or for Environmental Baseline Survey activities.

2.0 SCOPE

This procedure is applicable to the performance of a site reconnaissance for initial site characterization. The steps necessary to develop and carry out a site reconnaissance are presented here. These steps include a list of equipment and items which may be needed, areas of special interest during field observations, and methods by which the field observation team can ensure that necessary and appropriate observations have been made.

3.0 GLOSSARY

Site reconnaissance. An onsite inspection program used to identify site-specific conditions that control scheduling, manpower, and affect costs. A site reconnaissance usually consists of visual observations and, often, the use of field monitoring instruments to identify potential health and safety threats and potential sampling locations for site evaluation during subsequent field investigations.

4.0 RESPONSIBILITIES

Field Operations Leader (FOL) is responsible for ensuring that the survey is carried out in sufficient detail. To accomplish this, the FOL must assign the proper personnel and equipment to characterize the site adequately, in accordance with the requirements defined in this procedure and best engineering practices. Other disciplines which may be applicable include (but are not limited to): Geology/Hydrogeology; Health and Safety; Ecological Specialists; and/or Engineering. In addition, the FOL is responsible for supervising equipment preparation, including necessary calibrations, and supervising field data collection and documentation in accordance with the methods described in all referenced standard operation procedures.

Project Manager is responsible for the following:

- Supervising the retrieval and examination of available, applicable information regarding the site.
- Obtaining appropriate program approvals and ensuring the preparation of a site Health and Safety plan for the site reconnaissance.
- Coordinating the field activities with the client and regulatory agencies, as applicable.

Field Personnel are primarily responsible for observing and documenting, either through written documentation or photographic evidence, the site reconnaissance. Field personnel will take direction from the FOL.

5.0 PROCEDURES

5.1 Equipment Items/Needed

Below is a list of items that may be useful when conducting a site reconnaissance. All, or a portion of these items may be required, depending upon the objective of the site reconnaissance.

- Health and Safety equipment and information as required by the Site Safety Officer.
- Maps (U.S.G.S. quadrangle, geologic maps, street and highway maps, and client facility maps).
- Geologic tools (compass, tape measure, hand level, camera, etc.).
- Physical monitoring equipment, if applicable (PID, Immunoassay Test Kits, etc.)
- Regional publications (U.S.G.S reports, water well surveys, U.S.D.A. soil conservation surveys, etc.).
- Site-specific publications by previous investigators (EPA aerial photographic analyses, remedial investigation reports, data on waste disposal practices, boring logs, etc.).
- Marking items (ink markers, surveyor's flagging, spray paint, pin flags, wooden stakes).
- Field notebooks.
- Local telephone book with yellow pages (for obtaining utilities, site trailer, living accommodations, etc.).

Sufficient time will be required in order to obtain some of the aforementioned material. In general, most publications can be obtained in time to be used in the site reconnaissance if ordered approximately 2 weeks before the actual site visit takes place.

5.2 Observations

A site reconnaissance usually requires one to two days, however, additional time may be needed depending upon the objective, site size, etc. The following observations, when applicable, should be documented either on a site map, field notebook, or photographed.

- General Site Access. It should be noted whether site roads provide access to all proposed work locations, or if it will be necessary to prepare access roads with either a backhoe, dozer, chain saws, etc., in order to get drill rigs, excavators, or other work vehicles to specific locations. If temporary driveways must be constructed from existing public roads, regulatory permits may be required. Military facilities may have specific security requirements which require detailed clearance procedures.
- Location of the Command Post or Site Trailer and Sanitary Facilities. The ideal location for the site trailer and sanitary facilities is a level area, within an uncontaminated zone, and centralized in order to provide easy access to work areas on the site. However, certain utility companies may require that the site trailer be placed within a specified radius (usually 100 feet), of the nearest utility pole. Contact the necessary utility companies and inquire about the requirements regarding service before conducting the site reconnaissance. Information that may be required by the utility companies is: type of electric service needed (inquire with trailer vendor for this information); and utility pole number of interest (pole numbers are usually stamped on a brass plate on the pole).

- Potable Water Sources. Local fire departments may allow access to fire hydrants. Private water delivery companies may also be available in the area.

- Sources of Possible Contamination. Drums, tanks, sludge areas, areas of stressed vegetation, fill areas, and leachate seeps may indicate where sources of contamination exist. Filler pipes protruding from the ground surface may indicate the presence of underground storage tanks. Areas where the original ground surface has been reworked may be contaminated fill areas that have since been buried and covered with natural material. Previous environmental investigations may also identify source areas.

- Location of Decon Areas and Storage/Disposal Areas for Equipment and Wastes Generated by Field Activities.

- Locations of Surface Water Bodies. The locations of surface water bodies, both man-made and natural, and their relation to topographic highs may give an indication of the groundwater flow direction in the area (groundwater flow typically follows topography with the topographic highs serving as groundwater recharge areas, and the surface waters at topographic lows serve as groundwater discharge areas). Visible signs of contamination, the existence of aquatic life, flow rates, and approximate levels should also be observed and noted. Check if the surface water bodies could potentially be impacted by field activities. If so, appropriate sedimentation and erosion controls will be required.

- Existing Wells. Existing monitoring wells, or domestic wells within the site and off site, should be noted on a map, and access checked to see if the wells can be used for data collection.

- Outcrops. Outcrops can be useful in providing hydrogeologic data (lithologic description, strike and dip information, fracture and joint system analysis, identification of moist zones, etc.) Outcrops may occur naturally or be a part of a man-made feature such as a road-cut.

- Lineaments. A lineament is a straight lengthy feature on the earth's surface which is expressed topographically as a line of depression. Stream beds, vegetation patterns or soil characteristics may be aligned or controlled by this feature. Lineaments are due in some cases to the presence of intense jointing or faults beneath the ground surface. Groundwater in the bedrock may follow lineaments. Lineaments should be noted on site maps and described in the field notebooks.

- Bench or Property Markers. Benchmarks or property markers should be marked with paint or surveyor's flagging if encountered during a site reconnaissance. Surveyors may need to use these markers as a reference point when surveying. Benchmarks are typically a brass plate secured in concrete in the ground with numbering on the top. Property markers can range from a stake driven into the ground to a rock protruding from the ground surface. Facility contacts may also be aware of local benchmarks used during the course of other environmental or public work projects.

- Metal Cultural Effects. Overhead power lines, railroad tracks, junk automobiles, fences, etc. will greatly affect certain geophysical surveys. These features should be noted while conducting a site reconnaissance.

6.0 RECORDS

The data collected during a site reconnaissance may have to be compiled into a trip report when returning from the field. This trip report can then be distributed to the project team. A site reconnaissance checklist is located in Attachment A which can be copied and used while conducting the site reconnaissance.

ATTACHMENT A

SITE RECONNAISSANCE CHECKLIST

SITE SKETCH

Include the following as appropriate:

- Site Name
- Site location
- Site Boundaries
- Entrance locations
- Access Roads and Security Requirements
- Disposal locations
- Storage areas
- Office areas
- Well locations
- Treatment facility locations
- Surface drainage, outcrops, general topography descriptions
- Cultural interferences

CHEMICAL STORAGE FACILITIES DESCRIPTION

- Storage tanks - numbers, volumes, condition, contents, etc.
- Drums - number, conditions, labeling, etc.
- Lagoons and surface pits - number, size, use of liner, contents, etc.

TREATMENT SYSTEMS

Note the presence of any treatment systems. These can be difficult to evaluate visually. One should appraise general appearance, maintenance and visual integrity; ask operators for any monitoring records; note presence of odors; and visually characterize any effluents or residues. Describe type of wastes and volumes treated.

- Incinerators
- Flocculation/filtration
- Chemical/physical treatment
- Biological treatment
- Volume reduction
- Waste recycling
- Compositing
- Other

ATTACHMENT A
SITE RECONNAISSANCE CHECKLIST
PAGE TWO

DISPOSAL FACILITIES

Note the presence and use of any of the following operations. Include a description of the size, use of liners, soil type, and the presence of leachate. Provide a description of management practices. Interview site workers if possible. Describe waste types.

- Landfills
- Land forms
- Open dump
- Surface impoundment
- Underground injection
- Incineration

Also, records for disposal of concentrated/containerized waste should be reviewed.

HAZARDOUS SUBSTANCE CHARACTERISTICS

Ask facility contacts for manifests, inventories, or monitoring reports. Note markings on containers.

- Chemical identities
- Quantities
- Hazard characteristics (toxic, explosive, flammable, etc.)
- Container markings
- Monitoring data, other analytical data
- Physical state (liquid, solid, gas, sludge)

CHEMICAL PROCESS INFORMATION

- Manufacturing processes and chemicals
- Off-specification or by-product disposal processes
- Housekeeping practices
- Locations of Plant Operations

ATTACHMENT A
SITE RECONNAISSANCE CHECKLIST
PAGE THREE

HYDROGEOLOGIC ASSESSMENT

Look for situations that promote hazardous substance migration, i.e., porous soils, fractured bedrock formations, shallow water table and karst features.

- Soil type
- Surface water features
- Surface drainage pattern
- Outcrop studies
- Water wells (use, water depth, and construction details)
- Erosion potential
- Flooding potential
- Climatology

IDENTIFICATION OF SENSITIVE RECEPTORS

- Number and locations of private homes
- Public buildings including tenant usage
- Areas of dead or dying vegetation or animals
- Presence of sensitive ecosystems (wetlands, tidal marshes, etc.)
- Other public use areas (roads, parks, etc.)
- Natural areas

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B GROUNDWATER LEVEL MEASUREMENT SHEET 9

1.0 PURPOSE

The purpose of this procedure is to provide reference information regarding the proper methods for evaluating the physical condition and project utility of existing monitoring wells and determining water levels.

2.0 SCOPE

The procedures described herein are applicable to all existing monitoring wells and, for the most part, are independent of construction materials and methods.

3.0 GLOSSARY

Hydraulic Head - The height to which water will rise in a well.

Water Table - A surface in an unconfined aquifer where groundwater pressure is equal to atmospheric pressure (i.e., the pressure head is zero).

4.0 RESPONSIBILITIES

Site Geologist/Hydrogeologist - Has overall responsibility for the evaluation of existing wells, obtaining water level measurements and developing groundwater contour maps. The site geologist/hydrogeologist (in concurrence with the Project Manager) shall specify the reference point from which water levels are measured (usually a specific point on the upper edge of the inner well casing), the number and location of data points which shall be used for constructing a contour map, and how many complete sets of water levels are required to adequately define groundwater flow directions (e.g., if there are seasonal variations).

Field Personnel - Must have a basic familiarity with the equipment and procedures involved in obtaining water levels and must be aware of any project-specific requirements or objectives.

5.0 PROCEDURES

Accurate, valid and useful groundwater monitoring requires that four important conditions be met:

- Proper characterization of site hydrogeology.
- Proper design of the groundwater monitoring program, including adequate numbers of wells installed at appropriate locations and depths.
- Satisfactory methods of groundwater sampling and analysis to meet the project data quality objectives (DQOs).
- The assurance that specific monitoring well samples are representative of water quality conditions in the monitored interval.

To insure that these conditions are met, adequate descriptions of subsurface geology, well construction methods and well testing results must be available. The following steps will help to insure that the required data are available to permit an evaluation of the utility of existing monitoring wells for collecting additional samples.

5.1 Preliminary Evaluation

A necessary first step in evaluating existing monitoring well data is the study and review of the original work plan for monitoring well installation (if available). This helps to familiarize the site geologist/hydrogeologist with site-specific condition, and will promote an understanding of the original purpose of the monitoring wells.

The next step of the evaluation should involve a review of all available information concerning borehole drilling and well construction. This will allow interpretation of groundwater flow conditions and area geology, and will help to establish consistency between hydraulic properties of the well and physical features of the well or formation. The physical features which should be identified and detailed, if available, include:

- The well identification number, permit number and location by referenced coordinates, the distance from prominent site features, or the location of the well on a map.
- The installation dates, drilling methods, well development methods, past sampling dates, and drilling contractors.
- The depth to bedrock -- where rock cores were not taken, auger refusal, drive casing refusal or penetration test results (blow counts for split-barrel sampling) may be used to estimate bedrock interface.
- The soil profile and stratigraphy.
- The borehole depth and diameter.
- The elevation of the top of the protective casing, the top of the well riser, and the ground surface.
- The total depth of the well.
- The type of well materials, screen type, slot size, and length, and the elevation/depths of the screen, interval, and/or monitored interval.
- The elevation/depths of the tops and bottom of the filter pack and well seals and the type and size.

5.2 Field Inspection

During the onsite inspection of existing monitoring wells, features to be noted include:

- The condition of the protective casing, cap and lock.
- The condition of the cement seal surrounding the protective casing.
- The presence of depressions or standing water around the casing.
- The presence of and condition of dedicated sampling equipment.
- The presence of a survey mark on the inner well casing.

If the protective casing, cap and lock have been damaged or the cement collar appears deteriorated, or if there are any depressions around the well casing capable of holding water, surface water may have infiltrated into the well. This may invalidate previous sampling results unless the time when leakage started can be precisely determined.

The routine physical inspection must be followed by a more detailed investigation to identify other potential routes of contamination or sampling equipment malfunction. Any of these occurrences may invalidate

previously-collected water quality data. If the monitoring well is to be used in the future, considerations shown in the steps described above should be rectified to rehabilitate the well.

After disconnecting any wires, cables or electrical sources, remove the lock and open the cap. Check for the presence of organic vapors with a photoionization detector (PID) or flame-ionization detector (FID) to determine the appropriate worker safety level. The following information should be noted:

- Cap function.
- Physical characteristics and composition of the inner casing or riser, including inner diameter and annular space.
- Presence of grout between the riser and outer protective casing and the existence of drain holes in the protective casing.
- Presence of a riser cap, method of attachment to casing, and venting of the riser.
- Presence of dedicated sampling equipment; if possible, remove such equipment and inspect size, materials of construction and condition.

The final step of the field inspection is to confirm previous hydraulic or physical property data and to obtain data not previously available. This includes the determination of static water levels, total well depth and well obstruction. This may be accomplished using a weighted tape measure which can also be used to check for sediment (the weight will advance slowly if sediment is present, and the presence of sediment on the weight upon removal should be noted). If sediment is present and/or the well has not been sampled in 12 or more months, it should be redeveloped before sampling.

Lastly, as a final step, the location, condition and expected water quality of the wells should be reviewed in light of their usefulness for the intended purpose of the investigation.

See Attachment A, Monitoring Well Inspection Sheet.

5.3 Water Level (Hydraulic Head) Measurements

5.3.1 General

Groundwater level measurements can be made in monitoring wells, private or public water wells, piezometers, open boreholes, or test pits (after stabilization). Groundwater measurements should generally not be made in boreholes with drilling rods or auger flights present. If groundwater sampling activities are to occur, groundwater level measurements shall take place prior to well purging or sampling.

All groundwater level measurements shall be made to the nearest 0.01 foot, and recorded in the site geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet (Attachment B), along with the date and time of the reading. The total depth of the well shall be measured and recorded, if not already known. Weather changes that occur over the period of time during which water levels are being taken, such as precipitation and barometric pressure changes, should be noted.

In measuring groundwater levels, there shall be a clearly-established reference point of known elevation, which is normally identified by a mark on the upper edge of the inner well casing. To be useful, the reference point should be tied in with an established USGS benchmark or other properly surveyed elevation datum. An arbitrary datum could be used for an isolated group of wells, if necessary.

Cascading water within a borehole or steel well casings can cause false readings with some types of sounding devices (chalked line, electrical). Oil layers may also cause problems in determining the true water level in a well. Special devices (interface probes) are available for measuring the thickness of oil layers and true depth to groundwater, if required.

Water level readings shall be taken regularly, as required by the site geologist/hydrogeologist. Monitoring wells or open-cased boreholes that are subject to tidal fluctuations should be read in conjunction with a tidal chart (or preferably in conjunction with readings of a tide staff or tide level recorder installed in the adjacent water body); the frequency of such readings shall be established by the site hydrogeologist. All water level measurements at a site used to develop a groundwater contour map shall be made in the shortest practical time to minimize affects due to weather changes.

5.3.2 Water Level Measuring Techniques

There are several methods for determining standing or changing water levels in boreholes and monitoring wells. Certain methods have particular advantages and disadvantages depending upon well conditions. A general description of these methods is presented, along with a listing of various advantages and disadvantages of each technique. An effective technique shall be selected for the particular site conditions by the site geologist/hydrogeologist.

In most instances, preparation of accurate potentiometric surface maps require that static water level measurements be obtained to a precision of 0.01 feet. To obtain such measurements in individual accessible wells, electrical water level indicator methods have been found to be best, and thus should be utilized. Other, less precise methods, such as the popper or bell sound, or bailer line methods, should be avoided. When a large number of (or continuous) readings are required, time-consuming individual readings are not usually feasible. In such cases, it is best to use a pressure transducer.

5.3.3 Methods

Water levels can be measured by several different techniques, but the same steps shall be followed in each case. The proper sequence is as follows:

1. Check operation of recording equipment above ground. Prior to opening the well, don personal protective equipment, as required. Never remove an air-tight lock (such as a J-plug) with your face over the well. Pressure changes within the well may explosively force the cap off once loosened.

2. Record all information specified below in the geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet (Attachment B):

   - Well number.
   - Water level (to the nearest 0.01 foot). Water levels shall be taken from the surveyed reference mark on the top edge of the inner well casing. If the J-plug was on the well very tightly, it may take several minutes for the water level to stabilize.
   - Time and day of the measurement.
   - Thickness of free product if present.

Water level measuring devices with permanently marked intervals shall be used. The devices shall be free of kinks or folds which will affect the ability of the equipment to hang straight in the well pipe.

5.3.4 Water Level Measuring Devices

Electric Water Level Indicators

These are the most commonly used devices and consist of a spool of small-diameter cable and a weighted probe attached to the end. When the probe comes in contact with the water, an electrical circuit is closed and a meter, light, and/or buzzer attached to the spool will signal the contact.

There are a number of commercial electric sounders available, none of which is entirely reliable under all conditions likely to occur in a contaminated monitoring well. In conditions where there is oil on the water, groundwater with high specific conductance, water cascading into the well, steel well casing, or a turbulent water surface in the well, measuring with an electric sounder may be difficult.

For accurate readings, the probe shall be lowered slowly into the well adjacent to the survey mark on the inner well casing. The electric tape is read (to the nearest 0.01 ft.) at the measuring point and recorded where contact with the water surface was indicated.

Popper or Bell Sounder

A bell- or cup-shaped weight that is hollow on the bottom is attached to a measuring tape and lowered into the well. A "plopping" or "popping" sound is made when the weight strikes the surface of the water. An accurate reading can be determined by lifting and lowering the weight in short strokes, and reading the tape when the weight strikes the water. This method is not sufficiently accurate to obtain water levels to 0.01 feet, and thus is more appropriate for obtaining only approximate water levels quickly.

Pressure Transducer

Pressure transducers can be lowered into a well or borehole to measure the pressure of water and therefore the water elevation above the transducer. The transducer is wired into a recorder at the surface to record changes in water level with time. The recorder digitizes the information and can provide a printout or transfer the information to a computer for evaluation (using a well drawdown/recovery model). The pressure transducer should be initially calibrated with another water level measurement technique to ensure accuracy. This technique is very useful for hydraulic conductivity testing in highly permeable material where repeated, accurate water level measurements are required in a very short period of time. A sensitive transducer element is required to measure water levels to 0.01 foot accuracy.

Borehole Geophysics

Approximate water levels can be determined during geophysical logging of the borehole (although this is not the primary purpose for geophysical logging and such logging is not cost effective if used only for this purpose). Several logging techniques will indicate water level. Commonly-used logs which will indicate saturated/unsaturated conditions include the spontaneous potential (SP) log and the neutron log.

5.3.5 Data Recording

Water level measurements, time, data, and weather conditions shall be recorded in the geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet. All water level measurements shall be measured from a known reference point. The reference point is generally a marked point on the upper edge of the inner well casing that has been surveyed for an elevation. The exact reference point shall be marked with permanent ink on the casing since the top of the casing may not be entirely level. It is important to note changes in weather conditions because changes in the barometric pressure may affect the water level within the well.

5.3.6 Specific Quality Control Procedures for Water Level Measuring Devices

All groundwater level measurement devices must be cleaned before and after each use to prevent cross contamination of wells. Manufacturer's instructions for cleaning the device shall be strictly followed. Some devices used to measure groundwater levels may need to be calibrated. These devices shall be calibrated to 0.01 foot accuracy and any adjustments/corrections shall be recorded in the field logbook/notebook. After the corrections/adjustments are made to the measuring device and entered in the field logbook/notebook, the corrected readings shall be entered onto the Groundwater Level Measurement Sheet (Attachment B). Elevations will be entered on the sheet when they become available.

5.4 Equipment Decontamination

Equipment used for water level measurements provide a mechanism for potentially cross contaminating wells. Therefore, all portions of a device which project down the well casing must be decontaminated prior to advancing to the next well. Decontamination procedures vary based on the project objectives but must be defined prior to conducting any field activities including the collection of water level data. Consult the project planning documents and SA-7.1 Decontamination of Field Equipment.

5.5 Health and Safety Considerations

Groundwater contaminated by volatile organic compounds may release toxic vapors into the air space inside the well pipe. The release of this air when the well is initially opened is a health/safety hazard which must be considered. Initial monitoring of the well headspace and breathing zone concentrations using a PID or FID shall be performed to determine required levels of protection. Under certain conditions, air-tight well caps may explosively fly off the well when the pressure is relieved. Never stand directly over a well when uncapping it.

6.0 RECORDS

A record of all field procedures, tests and observations must be recorded in the site logbook or designated field notebook. Entries in the log/notebook should include the individuals participating in the field effort, and the date and time. The use of annotated sketches may help to supplement the evaluation.

ATTACHMENT A
MONITORING WELL INSPECTION SHEET

Monitoring Well Inspection Sheet

Project Name: _____ Date: _____

Location: _____ Time: _____

Tidally Influenced: Y / N Personnel: _____

Field Measurements				
Well ID	PID Reading PPM	Depth to Water *	Total Depth *	Flush Mt./ Stick-up

Well Construction Details (Taken from construction logs)		
Total Depth *	Ground Elev.	Top/Btm Screen *

Check List:

Riser Pipe Material:
Riser Notched for Surveyors:
Well ID Tag In-place:
Well security:
Photo taken:

Condition of Well:

Protective Case:
Riser:
Well Pad:
Other:

Presence/Evidence of:

Standing Water Around Well:
Existing Sampling Equipment:
Sediment build-up in Well Btm:

Comments:

** = Measurements are from the top of the inner case to the nearest 0.01'*

ATTACHMENT B
GROUNDWATER LEVEL MESUREMENT SHEET

Tetra Tech NUS, Inc.

GROUNDWATER LEVEL MEASUREMENT SHEET

Project Name: _____ **Project No.:** _____
Location: _____ **Personnel:** _____
Weather Conditions: _____ **Measuring Device:** _____
Tidally Influenced: Yes ____ No ____ **Remarks:** _____

Well or Piezometer Number	Date	Time	Elevation of Reference Point (feet)*	Water Level Indicator Reading (feet)*	Thickness of Free Product (feet)*	Comments

* All measurements to the nearest 0.01 foot, from surveyed mark (unless noted)

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1.0 PURPOSE

The purpose of this procedure is to describe the methods and equipment necessary to perform soil and rock borings and identify the equipment, sequence of events, and appropriate methods necessary to obtain soil, both surface and subsurface, and rock samples during field sampling activities.

2.0 SCOPE

This guideline addresses most of the accepted and standard drilling techniques, their benefits, and drawbacks. It should be used generally to determine what type of drilling techniques would be most successful depending on site-specific geologic conditions and the type of sampling required.

The sampling methods described within this procedure are applicable to collecting surface and subsurface soil samples, and obtaining rock core samples for lithologic and hydrogeologic evaluation, excavation/foundation design, remedial alternative design and related civil engineering purposes.

3.0 GLOSSARY

Rock Coring - A method in which a continuous solid cylindrical sample of rock or compact rock-like soil is obtained by the use of a double tube core barrel that is equipped with an appropriate diamond-studded drill bit which is advanced with a hydraulic rotary drilling machine.

Wire-Line Coring - As an alternative to conventional coring, this technique is valuable in deep hole drilling, since this method eliminates trips in and out of the hole with the coring equipment. With this technique, the core barrel becomes an integral part of the drill rod string. The drill rod serves as both a coring device and casing.

4.0 RESPONSIBILITIES

Project Manager - In consultation with the project geologist, the Project Manager is responsible for evaluating the drilling requirements for the site and specifying drilling techniques that will be successful given the study objectives and the known or suspected geologic conditions at the site. The Project Manager also determines the disposal methods for products generated by drilling, such as drill cuttings and well development water, as well as any specialized supplies or logistical support required for the drilling operations.

Field Operations Leader (FOL) - The FOL is responsible for the overall supervision and scheduling of drilling activities, and is strongly supported by the project geologist.

Project Geologist - The project geologist is responsible for ensuring that standard and approved drilling procedures are followed. The geologist will generate a detailed boring log for each test hole. This log shall include a description of materials, samples, method of sampling, blow counts, and other pertinent drilling and testing information that may be obtained during drilling (see SOPs SA-6.3 and GH-1.5). Often this position for inspecting the drilling operations may be filled by other geotechnical personnel, such as soils and foundation engineers, civil engineers, etc.

Determination of the exact location for borings is the responsibility of the site geologist. The final location for drilling must be properly documented on the boring log. The general area in which the borings are to be located will be shown on a site map included in the Work Plan and/or Sampling and Analysis Plan.

Drilling Subcontractor - Operates under the supervision of the FOL. Responsible for obtaining all drilling permits and clearances, and supplying all services (including labor), equipment and material required to perform the drilling, testing, and well installation program, as well as maintenance and quality control of such required equipment except as stated in signed and approved subcontracts.

The driller must report any major technical or analytical problems encountered in the field to the FOL within 24 hours of determination, and must provide advance written notification of any changes in field procedures, describing and justifying such changes. No such changes shall be made unless requested and authorized in writing by the FOL (with the concurrence of the Project Manager). Depending on the subcontract, the Project Manager may need to obtain written authorization from appropriate administrative personnel before approving any changes.

The drilling subcontractor is responsible for following decontamination procedures specified in the project plan documents. Upon completion of the work, the driller is responsible for demobilizing all equipment, cleaning up any materials deposited on site during drilling operations, and properly backfilling any open borings.

5.0 PROCEDURES

5.1 General

The purpose of drilling boreholes is:

- To determine the type, thickness, and certain physical and chemical properties of the soil, water and rock strata which underlie the site.
- To install monitoring wells or piezometers.

All drilling and sampling equipment will be cleaned between samples and borings using appropriate decontamination procedures as outlined in SOP SA-7.1. Unless otherwise specified, it is generally advisable to drill borings at "clean" locations first, and at the most contaminated locations last, to reduce the risk of spreading contamination between locations. All borings must be logged by the site geologist as they proceed (see SOPs SA-6.3 and GH-1.5). Situations where logging would not be required would include installation of multiple well points within a small area, or a "second attempt" boring adjacent to a boring that could not be continued through resistant material. In the latter case, the boring log can be resumed 5 feet above the depth at which the initial boring was abandoned, although the site geologist should still confirm that the stratigraphy at the redrilled location conforms essentially with that encountered at the original location. If significant differences are seen, each hole should be logged separately.

5.2 Drilling Methods

The selected drilling methods described below apply to drilling in subsurface materials, including, but not limited to, sand, gravel, clay, silt, cobbles, boulders, rock and man-made fill. Drilling methods should be selected after studying the site geology and terrain, the waste conditions at the site, and reviewing the purpose of drilling and the overall subsurface investigation program proposed for the site. The full range of different drilling methods applicable to the proposed program should be identified with final selection based on relative cost, availability, time constraints, and how well each method meets the sampling and testing requirements of the individual drilling program.

5.2.1 Continuous-Flight Hollow-Stem Auger Drilling

This method of drilling consists of rotating augers with a hollow stem into the ground. Cuttings are brought to the surface by the rotating action of the auger. This method is relatively quick and inexpensive. Advantages of this type of drilling include:

- Samples can be obtained without pulling the augers out of the hole. However, this is a poor method for obtaining grab samples from thin, discrete formations because of mixing of soils which occurs as the material is brought to the surface. Sampling of such formations requires the use of split-barrel or thin-wall tube samplers advanced through the hollow core of the auger.
- No drilling fluids are required.
- A well can be installed inside the auger stem and backfilled as the augers are withdrawn.

Disadvantages and limitations of this method of drilling include:

- Augering can only be done in unconsolidated materials.

- The inside diameter of hollow stem augers used for well installation should be at least 4 inches greater than the well casing. Use of such large-diameter hollow-stem augers is more expensive than the use of small-diameter augers in boreholes not used for well installation. Furthermore, the density of unconsolidated materials and depths become more of a limiting factor. More friction is produced with the larger diameter auger and subsequently greater torque is needed to advance the boring.

- The maximum effective depth for drilling is 150 feet or less, depending on site conditions and the size of augers used.

- In augering through clean sand formations below the water table, the sand will tend to flow into the hollow stem when the plug is removed for soil sampling or well installation. If the condition of "running" or "flowing" sands is persistent at a site, an alternative method of drilling is recommended, in particular for wells or boreholes deeper than 25 feet.

Hollow-stem auger drilling is the preferred method of drilling. Most alternative methods require the introduction of water or mud downhole (air rotary is the exception) to maintain the open borehole. With these other methods, great care must be taken to ensure that the method does not interfere with the collection of a representative sample (which may be the prime objective of the borehole construction). With this in mind, the preferred order of choice of drilling method after hollow-stem augering (HSA) is:

- Cable tool
- Casing drive (air)
- Air rotary
- Mud rotary
- Rotosonic
- Drive and wash
- Jetting

However, the use of any method will also depend on efficiency and cost-effectiveness. In many cases, mud rotary is the only feasible alternative to hollow-stem augering. Thus, mud rotary drilling is generally acceptable as a first substitute for HSA.

The procedures for sampling soils through holes drilled by hollow-stem auger shall conform with the applicable ASTM Standards: D1587-83 and D1586-84. The guidelines established in SOP SA-1.3 shall

also be followed. The hollow-stem auger may be advanced by any power-operated drilling machine having sufficient torque and ram range to rotate and force the auger to the desired depth. The machine must, however, be equipped with the accessory equipment needed to perform required sampling, or rock coring.

The hollow-stem auger may be used without the plug when boring for geotechnical examination or for well installation. However, when drilling below the water table, specially designed plugs which allow passage of formation water but not solid material shall be used (see Reference 1 of this guideline). This drilling configuration method also prevents blow back and plugging of the auger when the plug is removed for sampling.

Alternately, it may be necessary to keep the hollow stem full of water, at least to the level of the water table, to prevent blowback and plugging of the auger. If water is added to the hole, it must be sampled and analyzed to determine if it is free from contaminants prior to use. In addition, the amount of water introduced, the amount recovered upon attainment of depth, and the amount of water extracted during well development must be carefully logged in order to ensure that a representative sample of the formation water can be obtained. Well development should occur as soon after well completion as practicable (see SOP GH-2.8 for well development procedures). If gravelly or hard material is encountered which prevents advancing the auger to the desired depth, augering should be halted and either driven casing or hydraulic rotary methods should be attempted. If the depth to the bedrock/soil interface and bedrock lithology must be determined, then a 5-foot confirmatory core run should be conducted (see Section 5.2.9).

At the option of the Field Operations Leader (in communication with the Project Manager), when resistant materials prevent the advancement of the auger, a new boring can be attempted. The original boring must be properly backfilled and the new boring started a short distance away at a location determined by the site geologist. If multiple water bearing strata were encountered, the original boring must be grouted. In some formations, it may be prudent to also grout borings which penetrate only the water table aquifer, since loose soil backfill in the boring may still provide a preferred pathway for surface liquids to reach the water table. Backfilling requirements may also be driven by state or local regulations.

5.2.2 Continuous-Flight Solid-Stem Auger Drilling

This drilling method is similar to hollow-stem augering. Practical application of this method is severely restricted compared to use of hollow-stem augers. Split-barrel (split-spoon) sampling cannot be performed without pulling the augers out, which may allow the hole to collapse. The continuous-flight solid-stem auger drilling method is therefore very time consuming and is not cost effective. Also, augers would have to be withdrawn before installing a monitoring well, which again, may allow the hole to collapse. Furthermore, geologic logging by examining the soils brought to the surface is unreliable, and depth to water may be difficult to determine while drilling.

There would be very few situations where use of a solid-stem auger would be preferable to other drilling methods. The only practical applications of this method would be to drill boreholes for well installation where no lithologic information is desired and the soils are such that the borehole can be expected to remain open after the augers are withdrawn. Alternatively, this technique can be used to find depth to bedrock in an area when no other information is required from drilling.

5.2.3 Rotary Drilling

Direct rotary drilling includes air-rotary and fluid-rotary drilling. For air or fluid-rotary drilling, the rotary drill may be advanced to the desired depth by any power-operated drilling machine having sufficient torque

and ram range to rotate and force the bit to the desired depth. The drilling machine must, however, be equipped with any accessory equipment needed to perform required sampling, or coring. Prior to sampling, any settled drill cuttings in the borehole must be removed.

Air-rotary drilling is a method of drilling where the drill rig simultaneously turns and exerts a downward pressure on the drilling rods and bit while circulating compressed air down the inside of the drill rods, around the bit, and out the annulus of the borehole. Air circulation serves to both cool the bit and remove the cuttings from the borehole. Advantages of this method include:

- The drilling rate is high (even in rock).
- The cost per foot of drilling is relatively low.
- Air-rotary rigs are common in most areas.
- No drilling fluid is required (except when water is injected to keep down dust).
- The borehole diameter is large, to allow room for proper well installation procedures.

Disadvantages to using this method include:

- Formations must be logged from the cuttings that are blown to the surface and thus the depths of materials logged are approximate.
- Air blown into the formation during drilling may "bind" the formation and impede well development and natural groundwater flow.
- In-situ samples cannot be taken, unless the hole is cased.
- Casing must generally be used in unconsolidated materials.
- Air-rotary drill rigs are large and heavy.
- Large amounts of Investigation Derived Waste (IDW) may be generated which may require containerization, sampling, and off-site disposal.

A variation of the typical air-rotary drill bit is a down hole hammer which hammers the drill bit down as it drills. This makes drilling in hard rock faster. Air-rotary drills can also be adapted to use for rock coring although they are generally slower than other types of core drills. A major application of the air-rotary drilling method would be to drill holes in rock for well installation.

Fluid-Rotary drilling operates in a similar manner to air-rotary drilling except that a drilling fluid ("mud") or clean water is used in place of air to cool the drill bit and remove cuttings. There are a variety of fluids that can be used with this drilling method, including bentonite slurry and synthetic slurries. If a drilling fluid other than water/cuttings is used, it must be a natural clay (i.e., bentonite) and a "background" sample of the fluid should be taken for analysis of possible organic or inorganic contaminants.

Advantages to the fluid-rotary drilling method include:

- The ability to drill in many types of formations.
- Relatively quick and inexpensive.
- Split-barrel (split-spoon) or thin-wall (Shelby) tube samples can be obtained without removing drill rods if the appropriate size drill rods and bits (i.e., fish-tail or drag bit) are used.

- In some borings temporary casing may not be needed as the drilling fluids may keep the borehole open.
- Drill rigs are readily available in most areas.

Disadvantages to this method include:

- Formation logging is not as accurate as with hollow-stem auger method if split-barrel (split-spoon) samples are not taken (i.e., the depths of materials logged from cuttings delivered to the surface are approximate).
- Drilling fluids reduce permeability of the formation adjacent to the boring to some degree, and require more extensive well development than "dry" techniques (augering, air-rotary).
- No information on depth to water is obtainable while drilling.
- Fluids are needed for drilling, and there is some question about the effects of the drilling fluids on subsequent water samples obtained. For this reason as well, extensive well development may be required.
- In very porous materials (i.e., rubble fill, boulders, coarse gravel) drilling fluids may be continuously lost into the formation. This requires either constant replenishment of the drilling fluid, or the use of casing through this formation.
- Drill rigs are large and heavy, and must be supported with supplied water.
- Groundwater samples can be potentially diluted with drilling fluid.

The procedures for performing direct rotary soil investigations and sampling shall conform with the applicable ASTM standards: D2113-83, D1587-83, and D1586-84.

Soil samples shall be taken as specified by project plan documents, or more frequently, if requested by the project geologist. Any required sampling shall be performed by rotation, pressing, or driving in accordance with the standard or approved method governing use of the particular sampling tool.

When field conditions prevent the advancement of the hole to the desired depth, a new boring may be drilled at the request of the Field Operations Leader. The original boring shall be backfilled using methods and materials appropriate for the given site and a new boring started a short distance away at a location determined by the project geologist.

5.2.4 Rotosonic Drilling

The Rotosonic drilling method employs a high frequency vibrational and low speed rotational motion coupled with down pressure to advance the cutting edge of a drill string. This produces a uniform borehole while providing a continuous, undisturbed core sample of both unconsolidated and most bedrock formations. Rotosonic drilling advances a 4-inch diameter to 12-inch diameter core barrel for sampling and can advance up to a 12-inch diameter outer casing for the construction of standard and telescoped monitoring wells. During drilling, the core barrel is advanced ahead of the outer barrel in increments as determined by the site geologist and depending upon type of material, degree of subsurface contamination and sampling objectives.

The outer casing can be advanced at the same time as the inner drill string and core barrel, or advanced down over the inner drill rods and core barrel, or after the core barrel has moved ahead to collect the undisturbed sample and has been pulled out of the borehole. The outer casing can be advanced dry in most cases, or can be advanced with water or air depending upon the formations being drilled, the depth and diameter of the hole, or requirements of the project.

Advantages of this method include:

- Sampling and well installation are faster as compared to other drilling methods.
- Continuous sampling, with larger sample volume as compared to split-spoon sampling.
- The ability to drill through difficult formations such as cobbles or boulders, hard till and bedrock.
- Reduction of IDW by an average of 70 to 80 percent.
- Well installations are quick and controlled by elimination of potential bridging of annular materials during well installation, due to the ability to vibrate the outer casing during removal.

Disadvantages include:

- The cost for Rotosonic drilling as compared to other methods are generally higher. However, the net result can be a significant savings considering reduced IDW and shortened project duration.
- Rotosonic drill rigs are large and need ample room to drill, however, Rotosonic units can be placed on the ground or placed on an ATV.
- There are a limited number of Rotosonic drilling contractors at the present time.

5.2.5 Reverse Circulation Rotary Drilling

The common reverse-circulation rig is a water or mud-rotary rig with a large-diameter drill pipe which circulates the drilling water down the annulus and up the inside of the drill pipe (reverse flow direction from direct mud-rotary). This type of rig is used for the construction of large-capacity production water wells and is not suited for small, water quality sampling wells because of the use of drilling muds and the large-diameter hole which is created. A few special reverse-circulation rotary rigs are made with double-wall drill pipe. The drilling water or air is circulated down the annulus between the drill pipes and up inside the inner pipe.

Advantages of the latter method include:

- The formation water is not contaminated by the drilling water.
- Formation samples can be obtained, from known depths.
- When drilling with air, immediate information is available regarding the water-bearing properties of formations penetrated.
- Collapsing of the hole in unconsolidated formations is not as great a problem as when drilling with the normal air-rotary rig.

Disadvantages include:

- Double-wall, reverse-circulation drill rigs are rare and expensive to operate.
- Placing cement grout around the outside of the well casing above a well screen often is difficult, especially when the screen and casing are placed down through the inner drill pipe before the drill pipe is pulled out.

5.2.6 Drill-through Casing Driver

The driven-casing method consists of alternately driving casing (fitted with a sharp, hardened casing shoe) into the ground using a hammer lifted and dropped by the drill rig (or an air-hammer) and cleaning out the casing using a rotary chopping bit and air or water to flush out the materials. The casing is driven down in stages (usually 5 feet per stage); a continuous record is kept of the blows per foot in driving the casing (see SOP GH-1.5). The casing is normally advanced by a 300-pound hammer falling freely through a height of 30 inches. Simultaneous washing and driving of the casing is not recommended. If this procedure is used, the elevations within which wash water is used and in which the casing is driven must be clearly recorded.

The driven casing method is used in unconsolidated formations only. When the boring is to be used for later well installation, the driven casing used should be at least 4 inches larger in diameter than the well casing to be installed. Advantages to this method of drilling include:

- Split-barrel (split-spoon) sampling can be conducted while drilling.
- Well installation is easily accomplished.
- Drill rigs used are relatively small and mobile.
- The use of casing minimizes flow into the hole from upper water-bearing layers; therefore, multiple aquifers can be penetrated and sampled for rough field determinations of some water quality parameters.

Some of the disadvantages include:

- This method can only be used in unconsolidated formations.
- The method is slower than other methods (average drilling progress is 30 to 50 feet per day).
- Maximum depth of the borehole varies with the size of the drill rig and casing diameter used, and the nature of the formations drilled.
- The cost per hour or per foot of drilling may be substantially higher than other drilling methods.
- It is difficult and time consuming to pull back the casing if it has been driven very deep (deeper than 50 feet in many formations).

5.2.7 Cable Tool Drilling

A cable tool rig uses a heavy, solid-steel, chisel-type drill bit ("tool") suspended on a steel cable, which when raised and dropped, chisels or pounds a hole through the soils and rock. Drilling progress may be

expedited by the use of "slip-jars" which serve as a cable-activated down hole percussion device to hammer the bit ahead.

When drilling through the unsaturated zone, some water must be added to the hole. The cuttings are suspended in the water and then bailed out periodically. Below the water table, after sufficient ground water enters the borehole to replace the water removed by bailing, no further water needs to be added. When soft caving formations are encountered, it is usually necessary to drive casing as the hole is advanced to prevent collapse of the hole. Often the drilling can be only a few feet below the bottom of the casing. Because the drill bit is lowered through the casing, the hole created by the bit is smaller than the casing. Therefore, the casing (with a sharp, hardened casing shoe on the bottom) must be driven into the hole (see Section 5.2.5 of this guideline).

Advantages of the cable-tool method include the following:

- Information regarding water-bearing zones is readily available during the drilling. Even relative permeabilities and rough water quality data from different zones penetrated can be obtained by skilled operators.
- The cable-tool rig can operate satisfactorily in all formations, but is best suited for caving, boulder, cobble or coarse gravel type formations (e.g., glacial till) or formations with large cavities above the water table (such as limestones).
- When casing is used, the casing seals formation water out of the hole, preventing down hole contamination and allowing sampling of deeper aquifers for field-measurable water quality parameters.
- Split-barrel (split-spoon) or thin-wall (Shelby) tube samples can be collected through the casing.

Disadvantages include:

- Drilling is slow compared with rotary rigs.
- The necessity of driving the casing in unconsolidated formations requires that the casing be pulled back if exposure of selected water-bearing zones is desired. This process complicates the well completion process and often increases costs. There is also a chance that the casing may become stuck in the hole.
- The relatively large diameters required (minimum of 4-inch casing) plus the cost of steel casing result in higher costs compared to rotary drilling methods where casing is not required (e.g., such use of a hollow-stem auger).
- Cable-tool rigs have largely been replaced by rotary rigs. In some parts of the U.S., availability may be difficult.

5.2.8 Jet Drilling (Washing)

Jet drilling, which should be used only for piezometer or vadose zone sampler installation, consists of pumping water or drilling mud down through a small diameter (1/2- to 2-inch) standard pipe (steel or PVC). The pipe may be fitted with a chisel bit or a special jetting screen. Formation materials dislodged by the bit and jetting action of the water are brought to the surface through the annulus around the pipe. As the pipe is jetted deeper, additional lengths of pipe may be added at the surface.

Jet percussion is a variation of the jetting method, in which the casing is driven with a drive weight. Normally, this method is used to place 2-inch-diameter casing in shallow, unconsolidated sand formations, but this method has also been used to install 3- to 4-inch-diameter casings to a depth of 200 feet.

Jetting is acceptable in very soft formations, usually for shallow sampling, and when introduction of drilling water to the formation is acceptable. Such conditions would occur during rough stratigraphic investigation or installation of piezometers for water level measurement. Advantages of this method include:

- Jetting is fast and inexpensive.
- Because of the small amount of equipment required, jetting can be accomplished in locations where access by a normal drilling rig would be very difficult. For example, it would be possible to jet down a well point in the center of a lagoon at a fraction of the cost of using a drill rig.
- Jetting numerous well points just into a shallow water table is an inexpensive method for determining the water table contours, hence flow direction.

Disadvantages include the following:

- A large amount of foreign water or drilling mud is introduced above and into the formation to be sampled.
- Jetting is usually done in very soft formations which are subject to caving. Because of this caving, it is often not possible to place a grout seal above the screen to assure that water in the well is only from the screened interval.
- The diameter of the casing is usually limited to 2 inches.
- Jetting is only possible in very soft formations that do not contain boulders or coarse gravel, and the depth limitation is shallow (about 30 feet without jet percussion equipment).
- Large quantities of water are often needed.

5.2.9 Drilling with a Hand Auger

This method is applicable wherever the formation, total depth of sampling, and the site and groundwater conditions are such as to allow hand auger drilling. Hand augering can also be considered at locations where drill rig access is not possible. All hand auger borings will be performed according to ASTM D1452-80.

Samples should be taken continuously unless otherwise specified by the project plan documents. Any required sampling is performed by rotation, pressing, or driving in accordance with the standard or approved method governing use of the particular sampling tool. Typical equipment used for sampling and advancing shallow "hand auger" holes are Iwan samplers (which are rotated) or post hole diggers (which are operated like tongs). These techniques are slow but effective where larger pieces of equipment do not have access, and where very shallow holes are desired (less than 15 feet). Surficial soils must be composed of relatively soft and non-cemented formations to allow penetration by the auger.

5.2.10 Rock Drilling and Coring

When soil borings cannot be continued using augers or rotary methods due to the hardness of the soil or when rock or large boulders are encountered, drilling and sampling can be performed using a diamond bit corer in accordance with ASTM D2113.

Drilling is done by rotating and applying downward pressure to the drill rods and drill bit. The drill bit is a circular, hollow, diamond-studded bit attached to the outer core barrel in a double-tube core barrel. The use of single-tube core barrels is not recommended, as the rotation of the barrel erodes the sample and limits its use for detailed geological evaluation. Water or air is circulated down through the drill rods and annular space between the core barrel tubes to cool the bit and remove the cuttings. The bit cuts a core out of the rock which rises into an inner barrel mounted inside the outer barrel. The inner core barrel and rock core are removed by lowering a wire line with a coupling into the drill rods, latching onto the inner barrel and withdrawing the inner barrel. A less efficient variation of this method utilizes a core barrel that cannot be removed without pulling all of the drill rods. This variation is practical only if less than 50 feet of core is required.

Core borings are made through the casing used for the soil borings. The casing must be driven and sealed into the rock formation to prevent seepage from the overburden into the hole to be cored (see Section 5.3 of this guideline). A double-tube core barrel with a diamond bit and reaming shell or equivalent should be used to recover rock cores of a size specified in the project plans. The most common core barrel diameters are listed in Attachment A.

Soft or decomposed rock should be sampled with a driven split-barrel whenever possible or cored with a Denison or Pitcher sampler.

When coring rock, including shale and claystone, the speed of the drill and the drilling pressure, amount and pressure of water, and length of run can be varied to give the maximum recovery from the rock being drilled. Should any rock formation be so soft or broken that the pieces continually fall into the hole causing unsatisfactory coring, the hole should be reamed and a flush-joint casing installed to a point below the broken formation. The size of the flush-joint casing must permit securing the core size specified. When soft or broken rock is anticipated, the length of core runs should be reduced to less than 5 feet to avoid core loss and minimize core disturbance.

Advantages of core drilling include:

- Undisturbed rock cores can be recovered for examination and/or testing.
- In formations in which the cored hole will remain open without casing, water from the rock fractures may be recovered from the well without the installation of a well screen and gravel pack.
- Formation logging is extremely accurate.
- Drill rigs are relatively small and mobile.

Disadvantages include:

- Water or air is needed for drilling.
- Coring is slower than rotary drilling (and more expensive).
- Depth to water cannot accurately be determined if water is used for drilling.
- The size of the borehole is limited.

This drilling method is useful if accurate determinations of rock lithology are desired or if open wells are to be installed into bedrock. To install larger diameter wells in coreholes, the hole must be reamed out to the proper size after boring, using air or mud rotary drilling methods.

5.2.11 Drilling & Support Vehicles

In addition to the drilling method required to accomplish the objectives of the field program, the type of vehicle carrying the drill rig and/or support equipment and its suitability for the site terrain, will often be an additional deciding factor in planning the drilling program. The types of vehicles available are extensive, and depend upon the particular drilling subcontractor's fleet. Most large drilling subcontractors will have a wide variety of vehicle and drill types suited for most drilling assignments in their particular region, while smaller drilling subcontractors will usually have a fleet of much more limited diversity. The weight, size, and means of locomotion (tires, tracks, etc.) of the drill rig must be selected to be compatible with the site terrain to assure adequate mobility between borehole locations. Such considerations also apply to necessary support vehicles used to transport water and/or drilling materials to the drill rigs at the borehole locations. When the drill rigs or support vehicles do not have adequate mobility to easily traverse the site, provisions must be made for assisting equipment, such as bulldozers, winches, timber planking, etc., to maintain adequate progress during the drilling program.

Some of the typical vehicles which are usually available for drill rigs and support equipment are:

- Totally portable drilling/sampling equipment, where all necessary components (tripods, samplers, hammers, catheads, etc.) may be hand carried to the borehole site. Drilling/sampling methods used with such equipment include:

  - Hand augers and lightweight motorized augers.
  - Retractable plug samplers--driven by hand (hammer).
  - Motorized cathead - a lightweight aluminum tripod with a small gas-engine cathead mounted on one leg, used to install small-diameter cased borings. This rig is sometimes called a "monkey on a stick."

- Skid-mounted drilling equipment containing a rotary drill or engine-driven cathead (to lift hammers and drill string), a pump, and a dismounted tripod. The skid is pushed, dragged, or winched (using the cathead drum) between boring locations.

- Small truck-mounted drilling equipment using a Jeep, stake body or other light truck (4 to 6 wheels), upon which are mounted the drill and/or a cathead, a pump, and a tripod or small drilling derrick. On some rigs, the drill and/or a cathead are driven by a power take-off from the truck, instead of by a separate engine.

- Track-mounted drilling equipment is similar to truck-mounted rigs, except that the vehicle used has wide bulldozer tracks for traversing soft ground. Sometimes a continuous-track "all terrain vehicle" is also modified for this purpose. Some types of tracked drill rigs are called "bombardier" or "weasel" rigs.

- Heavy truck-mounted drilling equipment is mounted on tandem or dual tandem trucks to transport the drill, derrick, winches, and pumps or compressors. The drill may be provided with a separate engine or may use a power take-off from the truck engine. Large augers, hydraulic rotary and reverse circulation rotary drilling equipment are usually mounted on such heavy duty trucks. For soft-ground sites, the drilling equipment is sometimes mounted on vehicles having low pressure, very wide diameter tires and capable of floating; these vehicles are called "swamp buggy" rigs.

- Marine drilling equipment is mounted on various floating equipment for drilling borings in lakes, estuaries and other bodies of water. The floating equipment varies, and is often manufactured or customized by the drilling subcontractor to suit specific drilling requirements. Typically, the range of flotation vehicles include:

  - Barrel-float rigs - a drill rig mounted on a timber platform buoyed by empty 55-gallon drums or similar flotation units.

  - Barge-mounted drill rigs.

- Jack-up platforms - drilling equipment mounted on a floating platform having retractable legs to support the unit on the sea or lake bed when the platform is jacked up out of the water.

- Drill ships - for deep ocean drilling.

In addition to the mobility for the drilling equipment, similar consideration must be given for equipment to support the drilling operations. Such vehicles or floating equipment are needed to transport drill water, drilling supplies and equipment, samples, drilling personnel, etc. to and/or from various boring locations.

5.2.12 Equipment Sizes

In planning subsurface exploration programs, care must be taken in specifying the various drilling components, so that they will fit properly in the boring or well.

For drilling open boreholes using rotary drilling equipment, tri-cone drill bits are employed with air, water or drilling mud to remove cuttings and cool the bit. Tri-cone bits are slightly smaller than the holes they drill (i.e., 5-7/8-inch or 7-7/8-inch bits will nominally drill 6-inch and 8-inch holes, respectively).

For obtaining split-barrel samples of a formation, samplers are commonly manufactured in sizes ranging from 2 inches to 3-1/2 inches in outside diameter. However, the most commonly used size is the 2-inch O.D., 1-3/8-inch I.D. split-barrel sampler. When this sampler is used and driven by a 140-pound (± 2-pound) hammer dropping 30 inches (± 1 inch), the procedure is called a Standard Penetration Test, and the blows per foot required to advance the sampler into the formation can be correlated to the formation's density or strength.

In planning the drilling of boreholes using hollow-stem augers or casing, in which thin-wall tube samples or diamond core drilling will be performed, refer to the various sizes and clearances provided in Attachment A of this guideline. Sizes selected must be stated in the project plan documents.

5.2.13 Estimated Drilling Progress

To estimate the anticipated rates of drilling progress for a site, the following must be considered:

- The speed of the drilling method employed.

- Applicable site conditions (e.g., terrain, mobility between borings, difficult drilling conditions in bouldery soils, rubble fill or broken rock, etc.).

- Project-imposed restrictions (e.g., drilling while wearing personal protective equipment, decontamination of drilling equipment, etc.).

Based on recent experience in drilling average soil conditions (no boulders) and taking samples at 5-foot intervals, for moderate depth (30 feet to 50 feet) boreholes (not including installation or development of wells), the following daily rates of total drilling progress may be anticipated for the following drilling methods:

Drilling Method	Average Daily Progress (linear feet)
Hollow-stem augers	75'
Solid-stem augers	50'
Mud-Rotary Drilling	100' (cuttings samples)
Rotosonic Drilling	100'-160' (continuous core)
Reverse-Circulation Rotary	100' (cuttings samples)
Skid-Rig with driven casing	30'
Rotary with driven casing	50'
Cable Tool	30'
Hand Auger	Varies
Continuous Rock Coring	50'

5.3 Prevention of Cross-Contamination

A telescoping or multiple casing technique minimizes the potential for the migration of contaminated groundwater to lower strata below a confining layer. The telescoping technique consists of drilling to a confining layer utilizing a spun casing method with a diamond cutting or augering shoe (a method similar to the rock coring method described in Section 5.2.10, except that larger casing is used) or by using a driven-casing method (see Section 5.2.6 of this guideline) and installing a specified diameter steel well casing. The operation consists of three separate steps. Initially, a drilling casing (usually of 8-inch diameter) is installed followed by installation of the well casing (6-inch-diameter is common for 2-inch wells). This well casing is driven into the confining layer to ensure a tight seal at the bottom of the hole. The well casing is sealed at the bottom with a bentonite-cement slurry. The remaining depth of the boring is drilled utilizing a narrower diameter spun or driven casing technique within the outer well casing. A smaller diameter well casing with an appropriate length of slotted screen on the lower end, is installed to the surface.

Clean sand is placed in the annulus around and to a point of about 2 feet above the screen prior to withdrawal of the drilling casing. The annular space above the screen and to a point 2 feet above the bottom of the outer well casing is sealed with a tremied cement-bentonite slurry which is pressure-grouted or displacement-grouted into the hole. The remaining casing annulus is backfilled with clean material and grouted at the surface, or it is grouted all the way to the surface.

5.4 Cleanout of Casing Prior to Sampling

The boring hole must be completely cleaned of disturbed soil, segregated coarse material and clay adhering to the inside walls of the casing. The cleaning must extend to the bottom edge of the casing and, if possible, a short distance further (1 or 2 inches) to bypass disturbed soil resulting from the advancement of the casing. Loss of wash water during cleaning should be recorded.

For disturbed samples both above and below the water table and where introduction of relatively large volumes of wash water is permissible, the cleaning operation is usually performed by washing the material out of the casing with water; however, the cleaning should never be accomplished with a strong, downward-directed jet which will disturb the underlying soil. When clean out has reached the bottom of the casing or slightly below (as specified above), the string of tools should be lifted one foot off the bottom with the water still flowing, until the wash water coming out of the casing is clear of granular soil particles. In formations where the cuttings contain gravel and other larger particles, it is often useful to repeatedly raise and lower the drill rods and wash bit while washing out the hole, to surge these large particles upward out of the hole. As a time saver, the drilling contractor may be permitted to use a split-barrel (split-spoon) sampler with the ball check valve removed as the clean-out tool, provided the material below the spoon is not disturbed and the shoe of the spoon is not damaged. However, because the ball check valve has been removed, in some formations it may be necessary to install a flap valve or spring sample retainer in the split-spoon bit, to prevent the sample from falling out as the sampler is withdrawn from the hole. The use of jet-type chopping bits is discouraged except where large boulders and cobbles or hard-cemented soils are encountered. If water markedly softens the soils above the water table, clean out should be performed dry with an auger.

For undisturbed samples below the water table, or where wash water must be minimized, clean out is usually accomplished with an appropriate diameter clean out auger. This auger has cutting blades at the bottom to carry loose material up into the auger, and up-turned water jets just above the cutting blades to carry the removed soil to the surface. In this manner, there is a minimum of disturbance at the top of the material to be sampled. If any gravel material washes down into the casing and cannot be removed by the clean out auger, a split-barrel sample can be taken to remove it; bailers and sandpumps should not be used. For undisturbed samples above the groundwater table, all operations must be performed in a dry manner.

If all of the cuttings created by drilling through the overlying formations are not cleaned from the borehole prior to sampling, some of the problems which may be encountered during sampling include:

- When sampling is attempted through the cuttings remaining in the borehole, all or part of the sampler may become filled with the cuttings. This limits the amount of sample from the underlying formation which can enter and be retained in the sampler, and also raises questions as to the validity of the sample.

- If the cuttings remaining in the borehole contain coarse gravel and/or other large particles, these may block the bit of the sampler and prevent any materials from the underlying formation from entering the sampler when the sampler is advanced.

- In cased borings, should sampling be attempted through cuttings which remain in the lower portion of the casing, these cuttings could cause the sampler to become bound into the casing, such that it becomes very difficult to either advance or retract the sampler.

- When sampler blow counts are used to estimate the density or strength of the formation being sampled, the presence of cuttings in the borehole will usually give erroneously high sample blow counts.

To confirm that all cuttings have been removed from the borehole prior to attempting sampling, it is important that the site geologist measure the "stickup" of the drill string. This is accomplished by measuring the assembled length of all drill rods and bits or samplers (the drill string) as they are lowered to the bottom of the hole, below some convenient reference point of the drill string, then measuring the height of this reference point above the ground surface. The difference of these measurements is the

depth of the drill string (lower end of the bit or sampler) below the ground surface, which must then be compared with the depth of sampling required (installed depth of casing or depth of borehole drilled). If the length of drill string below grade is more than the drilled or casing depth, the borehole has been cleaned too deeply, and this deeper depth of sampling must be recorded on the log. If the length of drill string below grade is less than the drilled or casing depth, the difference represents the thickness of cuttings which remain in the borehole. In most cases, an inch or two of cuttings may be left in the borehole with little or no problem. However, if more than a few inches of cuttings are encountered, the borehole must be recleaned prior to attempting sampling.

5.5 Materials of Construction

The effects of monitoring well construction materials on specific chemical analytical parameters are described and/or referenced in SOP GH-2.8. However, there are several materials used during drilling, particularly drilling fluids and lubricants, which must be used with care to avoid compromising the representativeness of soil and ground water samples.

The use of synthetic or organic polymer slurries is not permitted at any location where soil samples for chemical analysis are to be collected. These slurry materials could be used for installation of long-term monitoring wells, but the early time data in time series collection of ground water data may then be suspect. If synthetic or organic polymer muds are proposed for use at a given site, a complete written justification including methods and procedures for their use must be provided by the site geologist and approved by the Project Manager. The specific slurry composition and the concentration of suspected contaminants for each site must be known.

For many drilling operations, potable water is an adequate lubricant for drill stem and drilling tool connections. However, there are instances, such as drilling in tight clayey formations or in loose gravels, when threaded couplings must be lubricated to avoid binding. In these instances, to be determined in the field by the judgment of the site geologist and noted in the site logbook, and only after approval by the Project Manager, a vegetable oil or silicone-based lubricant should be used. Petroleum based greases, etc. will not be permitted. Samples of lubricants used must be provided and analyzed for chemical parameters appropriate to the given site.

5.6 Subsurface Soil Samples

Subsurface soil samples are used to characterize subsurface stratigraphy. This characterization can indicate the potential for migration of chemical contaminants in the subsurface. In addition, definition of the actual migration of contaminants can be obtained through chemical analysis of the soil samples. Where the remedial activities may include in-situ treatment or excavation and removal of the contaminated soil, the depth and areal extent of contamination must be known as accurately as possible.

Engineering and physical properties of soil may also be of interest should site construction activities be planned. Soil types, grain size distribution, shear strength, compressibility, permeability, plasticity, unit weight, and moisture content are some of the physical characteristics that may be determined for soil samples.

Penetration tests are also described in this procedure. The tests can be used to estimate various physical and engineering parameters such as relative density, unconfined compressive strength, and consolidation characteristics of soils.

Surface protocols for various soil sampling techniques are discussed in SOP SA-1.3. Continuous-core soil sampling and rock coring are discussed below. The procedures described here are representative of

a larger number of possible drilling and sampling techniques. The choice of techniques is based on a large number of variables such as cost, local geology, etc. The final choice of methods must be made with the assistance of drilling subcontractors familiar with the local geologic conditions. Alternative techniques must be based upon the underlying principles of quality assurance implicit in the following procedures.

The CME continuous sample tube system provides a method of sampling soil continuously during hollow-stem augering. The 5-foot sample barrel fits within the lead auger of a hollow-auger column. The sampling system can be used with a wide range of I.D. hollow-stem augers (from 3-1/4-inch to 8-1/4-inch I.D.). This method has been used to sample many different materials such as glacial drift, hard clays and shales, mine tailings, etc. This method is particularly used when SPT samples are not required and a large volume of material is needed. Also, this method is useful when a visual description of the subsurface lithology is required. Rotosonic drilling methods also provide a continuous soil sample.

5.7 Rock Sampling (Coring) (ASTM D2113-83)

Rock coring enables a detailed assessment of borehole conditions to be made, showing precisely all lithologic changes and characteristics. Because coring is an expensive drilling method, it is commonly used for shallow studies of 500 feet or less, or for specific intervals in the drill hole that require detailed logging and/or analyzing. Rock coring can, however, proceed for thousands of feet continuously, depending on the size of the drill rig, and yields better quality data than air-rotary drilling, although at a substantially reduced drilling rate. Rate of drilling varies widely, depending on the characteristics of lithologies encountered, drilling methods, depth of drilling, and condition of drilling equipment. Average output in a 10-hour day ranges from 40 to over 200 feet. Down hole geophysical logging or television camera monitoring is sometimes used to complement the data generated by coring.

Borehole diameter can be drilled to various sizes, depending on the information needed. Standard sizes of core barrels (showing core diameter) and casing are shown in Figure 1.

Core drilling is used when formations are too hard to be sampled by soil sampling methods and a continuous solid sample is desired. Usually, soil samples are used for overburden, and coring begins in sound bedrock. Casing is set into bedrock before coring begins to prevent loose material from entering the borehole, to prevent loss of drilling fluid, and to prevent cross-contamination of aquifers.

Drilling through bedrock is initiated by using a diamond-tipped core bit threaded to a drill rod (outer core barrel) with a rate of drilling determined by the downward pressure, rotation speed of drill rods, drilling fluid pressure in the borehole, and the characteristics of the rock (mineralogy, cementation, weathering).

FIGURE 1

STANDARD SIZES OF CORE BARRELS AND CASING

Coring Bit Size	Nominal*		Set Size*	
	O.D.	I.D.	O.D.	I.D.
RWT	1 5/32	3/4	1.160	0.735
EWT	1 1/2	29/32	1.470	0.905
EX, EXL, EWG, EWM	1 1/2	13/16	1.470	0.845
AWT	1 7/8	1 9/32	1.875	1.281
AX, AXL, AWG, AWM	1 7/8	1 3/16	1.875	1.185
BWT	2 3/8	1 3/4	2.345	1.750
BX, BXL, BWG, BWM	2 3/8	1 5/8	2.345	1.655
NWT	3	2 5/16	2.965	2.313
NX, NXL, NWG, NWM	3	2 1/8	2.965	2.155
HWT	3 29/32	3 3/16	3.889	3.187
HWG	3 29/32	3	3.889	3.000
2 3/4 x 3 7/8	3 7/8	2 3/4	3.840	2.690
4 x 5 1/2	5 1/2	4	5.435	3.970
6 x 7 3/4	7 3/4	6	7.655	5.970
AX Wire line ___|___/	1 7/8	1	1.875	1.000
BX Wire line ___|___/	2 3/8	1 7/16	2.345	1.437
NX Wire line ___|___/	3	1 15/16	2.965	1.937

* All dimensions are in inches; to convert to millimeters, multiply by 25.4.

___|___/ Wire line dimensions and designations may vary according to manufacturer.

FIGURE 1
STANDARD SIZES OF CORE BARRELS AND CASING
PAGE TWO

Size Designations			Casing Coupling					Approximate Core Diameter	
Casing; Casing coupling; Casing bits; Core barrel bits	Rod; rod couplings	Casing O.D., Inches	O.D., Inches	I.D., Inches	Casing bit O.D., Inches	Core barrel bit O.D., Inches*	Drill rod O.D., Inches	Normal, Inches	Thinwall, Inches
RX	RW	1.437	1.437	1.188	1.485	1.160	1.094	---	0.735
EX	E	1.812	1.812	1.500	1.875	1.470	1.313	0.845	0.905
AX	A	2.250	2.250	1.906	2.345	1.875	1.625	1.185	1.281
BX	B	2.875	2.875	2.375	2.965	2.345	1.906	1.655	1.750
NX	N	3.500	3.500	3.000	3.615	2.965	2.375	2.155	2.313
HX	HW	4.500	4.500	3.938	4.625	3.890	3.500	3.000	3.187
RW	RW	1.437	Flush Joint	No Coupling	1.485	1.160	1.094	---	0.735
EW	EW	1.812			1.875	1.470	1.375	0.845	0.905
AW	AW	2.250			2.345	1.875	1.750	1.185	1.281
BW	BW	2.875			2.965	2.345	2.125	1.655	1.750
NW	NW	3.500			3.615	2.965	2.625	2.155	2.313
HW	HW	4.500			4.625	3.890	3.500	3.000	3.187
PW	---	5.500			5.650	---	---	---	---
SW	---	6.625			6.790	---	---	---	---
UW	---	7.625			7.800	---	---	---	---
ZW	---	8.625			8.810	---	---	---	---
---	AX __|__\	---	---	---	---	1.875	1.750	1.000	---
---	BX __|__\	---	---	---	---	2.345	2.250	1.437	---
---	NX __|__\	---	---	---	---	2.965	2.813	1.937	---

* All dimensions are in inches; to convert to millimeters, multiply by 25.4.

___|___/ Wire line dimensions and designations may vary according to manufacturer.

NOMINAL DIMENSIONS FOR DRILL CASINGS AND ACCESSORIES. (DIAMOND CORE DRILL MANUFACTURERS ASSOCIATION). 288-D-2889

5.7.1 Diamond Core Drilling

A penetration of typically less than 6 inches per 50 blows using a 140-lb. hammer dropping 30 inches with a 2-inch split-barrel sampler shall be considered an indication that soil sampling methods may not be applicable and that coring may be necessary to obtain samples.

When formations are encountered that are too hard to be sampled by soil sampling methods, the following diamond core drilling procedure may be used:

- Firmly seat a casing into the bedrock or the hard material to prevent loose materials from entering the hole and to prevent the loss of drilling fluid return. Level the surface of the rock or hard material when necessary by the use of a fishtail or other bits. If the drill hole can be retained open without the casing and if cross-contamination of aquifers in the unconsolidated materials is unlikely, leveling may be omitted.

- Begin the core drilling using a double-tube swivel-core barrel of the desired size. After drilling no more than 10 feet (3 m), remove the core barrel from the hole and take out the core. If the core blocks the flow of the drilling fluid during drilling, remove the core barrel immediately. In soft materials, a large starting size may be specified for the coring tools; where local experience indicates satisfactory core recovery or where hard, sound materials are anticipated, a smaller size or the single-tube type may be specified and longer runs may be drilled. NX/NW size coring equipment is the most commonly used size.

- When soft materials are encountered that produce less than 50 percent recovery, stop the core drilling. If soil samples are desired, secure such samples in accordance with the procedures described in ASTM Method D 1586 (Split-barrel Sampling) or in Method D 1587 (Thin-Walled Tube Sampling); sample soils per SOP SA-1.3. Resume diamond core drilling when refusal materials are again encountered.

- Since rock structures and the occurrence of seams, fissures, cavities, and broken areas are among the most important items to be detected and described, take special care to obtain and record these features. If such broken zones or cavities prevent further advance of the boring, one of the following three steps shall be taken: (1) cement the hole; (2) ream and case; or (3) case and advance with the next smaller size core barrel, as conditions warrant.

- In soft, seamy, or otherwise unsound rock, where core recovery may be difficult, M-design core barrels may be used. In hard, sound rock where a high percentage of core recovery is anticipated, the single-tube core barrel may be employed.

5.7.2 Rock Sample Preparation and Documentation

Once the rock coring has been completed and the core recovered, the rock core shall be carefully removed from the barrel, placed in a core tray (previously labeled "top" and "bottom" to avoid confusion), classified, and measured for percentage of recovery as well as the rock quality designation (RQD). Each core shall be described, classified, and logged using a uniform system as presented in SOP GH-1.5. If moisture content will be determined or if it is desirable to prevent drying (e.g., to prevent shrinkage of clay formations) or oxidation of the core, the core shall be wrapped in plastic sleeves immediately after logging. Each plastic sleeve shall be labeled with indelible ink. The boring number, run number, and the footage represented in each sleeve shall be included, as well as designating the top and bottom of the core run.

After sampling, rock cores shall be placed in the sequence of recovery in well-constructed wooden boxes provided by the drilling contractor. Rock cores from two different borings shall not be placed in the same core box unless accepted by the Project Geologist. The core boxes shall be constructed to accommodate at least 20 linear feet of core in rows of approximately 5 feet each and shall be constructed with hinged tops secured with screws, and a latch (usually a hook and eye) to keep the top securely fastened down. Wood partitions shall be placed at the end of each core run and between rows.

The depth from the surface of the boring to the top and bottom of the drill run and run number shall be marked on the wooden partitions with indelible ink. A wooden partition (wooden block) shall be placed at the end of each run with the depth of the bottom of the run written on the block. These blocks will serve to separate successive core runs and indicate depth intervals for each run. The order of placing cores shall be the same in all core boxes. Rock core shall be placed in the box so that, when the box is open, with the inside of the lid facing the observer, the top of the cored interval contained within the box is in the upper left corner of the box, and the bottom of the cored interval is in the lower right corner of the box. The top and bottom of each core obtained and its true depth shall be clearly and permanently marked on each box. The width of each row must be compatible with the core diameter to prevent lateral movement of the core in the box. Similarly, an empty space in a row shall be filled with an appropriate filler material or spacers to prevent longitudinal movement of the core in the box.

The inside and outside of the core-box lid shall be marked by indelible ink to show all pertinent data on the box's contents. At a minimum, the following information shall be included:

- Project name.
- Project number.
- Boring number.
- Run numbers.
- Footage (depths).
- Recovery.
- RQD (%).
- Box number and total number of boxes for that boring (Example: Box 5 of 7).

For easy retrieval when core boxes are stacked, the sides and ends of the box shall also be labeled and include project number, boring number, top and bottom depths of core and box number.

Prior to final closing of the core box, a photograph of the recovered core and the labeling on the inside cover shall be taken. If moisture content is not critical, the core shall be wetted and wiped clean for the photograph. (This will help to show true colors and bedding features in the cores).

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ATTACHMENT A

DRILLING EQUIPMENT SIZES

Drilling Component	Designation or Hole Size (Inches)	O.D. (Inches)	I.D. (Inches)	Coupling I.D. (Inches)
Hollow-stem augers (Ref. 7)	6 1/4	5	2 1/4	
	6 3/4	5 3/4	2 3/4	---
	7 1/4	6 1/4	3 1/4	---
	13 1/4	12	6	---
Thin Wall Tube Samplers (Ref. 7)	---	2	1 7/8	---
	---	2 1/2	2 3/8	---
	---	3	2 7/8	---
	---	3 1/2	3 3/8	---
	---	4 1/2	4 3/8	---
	---	5	4 3/4	---
Drill Rods (Ref. 7)	RW	1 3/32	23/32	13/32
	EW	1 3/8	15/16	7/16
	AW	1 3/4	1 1/4	5/8
	BW	2 1/8	1 3/4	3/4
	NW	2 5/8	2 1/4	1 3/8
	HW	3 1/2	3 1/16	2 3/8
	E	1 5/16	7/8	7/16
	A	1 5/8	1 1/8	9/16
	B	1 7/8	1 1/4	5/8
	N	2 3/8	2	1
				Wall Thickness (Inches)
Driven External Coupled Extra Strong Steel* Casing (Ref. 8)	2 1/2	2.875	2.323	0.276
	3	3.5	2.9	0.300
	3 1/2	4.0	3.364	0.318
	4	4.5	3.826	0.337
	5	5.63	4.813	0.375
	6	6.625	5.761	0.432
	8	8.625	7.625	0.500
	10	10.750	9.750	0.500
	12	12.750	11.750	0.500

* Add twice the casing wall thickness to casing O.D. to obtain the approximate O.D. of the external pipe couplings.

ATTACHMENT A
DRILLING EQUIPMENT SIZES
PAGE TWO

Drilling Component	Designation or Hole Size (Inches)	O.D. (Inches)	I.D. (Inches)	Coupling I.D. (Inches)
Flush Coupled Casing (Ref. 7)	RX	1 7/16	1 3/16	1 3/16
	EX	1 13/16	1 5/8	1 1/2
	AX	2 1/4	2	1 29/32
	BX	2 7/8	2 9/16	2 3/8
	NX	3 1/2	3 3/16	3
	HX	4 1/2	4 1/8	3 15/16
Flush Joint Casing (Ref. 7)	RW	1 7/16	1 3/16	
	EW	1 13/16	1 1/2	
	AW	2 1/4	1 29/32	
	BW	2 7/8	2 3/8	
	NW	3 1/2	3	
	HW	4 1/2	4	
	PW	5 1/2	5	
	SW	6 5/8	6	
	UW	7 5/8	7	
	ZW	8 5/8	8	
Diamond Core Barrels (Ref. 7)	EWM	1 1/2	7/8**	
	AWM	1 7/8	1 1/8**	
	BWM	2 3/8	1 5/8**	
	NWM	3	2 1/8	
	HWG	3 7/8	3	
	2 3/4 x 3 7/8	3 7/8	2 11/16	
	4 x 5 1/2	5 1/2	3 15/16	
	6 x 7 3/4	7 3/4	5 15/16	
	AQ (wireline)	1 57/64	1 1/16**	
	BQ (wireline)	2 23/64	1 7/16**	
	NQ (wireline)	2 63/64	1 7/8	
	HQ (wireline)	3 25/32	2 1/2	

** Because of the fragile nature of the core and the difficulty to identify rock details, use of small-diameter core (1 3/8") is not recommended.

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1.0 PURPOSE

The purpose of this document is to establish standard procedures and technical guidance on borehole and sample logging.

2.0 SCOPE

These procedures provide descriptions of the standard techniques for borehole and sample logging. These techniques shall be used for each boring logged to provide consistent descriptions of subsurface lithology. While experience is the only method to develop confidence and accuracy in the description of soil and rock, the field geologist/engineer can do a good job of classification by careful, thoughtful observation and by being consistent throughout the classification procedure.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Site Geologist. Responsible for supervising all boring activities and assuring that each borehole is completely logged. If more than one rig is being used on site, the Site Geologist must make sure that each field geologist is properly trained in logging procedures. A brief review or training session may be necessary prior to the start up of the field program and/or upon completion of the first boring.

5.0 PROCEDURES

The classification of soil and rocks is one of the most important jobs of the field geologist/engineer. To maintain a consistent flow of information, it is imperative that the field geologist/engineer understand and accurately use the field classification system described in this SOP. This identification is based on visual examination and manual tests.

5.1 Materials Needed

When logging soil and rock samples, the geologist or engineer may be equipped with the following:

- Rock hammer
- Knife
- Camera
- Dilute hydrochloric acid (HCl)
- Ruler (marked in tenths and hundredths of feet)
- Hand Lens

5.2 Classification of Soils

All data shall be written directly on the boring log (Figure 1) or in a field notebook if more space is needed. Details on filling out the boring log are discussed in Section 5.5.

FIGURE 1
BORING LOG (EXAMPLE)

BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING NUMBER: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	Color	Material Classification	USCS *	Remarks	PID/FID Reading (ppm): Sample	Sampler BZ	Borehole**	Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.

Drilling Area Background (ppm): ____

Remarks: _____

Converted to Well: Yes _____ No _____ Well I.D. #: _____

FIGURE 1 (CONTINUED)

SOIL TERMS

UNIFIED SOIL CLASSIFICATION (USCS)

COARSE-GRAINED SOILS More Than Half of Material is LARGER Than No. 200 Sieve Size					FINE-GRAINED SOILS More Than Half of Material is SMALLER Than No. 200 Sieve Size						
FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights)			GROUP SYMBOL	TYPICAL NAMES	FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights)				GROUP SYMBOL	TYPICAL NAMES
					Identification Procedures on Fraction Smaller than No. 40 Sieve Size					
						DRY STRENGTH (Crushing Characteristics)	DILATANCY (Reaction to Shaking)	TOUGHNESS (Consistency Near Plastic Limit)		
GRAVELS (More Than 50% of Coarse Fraction Larger Than 4.75 mm)	CLEAN GRAVELS (Less than 5% Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	GW	Well graded gravels, gravel-sand mixtures, little or no fines.	SILTS AND CLAYS Liquid Limit < 50	None to Slight	Quick to Slow	None	ML	Inorganic silts and very fine sands, rock flour, silty or clayey fine sands with slight plasticity.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	GP	Poorly graded gravels, gravel-sand mixtures, little or no fines.		Medium to High	None to Very Slow	Medium	CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays.
	GRAVELS WITH FINES (More Than 12% Fines)	Non-plastic fines (for identification procedures, see ML)	GM	Silty gravels, poorly graded gravel-sand-silt mixtures.		Slight to Medium	Slow	Slight	OL	Organic silts and organic silt-clays of low plasticity.
		Plastic fines (for identification procedures, see CL)	GC	Clayey gravels, poorly graded gravel-sand-clay mixtures.	SILTS AND CLAYS Liquid Limit > 50	Slight to Medium	Slow to None	Slight to Medium	MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts.
SANDS (More Than 50% of Coarse Fraction Smaller Than 4.75 mm)	CLEAN SANDS (Less Than 5% Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	SW	Well graded sand, gravelly sands, little or no fines.		High to Very High	None	High	CH	Inorganic clays of high plasticity, fat clays.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	SP	Poorly graded sands, gravelly sands, little or no fines.		Medium to High	None to Very Slow	Slight to Medium	OH	Organic clays of medium to high plasticity.
	SANDS WITH FINES (More Than 12% Fines)	Non-plastic fines (for identification procedures, see ML)	SM	Silty sands, poorly graded sand-silt mixtures.	HIGHLY ORGANIC SOILS	Readily identified by color, odor, spongy feel and frequently by fibrous texture.			Pt	Peat and other organic soils
		Plastic fines (for identification procedures, see CL)	SC	Clayey sands, poorly graded sand-clay mixtures.						

Boundary classifications: Soils possessing characteristics of two groups are designated by combining group symbols. For example, GW-GC, well graded gravel-sand mixture with clay binder.
All sieve sizes on this chart are U.S. Standard.

DENSITY OF GRANULAR SOILS	
DESIGNATION	STANDARD PENETRATION RESISTANCE-BLOWS/FOOT
Very Loose	0-4
Loose	5-10
Medium Loose	11-30
Dense	31-50
Very Dense	Over 50

CONSISTENCY OF COHESIVE SOILS			
CONSISTENCY	UNCONFINED COMPRESSIVE STRENGTH (TONS/SQ. FT.)	STANDARD PENETRATION RESISTANCE-BLOWS/FOOT	FIELD IDENTIFICATION METHODS
Very Soft	Less than 0.25	0 to 2	Easily penetrated several inches by fist
Soft	0.25 to 0.50	2 to 4	Easily penetrated several inches by thumb.
Medium Stiff	0.50 to 1.0	4 to 8	Can be penetrated several inches by thumb.
Stiff	1.0 to 2.0	8 to 15	Readily indented by thumb.
Very Stiff	2.0 to 4.0	15 to 30	Readily indented by thumbnail.
Hard	More than 4.0	Over 30	Indented with difficulty by thumbnail.

ROCK TERMS

ROCK HARDNESS (FROM CORE SAMPLES)			ROCK BROKENNESS		
Descriptive Terms	Scratcher or Knife Effects	Hammer Effects	Descriptive Terms	Abbreviation	Spacing
Soft	Easily Gouged	Crushes when pressed with hammer	Very Broken	(V.Br.)	< 2"
Medium Soft	Can be Gouged	Breaks (one blow); crumbly edges	Broken	(Br.)	2"-6"
Medium Hard	Can be scratched	Breaks (one blow); sharp edges	Blocky	(Bl.)	6"-30"
Hard	Cannot be scratched	Breaks considerably (several blows); sharp edges	Massive	(M.)	30"-96"

LEGEND :

SOIL SAMPLES - TYPES	ROCK SAMPLES - TYPES	WATER LEVELS	
5-1/2" Split Barrel Sample	X-NX (Conventional) Core (2-1/8" O.D.)	12/96	
STP 3" O.D. Undisturbed Sample	ONQ (Winline) Core (2-1/8" O.D.)		▼ 12.5' Initial Level w/Date & Depth
O - Other Samples, Specify in Remarks	Z - Other Core Sizes, Specify in Remarks	12/96	
			▼ 12.5' Stabilized Level w/Date & Depth

5.2.1 USCS Classification

Soils are to be classified according to the Unified Soil Classification System (USCS). This method of classification is detailed in Figure 1 (Continued).
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 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 shall be divided into 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 shall be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges 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 shall be followed by a size designation such as "(1/4 inchΦ-1/2 inchΦ)" 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.

5.2.2 Color

Soil colors shall 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." Since 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 shall 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" shall be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

Soil Color Charts shall not be used unless specified by the project manager.

5.2.3 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 noncohesive (particles do not adhere well when compressed). Finer-grained soils (silts and clays) are cohesive (particles will adhere together when compressed).

The density of noncohesive, granular soils is classified according to standard penetration resistances obtained from split-barrel sampling performed according to the methods detailed in Standard Operating Procedures GH-1.3 and SA-1.3. Those designations are:

Designation	Standard Penetration Resistance (Blows per Foot)
Very loose	0 to 4
Loose	5 to 10
Medium dense	11 to 30
Dense	31 to 50
Very dense	Over 50

Standard penetration resistance is the number of blows required to drive a split-barrel sampler with a 2-inch outside diameter 12 inches into the material using a 140-pound hammer falling freely through 30 inches. The sampler is driven through an 18-inch sample interval, and the number of blows is recorded for each 6-inch increment. The density designation of granular soils is obtained by adding the number of blows required to penetrate the last 12 inches of each sample interval. It is important to note that if gravel or rock fragments are broken by the sampler or if rock fragments are lodged in the tip, the resulting blow count will be erroneously high, reflecting a higher density than actually exists. This shall be noted on the log and referenced to the sample number. 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 Figure 2.

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

The consistency of cohesive soils is determined either by blow counts, a pocket penetrometer (values listed in the table as Unconfined Compressive Strength), or by hand by determining the resistance to penetration by the thumb. The pocket penetrometer and thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample in the split-barrel sampler. The sample shall be broken in half and the thumb or penetrometer 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. Consistency shall not be determined solely by blow counts. One of the other methods shall be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in Figure 2.

5.2.4 Weight Percentages

In nature, soils are comprised 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

FIGURE 2

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

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.

5.2.5 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 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 drilling job.

Laboratory tests for water content shall be performed if the natural water content is important.

5.2.6 Stratification

Stratification can only be determined after the sample barrel is opened. The stratification or bedding thickness for soil and rock is depending on grain size and composition. The classification to be used for stratification description is shown in Figure 3.

5.2.7 Texture/Fabric/Bedding

The texture/fabric/bedding of the soil shall be described. Texture is described as the relative angularity of the particles: rounded, subrounded, subangular, and angular. Fabric shall be noted as to whether the particles are flat or bulky and whether there is a particular relation between particles (i.e., all the flat particles are parallel or there is some cementation). The bedding or structure shall also be noted (e.g., stratified, lensed, nonstratified, heterogeneous varved).

5.2.8 Summary of Soil Classification

In summary, soils shall 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
- Stratification
- Texture, fabric, bedding
- Other distinguishing features

FIGURE 3

BEDDING THICKNESS CLASSIFICATION

Thickness (metric)	Thickness (Approximate English Equivalent)	Classification
> 1.0 meter	> 3.3'	Massive
30 cm - 1 meter	1.0' - 3.3'	Thick Bedded
10 cm - 30 cm	4" - 1.0'	Medium Bedded
3 cm - 10 cm	1" - 4"	Thin Bedded
1 cm - 3 cm	2/5" - 1"	Very Thin Bedded
3 mm - 1 cm	1/8" - 2/5"	Laminated
1 mm - 3 mm	1/32" - 1/8"	Thinly Laminated
< 1 mm	<1/32"	Micro Laminated

(Weir, 1973 and Ingram, 1954)

5.3 Classification of Rocks

Rocks are grouped into three main divisions: sedimentary, igneous and metamorphic. Sedimentary rocks are by far the predominant type exposed at the earth's surface. The following basic names are applied to the types of rocks found in sedimentary sequences:

- Sandstone - Made up predominantly of granular materials ranging between 1/16 to 2 mm in diameter.
- Siltstone - Made up of granular materials less than 1/16 to 1/256 mm in diameter. Fractures irregularly. Medium thick to thick bedded.
- Claystone - Very fine-grained rock made up of clay and silt-size materials. Fractures irregularly. Very smooth to touch. Generally has irregularly spaced pitting on surface of drilled cores.
- Shale - A fissile very fine-grained rock. Fractures along bedding planes.
- Limestone - Rock made up predominantly of calcite ($CaCO_3$). Effervesces strongly upon the application of dilute hydrochloric acid.
- Coal - Rock consisting mainly of organic remains.
- Others - Numerous other sedimentary rock types are present in lesser amounts in the stratigraphic record. The local abundance of any of these rock types is dependent upon the depositional history of the area. Conglomerate, halite, gypsum, dolomite, anhydrite, lignite, etc. are some of the rock types found in lesser amounts.

In classifying a sedimentary rock the following hierarchy shall be noted:

- Rock type
- Color
- Bedding thickness
- Hardness
- Fracturing
- Weathering
- Other characteristics

5.3.1 Rock Type

As described above, there are numerous types of sedimentary rocks. In most cases, a rock will be a combination of several grain types, therefore, a modifier such as a sandy siltstone, or a silty sandstone can be used. The modifier indicates that a significant portion of the rock type is composed of the modifier. Other modifiers can include carbonaceous, calcareous, siliceous, etc.

Grain size is the basis for the classification of clastic sedimentary rocks. Figure 4 is the Udden-Wentworth classification that will be assigned to sedimentary rocks. The individual boundaries are slightly different than the USCS subdivision for soil classification. For field determination of grain sizes, a scale can be used for the coarse grained rocks. For example, the division between siltstone and claystone may not be measurable in the field. The boundary shall be determined by use of a hand lens. If the grains cannot be seen with the naked eye but are distinguishable with a hand lens, the rock is a siltstone. If the grains are not distinguishable with a hand lens, the rock is a claystone.

FIGURE 4

GRAIN SIZE CLASSIFICATION FOR ROCKS

Particle Name	Grain Size Diameter
Cobbles	> 64 mm
Pebbles	4 - 64 mm
Granules	2 - 4 mm
Very Coarse Sand	1 - 2 mm
Coarse Sand	0.5 - 1 mm
Medium Sand	0.25 - 0.5 mm
Fine Sand	0.125 - 0.25 mm
Very Fine Sand	0.0625 - 0.125 mm
Silt	0.0039 - 0.0625 mm

After Wentworth, 1922

5.3.2 Color

The color of a rock can be determined in a similar manner as for soil samples. Rock core samples shall be classified while wet, when possible, and air cored samples shall be scraped clean of cuttings prior to color classifications.

Rock color charts shall not be used unless specified by the Project Manager.

5.3.3 Bedding Thickness

The bedding thickness designations applied to soil classification (see Figure 3) will also be used for rock classification.

5.3.4 Hardness

The hardness of a rock is a function of the compaction, cementation, and mineralogical composition of the rock. A relative scale for sedimentary rock hardness is as follows:

- Soft - Weathered, considerable erosion of core, easily gouged by screwdriver, scratched by fingernail. Soft rock crushes or deforms under pressure of a pressed hammer. This term is always used for the hardness of the saprolite (decomposed rock which occupies the zone between the lowest soil horizon and firm bedrock).

- Medium soft - Slight erosion of core, slightly gouged by screwdriver, or breaks with crumbly edges from single hammer blow.

- Medium hard - No core erosion, easily scratched by screwdriver, or breaks with sharp edges from single hammer blow.

- Hard - Requires several hammer blows to break and has sharp conchoidal breaks. Cannot be scratched with screwdriver.

Note the difference in usage here of the works "scratch" and "gouge." A scratch shall be considered a slight depression in the rock (do not mistake the scraping off of rock flour from drilling with a scratch in the rock itself), while a gouge is much deeper.

5.3.5 Fracturing

The degree of fracturing or brokenness of a rock is described by measuring the fractures or joint spacing. After eliminating drilling breaks, the average spacing is calculated and the fracturing is described by the following terms:

- Very broken (V. BR.) - Less than 2-inch spacing between fractures
- Broken (BR.) - 2-inch to 1-foot spacing between fractures
- Blocky (BL.) - 1- to 3-foot spacing between fractures
- Massive (M.) - 3 to 10-foot spacing between fractures

The structural integrity of the rock can be approximated by calculating the Rock Quality Designation (RQD) of cores recovered. The RQD is determined by adding the total lengths of all pieces exceeding 4 inches and dividing by the total length of the coring run, to obtain a percentage.

Method of Calculating RQD
(After Deere, 1964)

$$RQD\ \% = r/l \times 100$$

r = Total length of all pieces of the lithologic unit being measured, which are greater than 4 inches length, and have resulted from natural breaks. Natural breaks include slickensides, joints, compaction slicks, bedding plane partings (not caused by drilling), friable zones, etc.

l = Total length of the coring run.

5.3.6 Weathering

The degree of weathering is a significant parameter that is important in determining weathering profiles and is also useful in engineering designs. The following terms can be applied to distinguish the degree of weathering:

- Fresh - Rock shows little or no weathering effect. Fractures or joints have little or no staining and rock has a bright appearance.
- Slight - Rock has some staining which may penetrate several centimeters into the rock. Clay filling of joints may occur. Feldspar grains may show some alteration.
- Moderate - Most of the rock, with exception of quartz grains, is stained. Rock is weakened due to weathering and can be easily broken with hammer.
- Severe - All rock including quartz grains is stained. Some of the rock is weathered to the extent of becoming a soil. Rock is very weak.

5.3.7 Other Characteristics

The following items shall be included in the rock description:

- Description of contact between two rock units. These can be sharp or gradational.
- Stratification (parallel, cross stratified).
- Description of any filled cavities or vugs.
- Cementation (calcareous, siliceous, hematitic).
- Description of any joints or open fractures.
- Observation of the presence of fossils.
- Notation of joints with depth, approximate angle to horizontal, any mineral filling or coating, and degree of weathering.

All information shown on the boring logs shall be neat to the point where it can be reproduced on a copy machine for report presentation. The data shall be kept current to provide control of the drilling program and to indicate various areas requiring special consideration and sampling.

5.3.8 Additional Terms Used in the Description of Rock

The following terms are used to further identify rocks:

- Seam - Thin (12 inches or less), probably continuous layer.
- Some - Indicates significant (15 to 40 percent) amounts of the accessory material. For example, rock composed of seams of sandstone (70 percent) and shale (30 percent) would be "sandstone -- some shale seams."
- Few - Indicates insignificant (0 to 15 percent) amounts of the accessory material. For example, rock composed of seam of sandstone (90 percent) and shale (10 percent) would be "sandstone -- few shale seams."
- Interbedded - Used to indicate thin or very thin alternating seams of material occurring in approximately equal amounts. For example, rock composed of thin alternating seams of sandstone (50 percent) and shale (50 percent) would be "interbedded sandstone and shale."
- Interlayered - Used to indicate thick alternating seams of material occurring in approximately equal amounts.

The preceding sections describe the classification of sedimentary rocks. The following are some basic names that are applied to igneous rocks:

- Basalt - A fine-grained extrusive rock composed primarily of calcic plagioclase and pyroxene.
- Rhyolite - A fine-grained volcanic rock containing abundant quartz and orthoclase. The fine-grained equivalent of a granite.
- Granite - A coarse-grained plutonic rock consisting essentially of alkali feldspar and quartz.
- Diorite - A coarse-grained plutonic rock consisting essentially of sodic plagioclase and hornblende.
- Gabbro - A coarse-grained plutonic rock consisting of calcic plagioclase and clinopyroxene. Loosely used for any coarse-grained dark igneous rock.

The following are some basic names that are applied to metamorphic rocks:

- Slate - A very fine-grained foliated rock possessing a well developed slaty cleavage. Contains predominantly chlorite, mica, quartz, and sericite.
- Phyllite - A fine-grained foliated rock that splits into thin flaky sheets with a silky sheen on cleavage surface.
- Schist - A medium to coarse-grained foliated rock with subparallel arrangement of the micaceous minerals which dominate its composition.
- Gneiss - A coarse-grained foliated rock with bands rich in granular and platy minerals.
- Quartzite - A fine- to coarse-grained nonfoliated rock breaking across grains, consisting essentially of quartz sand with silica cement.

5.4 Abbreviations

Abbreviations may be used in the description of a rock or soil. However, they shall be kept at a minimum. Following are some of the abbreviations that may be used:

C	-	Coarse	Lt	-	Light	Yl	-	Yellow
Med	-	Medium	BR	-	Broken	Or	-	Orange
F	-	Fine	BL	-	Blocky	SS	-	Sandstone
V	-	Very	M	-	Massive	Sh	-	Shale
Sl	-	Slight	Br	-	Brown	LS	-	Limestone
Occ	-	Occasional	Bl	-	Black	Fgr	-	Fine-grained
Tr	-	Trace						

5.5 Boring Logs and Documentation

This section describes in more detail the procedures to be used in completing boring logs in the field. Information obtained from the preceding sections shall be used to complete the logs. A sample boring log has been provided as Figure 5.

The field geologist/engineer shall use this example as a guide in completing each boring log. Each boring log shall be fully described by the geologist/engineer as the boring is being drilled. Every sheet contains space for 25 feet of log. Information regarding classification details is provided either on the back of the boring log or on a separate sheet, for field use.

5.5.1 Soil Classification

- Identify site name, boring number, job number, etc. Elevations and water level data to be entered when surveyed data is available.

- Enter sample number (from SPT) under appropriate column. Enter depth sample was taken from (1 block = 1 foot). Fractional footages, i.e., change of lithology at 13.7 feet, shall be lined off at the proportional location between the 13- and 14-foot marks. Enter blow counts (Standard Penetration Resistance) diagonally (as shown). Standard penetration resistance is covered in Section 5.2.3.

- Determine sample recovery/sample length as shown. Measure the total length of sample recovered from the split-spoon sampler, including material in the drive shoe. Do not include cuttings or wash material that may be in the upper portion of the sample tube.

- Indicate any change in lithology by drawing a line at the appropriate depth. For example, if clayey silt was encountered from 0 to 5.5 feet and shale from 5.5 to 6.0 feet, a line shall be drawn at this increment. This information is helpful in the construction of cross-sections. As an alternative, symbols may be used to identify each change in lithology.

- The density of granular soils is obtained by adding the number of blows for the last two increments. Refer to Density of Granular Soils Chart on back of log sheet. For consistency of cohesive soils refer also to the back of log sheet - Consistency of Cohesive Soils. Enter this information under the appropriate column. Refer to Section 5.2.3.

FIGURE 5
COMPLETED BORING LOG (EXAMPLE)

BORING LOG

Page 1 of 1

PROJECT NAME: NSB- SITE
PROJECT NUMBER: 9594
DRILLING COMPANY: SOILTEST CO.
DRILLING RIG: CME-55
BORING NUMBER: SB/MW 1
DATE: 3/8/96
GEOLOGIST: SJ CONTI
DRILLER: R. ROCK

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	Soil Density/ Consistency or Rock Hardness	Color	Material Classification	USCS *	Remarks	PID/FID Reading (ppm) Sample	Sampler BZ	Borehole**	Driller BZ**
S-1 @ 0800	0.0	7/6	1.5/2.0		M DENSE	BRN TO BLK	SILTY SAND - SOME	SM	MOIST SL. ORG. ODOR	5	0	0	0
	2.0	9/10					ROCK FR: TR BRICKS (FILL)		FILL TO 4'±				
	4.0			4.0									
S-2 @ 0810	6.0	5/7	2.0/2.0		M DENSE	BRN	SILTY SAND - TR FINE GRAVEL	SM	MOIST - W ODOR NAT. MATL. TOOK SAMPLE SB01-0406 FOR ANALYSIS	10	0	-	-
	•	9/8		7' ▽									
	8.0			8.0									
S-3 @ 0820	10.0	6/8	1.9/2.0		DENSE	TAN BRN	FINE TO COARSE SAND TR. F. GRAVEL	SW	WET HIT WATER: 7'±	0	0	0	0
	12.0	17/16		12.0									
S-4 @ 0830	14.0	7/6 5/8	1.6/2.0		STIFF	GRAY	SILTY CLAY	CL	MOIST → WET	0	.5	-	-
	15.0			15.0					AUGER REF @ 15'				
9/5	①		4.0/5.0	16	M HARD	BRN	SILTSTONE	VBR	WEATHERED LO ✱ JNTS @ 15.5 WATER STAINS @ 16.5, 17.1, 17.5	0	0	0	0
				19'					LOSING SOME				
	20.0				HARD	GRAY	SANDSTONE - SOME SILTSTONE	BR	DRILL H2O @ 17'± SET TEMP 6" CAS TO 15.5				
4.0/5.0	②		5.0/5.0						SET 2"Φ PVC SCREEN 16-25	0	0	0	0
									SAND 14-25				
	25.0			25					PELLETS 12-14				

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.

• 1-20Z 1-80Z

Drilling Area Background (ppm): 0

Remarks: CME-55 R16, 4¼" ID HSA - 9" OD ±
2" SPLIT SPOONS - 140 LB HAMMER - 30" DROP
NX CORE IN BEDROCK RUN ① = 25 min, RUN ② = 15 min

Converted to Well: Yes ✓ No _____ Well I.D. #: MW-1

- Enter color of the material in the appropriate column.

- Describe material using the USCS. Limit this column for sample description only. The predominant material is described last. If the primary soil is silt but has fines (clay) - use clayey silt. Limit soil descriptors to the following:

  - Trace: 0 - 10 percent
  - Some: 11 - 30 percent
  - And/Or: 31 - 50 percent

- Also indicate under Material Classification if the material is fill or natural soils. Indicate roots, organic material, etc.

- Enter USCS symbol - use chart on back of boring log as a guide. If the soils fall into one of two basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example ML/CL or SM/SP.

- The following information shall be entered under the "Remarks" column and shall include, but is not limited by, the following:

  - Moisture - estimate moisture content using the following terms - dry, moist, wet and saturated. These terms are determined by the individual. Whatever method is used to determine moisture, be consistent throughout the log.

  - Angularity - describe angularity of coarse grained particles using the terms angular, subangular, subrounded, or rounded. Refer to ASTM D 2488 or Earth Manual for criteria for these terms.

  - Particle shape - flat, elongated, or flat and elongated.

  - Maximum particle size or dimension.

  - Water level observations.

  - Reaction with HCl - none, weak, or strong.

- Additional comments:

  - Indicate presence of mica, caving of hole, when water was encountered, difficulty in drilling, loss or gain of water.

  - Indicate odor and Photoionization Detector (PID) or Flame Ionization Detector (FID) reading if applicable.

  - Indicate any change in lithology by drawing a line through the lithology change column and indicate the depth. This will help when cross-sections are subsequently constructed.

  - At the bottom of the page indicate type of rig, drilling method, hammer size and drop, and any other useful information (i.e., borehole size, casing set, changes in drilling method).

- Vertical lines shall be drawn (as shown in Figure 5) in columns 6 to 8 from the bottom of each sample to the top of the next sample to indicate consistency of material from sample to sample, if the material is consistent. Horizontal lines shall be drawn if there is a change in lithology, then vertical lines drawn to that point.

- Indicate screened interval of well, as needed, in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.2 Rock Classification

- Indicate depth at which coring began by drawing a line at the appropriate depth. Indicate core run depths by drawing coring run lines (as shown) under the first and fourth columns on the log sheet. Indicate RQD, core run number, RQD percent, and core recovery under the appropriate columns.

- Indicate lithology change by drawing a line at the appropriate depth as explained in Section 5.5.1.

- Rock hardness is entered under designated column using terms as described on the back of the log or as explained earlier in this section.

- Enter color as determined while the core sample is wet; if the sample is cored by air, the core shall be scraped clean prior to describing color.

- Enter rock type based on sedimentary, igneous or metamorphic. For sedimentary rocks use terms as described in Section 5.3. Again, be consistent in classification. Use modifiers and additional terms as needed. For igneous and metamorphic rock types use terms as described in Sections 5.3.8.

- Enter brokenness of rock or degree of fracturing under the appropriate column using symbols VBR, BR, BL, or M as explained in Section 5.3.5 and as noted on the back of the Boring Log.

- The following information shall be entered under the remarks column. Items shall include but are not limited to the following:

  - Indicate depths of joints, fractures and breaks and also approximate to horizontal angle (such as high, low), i.e., 70° angle from horizontal, high angle.
  - Indicate calcareous zones, description of any cavities or vugs.
  - Indicate any loss or gain of drill water.
  - Indicate drop of drill tools or change in color of drill water.

- Remarks at the bottom of Boring Log shall include:

  - Type and size of core obtained.
  - Depth casing was set.
  - Type of rig used.

- As a final check the boring log shall include the following:

  - Vertical lines shall be drawn as explained for soil classification to indicate consistency of bedrock material.

  - If applicable, indicate screened interval in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.3 Classification of Soil and Rock from Drill Cuttings

The previous sections describe procedures for classifying soil and rock samples when cores are obtained. However, some drilling methods (air/mud rotary) may require classification and borehole logging based on identifying drill cuttings removed from the borehole. Such cuttings provide only general information on subsurface lithology. Some procedures that shall be followed when logging cuttings are:

- Obtain cutting samples at approximately 5-foot intervals, sieve the cuttings (if mud rotary drilling) to obtain a cleaner sample, place the sample into a small sample bottle or "zip lock" bag for future reference, and label the jar or bag (i.e. hole number, depth, date, etc.). Cuttings shall be closely examined to determine general lithology.
- Note any change in color of drilling fluid or cuttings, to estimate changes in lithology.
- Note drop or chattering of drilling tools or a change in the rate of drilling, to determine fracture locations or lithologic changes.
- Observe loss or gain of drilling fluids or air (if air rotary methods are used), to identify potential fracture zones.
- Record this and any other useful information onto the boring log as provided in Figure 1.

This logging provides a general description of subsurface lithology and adequate information can be obtained through careful observation of the drilling process. It is recommended that split-barrel and rock core sampling methods be used at selected boring locations during the field investigation to provide detailed information to supplement the less detailed data generated through borings drilled using air/mud rotary methods.

5.6 Review

Upon completion of the borings logs, copies shall be made and reviewed. Items to be reviewed include:

- Checking for consistency of all logs.
- Checking for conformance to the guideline.
- Checking to see that all information is entered in their respective columns and spaces.

6.0 REFERENCES

Unified Soil Classification System (USCS).

ASTM D2488, 1985.

Earth Manual, U.S. Department of the Interior, 1974.

7.0 RECORDS

Originals of the boring logs shall be retained in the project files.

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1.0 PURPOSE

The purpose of this procedure is to provide a basic understanding of developing contour maps and the approaches used to identify and quantify the direction and rate of groundwater flow and contaminant plume movement.

2.0 SCOPE

This procedure provides only a general overview of the field techniques, mathematical and physical relationships and data handling procedures used for determining groundwater flow direction and rate. The references identified herein can provide a more complete explanation of particular methods cited, as well as a more comprehensive discussion on the interpretation of hydrogeologic data.

3.0 GLOSSARY

Aquifer - A geologic formation capable of transmitting usable quantities of groundwater to a well or other discharge point.

Aquitard - A geologic formation which retards the flow of groundwater due to its low permeability.

Confined Aquifer - An aquifer that is overlain and underlain by zones of lower permeability (aquitards). If the aquifer is "artesian," the potentiometric head of the aquifer at a given point is higher than the top of the zone comprising the aquifer at that point.

Equipotential Line - A line connecting points of equal elevation of the water table or potentiometric surface. Equipotential lines on the water table are also called water table contour lines.

Flow Line - A flow line indicates the direction of groundwater movement within the saturated zone. Flow lines are drawn perpendicular to equipotential lines.

Flow Net - A diagram of groundwater flow showing flow lines and equipotential lines.

Hydraulic Conductivity (K) - A quantitative measure of the ability of porous material to transmit water. Volume of water that will flow through a unit cross sectional area of porous material per unit time under a head gradient. Hydraulic conductivity is dependent upon properties of the medium and fluid.

Hydraulic Gradient (i) - The rate of change of hydraulic head per unit distance of flow at a given point and in the downgradient direction.

Hydraulic Head - The height to which water will rise inside a well casing, equal to the elevation head plus the pressure head. In a well screened across the water table, hydraulic head equals the elevation head, as the pressure head equals 0. In wells screened below the water table in an unconfined aquifer or screened at any interval within a confined aquifer, the head is the sum of the elevation of the aquifer (the elevation head) and the fluid pressure of the water confined in the aquifer (the pressure head).

Potentiometric (piezometric) Surface - A hypothetical surface that coincides with the static level of the water in an aquifer (i.e., the maximum elevation to which water will rise in a well or piezometer penetrating the aquifer). The term "potentiometric surface" is usually applied to confined aquifers, although the water table is the potentiometric surface of an unconfined aquifer.

Unconfined Aquifer - An aquifer in which the water table forms the upper boundary.

Water Table - The surface in the groundwater system at which the fluid pressure is equal to atmospheric pressure (i.e., the net pressure head is zero) and below which all strata are saturated with water.

4.0 RESPONSIBILITIES

Project Hydrogeologist - The project hydrogeologist has overall responsibility for obtaining water level measurements and developing groundwater contour maps. The hydrogeologist (with the concurrence of the Project Manager) shall specify the reference point from which water levels are measured (usually a specific point on the upper edge of the inner well casing), the number of data points needed and which wells shall be used for a contour map, and how many complete sets of water levels are required to adequately define groundwater flow directions (e.g., if there are seasonal variations).

Field Personnel - All supporting field personnel must have a basic familiarity with the equipment and procedures involved in obtaining water levels, and must be aware of any project-specific requirements.

5.0 PROCEDURES

5.1 Potentiometric Surface Mapping

5.1.1 Selection of Wells

All wells used to prepare a flow net in a plan or map view should represent the same hydrogeologic unit, be it aquifer or aquitard. All water level measurements used shall be collected on the same day, preferably within 2-3 hours. This is especially important when working in an area where groundwater levels are tidally influenced or influenced by pumping.

The recorded water levels, monitoring-well construction data, site geology, and topographic setting must be reviewed to ascertain that the wells are completed in the same hydrogeologic unit and to determine if strong vertical hydraulic gradients may be present. Such conditions will be manifested by a pronounced correlation between well depth and water level, or by a difference in water level between two wells located near each other but set to different depths or having different screen lengths. Professional judgment of the hydrogeologist is important in this determination. If vertical gradients are significant, the data to be used must be limited vertically, and only wells finished in a chosen vertical zone of the hydrogeologic unit can be used.

At least three wells must be used to provide an estimation of the direction of groundwater flow; information from many more wells are needed to provide an accurate contour map. Generally, shallow systems require data from more wells than deep systems for accurate contour mapping. Potentiometric surface mapping for shallow flow systems also requires water level measurements from nearby surface water bodies.

5.1.2 Water Level Measurements

After selection of the wells to be used for mapping, the next step in determining the direction of groundwater flow is to obtain water level elevations from the selected points. In addition, any other readily available wells/surface water bodies should be measured to ensure that sufficient data are available for interpretation purposes.

Elevations are obtained from measurements of the depth to water in a monitoring well or piezometer taken from the top of the well casing (see SOP GH-1.2) and then referencing the elevation of the casing to a chosen and consistent datum point, usually mean sea level. Subtracting the depth to water from the casing elevation provides the elevation of the potentiometric surface. Elevations of points and areas of groundwater discharge or recharge such as springs, seeps, streams, rivers, and lakes also need to be determined, typically through staff gauge measurements. Comparison of these elevations, which represent hydraulic heads, will reveal the direction of flow because groundwater flows from areas of high head to areas of low head.

5.1.3 Construction of Equipotential Lines

Graphical methods available for depicting the flow of groundwater include the use of equipotential lines and flow lines to construct potentiometric surface maps and vertical flow nets. If the hydrogeologic system consists of a water table aquifer and one or more confined aquifers, separate contour maps should be prepared for each aquifer system. Water table maps should be developed using water level measurements obtained from monitoring wells screened at the unsaturated-saturated interface. Water level measurements collected from monitoring wells screened in the deeper portions of an unconfined aquifer should generally be contoured as a separate potentiometric surface map. Surface water discharge or recharge features are contoured in the water table system. Vertical flow nets should be constructed using a cross section aligned parallel to the direction of groundwater flow. All water level measurements along this cross section, both deep and shallow, are used in developing equipotential lines and flow lines for the flow net.

To construct equipotential lines, water level elevations in the chosen wells are plotted on a site map. Other hydrogeologic features associated with the zone of interest -- such as seeps, wetlands, and surface-water bodies -- should also be plotted along with their elevations.

The data should then be contoured, using mathematically valid and generally accepted techniques. Linear interpolation is the most commonly used technique. However, quadratic interpolation or any technique of trend-surface analysis or data smoothing is acceptable. Computer-generated contour maps may be useful rough mapping of large data sets; however, final, detailed mapping must always be performed by hand by an experienced hydrogeologist. Contour lines shall be drawn as smooth, continuous lines which never cross one another.

Inspect the contour map, noting known features, such as pumping wells and site topography. The contour lines must be adjusted utilizing the professional judgment of the hydrogeologist in accordance with these features. Closed contours should be avoided unless a known groundwater sink (i.e., pumping well) or mound exists. Groundwater mounding is common under landfills and lagoons; if the data imply this, the feature must be evident in the contour plot.

5.1.4 Determination of Groundwater-Flow Direction

Flow lines shall be drawn so that they are perpendicular to equipotential lines. Flow lines will begin at high head elevations and end at low head elevations. Closed highs will be the source of additional flow lines. Closed depressions (i.e., wells) will be the termination of some flow lines. Care must be used in areas with significant vertical gradients to avoid erroneous conclusions concerning gradients and flow directions.

5.2 Groundwater Flow Considerations

Groundwater movement is an integral part of the hydrologic cycle. Recharge to the shallow groundwater environment generally occurs by infiltration of precipitation through an upper unsaturated soil zone. Movement is downward under the force of gravity until the water reaches the saturated zone of the water table aquifer. Once water is part of the water table aquifer, movement is controlled by differences in hydraulic head, with movement from areas of high head to areas of low head. Areas of low head include natural discharge areas such as springs, lakes, rivers, and, ultimately, the ocean. These features can be considered as outcrops of the water table. Points of low head also are created by pumping wells.

Local head differences and consequent vertical flow patterns within an aquifer can be detected by well clusters. A well cluster consists of several adjacent wells, generally installed within a few feet of each other, and screened at different depths. Variations in water levels in these closely spaced wells indicates the vertical component of groundwater flow within an aquifer, provided that the wells are all screened within the same aquifer.

The number, location, and extent of geologic units and their properties with regard to aquifer or aquitard characteristics must be understood to properly interpret water level data gathered from the monitoring system. This firm understanding of the hydrogeologic system must be developed through a program of borings, wells, and interpretation of subsurface geology. The adequacy of the positions and depths of borings/wells used to define relevant subsurface hydrogeologic conditions must also be assessed. The location of surface water discharge or recharge points must be considered. Surface water features influence the system, as flow is most likely toward them (if they are discharge points) or away from them (if they are recharge points). Man-made discharge or recharge features such as pumping or injection wells, ditches, and trenches can also affect the flow of groundwater.

5.3 Determination of Flow Rate

Darcy's Law states that the quantity of water flowing through a geologic material is dependent upon the permeability of the material, the hydraulic gradient, and the cross sectional area through which the water flows. This relation is expressed in the equation:

$$Q = KiA$$

where:

Q	=	volume of water flowing through the cross sectional area of the formation (L^3/T).
K	=	hydraulic conductivity (L/T).
i	=	hydraulic gradient (L/L, i.e., dimensionless).
A	=	cross sectional area of formation being considered (L^2).

The relation is similar to one used in stream flow measurements where:

$$Q = VA$$

where:

Q	=	discharge from the cross sectional area of a stream or pipe (L^3/T).
V	=	average velocity of flowing water (L/T).
A	=	cross sectional area through which water flows (L^2).

The velocity of water movement in a geologic formation depends on the specific formation properties and the head differences across the formation. This relation is defined in the equation:

$$V = \frac{Ki}{n}$$

where:

V	=	average linear velocity of groundwater through the formation (L/T)
K	=	hydraulic conductivity (L/T)
i	=	hydraulic gradient (dimensionless)
n	=	porosity (expressed as a fraction).

Values of porosity for several geologic materials are given in Attachment A. More accurate and specific values of porosity can be obtained by laboratory analysis of a formation sample or from an unconfined aquifer pumping test.

Hydraulic conductivity is related to the permeability of the formation and depends on the size and interconnection of the pore spaces. In isotropic and homogeneous formations, the hydraulic conductivity will be the same vertically and horizontally. In anisotropic formations, horizontal and vertical conductivity can be markedly different and the vertical hydraulic conductivity can be up to several orders of magnitude lower than the horizontal hydraulic conductivity. Typically, most formations are anisotropic with horizontal hydraulic conductivities at least several times as high as the vertical hydraulic conductivities.

Generally, hydraulic conductivities are high for sands, gravels, and limestone containing large solution cavities and low for silts, clays, and tightly fractured rock. Attachment A gives values of hydraulic conductivity for several geologic materials. More accurate values can be obtained during field testing of aquifers or from laboratory measurements on undisturbed cores. Results from field testing usually provide higher (and more representative) hydraulic conductivities than laboratory testing because full-scale field testing includes the effects of the formational macrostructure (i.e., secondary permeability due to jointing or fractures) which is not reflected in the testing of a small sample in the laboratory.

The hydraulic gradient, i, is determined from field measurements of hydraulic head obtained from water level measuring points. Do <u>not</u> measure gradient from well to well; measure across equipotential lines that are drawn based on the well (and other) data. Once a potentiometric surface map has been generated using the hydraulic head data, the hydraulic gradient can be calculated using the following formula:

$$i = \frac{dh}{dl}$$

where:

dh	=	change in head (L)
dl	=	distance between equipotential lines (L)

The hydraulic gradient along any flow line can be calculated from a potentiometric surface map by dividing the change in head by the length of the flow line, typically beginning and ending at equipotential lines. The longer the distance over which the head change is measured, the more representative the gradient is of overall conditions.

When chemical solutes are traveling in groundwater, as in cases of groundwater contamination, the calculated groundwater velocity may predict migration rates in excess of what is actually observed. The difference in chemical versus water velocities may be due to attenuation or biodegradation of the chemical species in the aquifer. Attenuation is most often caused by adsorption of the chemical contaminant onto the formation grains or matrix. The result is that the chemical does not appear at the downgradient sampling point as quickly as the velocity calculation predicts. An equation to correct for this attenuation is:

$$V_c = V_w / (1 + K_d P_b / n)$$

where:

V_c	=	velocity of the chemical solute flow (L/T)
V_w	=	velocity of groundwater flow (L/T)
P_b	=	formation mass bulk density (M/L^3)
n	=	formation porosity (expressed as a fraction)
K_d	=	distribution coefficient = (L^3/M)

The K_d is equal to the mass of solute per unit mass of solid phase divided by the concentration of solute in solution. The term in the denominator is known as the retardation factor.

Density and/or viscosity differences between water and contaminants can also cause velocity determination errors. Light hydrocarbons such as gasoline are less dense than water and consequently float on the water table. These contaminants can migrate along the water table surface at rates faster or slower than the rate of groundwater movement, depending on specific conditions, and may also volatilize into unsaturated soil pore spaces. Oils are more viscous than water and will typically migrate more slowly due to the viscosity difference. Contaminants denser than water such as heavy hydrocarbons (e.g., coal tar) or chlorinated compounds (e.g., TCE, PCE) tend to sink to the bottom of an aquifer if present in concentrations exceeding their solubility limit (these chemicals are often referred to as dense, nonaqueous phase liquids, or DNAPLs if present as a separate-phase liquid). Here, the contamination may move at faster or slower rates than the overlying groundwater or may actually move in a direction opposite to that of the groundwater, depending on the geologic characteristics of the aquifer base and direction of dip of the underlying aquitard.

Other factors involving the physicochemical interaction between the chemical and the groundwater, such as dilution (mixing contaminated water or chemicals with additional quantities of groundwater) and dispersion (molecular diffusion of the chemical throughout the groundwater regime), can also affect the observed rates of travel of contaminants in groundwater. In addition to such physicochemical characteristics, all of the aquifer and aquitard properties and groundwater flow characteristics described above must be known so that adequate and accurate estimations of the extent and rate of groundwater contaminant migration can be developed.

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ATTACHMENT A

GENERALIZED POROSITY AND HYDRAULIC CONDUCTIVITY VALUES FOR GEOLOGIC MATERIALS

Material	Porosity Range (%)	Hydraulic Conductivity Range	
		cm/sec	ft/day
Gravel	30-40	10^{-1} to 10^{-2}	280 to 2.8×10^{5}
Coarse sand (clean)	30-40	10^{-1} to 1	280 to 2,800
Medium sand (clean)	35-45	10^{-2} to 10^{-1}	28 to 280
Fine sand (clean)	40-50	5×10^{-4} to 10^{-2}	1.4 to 28
Silty sand	25-40	10^{-5} to 10^{-2}	0.03 to 280
Glacial Till	Variable	10^{-10} to 10^{-4}	3×10^{-7} to 0.3
Unweathered Clay/Shale	45-55 (clay)	10^{-7} to 10^{-4}	3×10^{-4} to 0.3 (horizontal)
		10^{-10} to 10^{-6}	3×10^{-7} to 3×10^{-3} (vertical)
Karst Limestone	---	10^{-4} to 10^{-1}	0.3 to 2,800
Fractured Igneous/Metamorphic Rocks	---	10^{-6} to 10^{-1}	3×10^{-3} to 280
Sandstone	5-30	10^{-8} to 10^{-4}	3×10^{-5} to 0.3

Source: References 1 and 2

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1.0 PURPOSE

This procedure provides general guidance and information pertaining to proper monitoring well design, installation, and development.

2.0 SCOPE

This procedure is applicable to the construction of monitoring wells. The methods described herein may be modified by project-specific requirements for monitoring well construction. In addition, many regulatory agencies have specific regulations pertaining to monitoring well construction and permitting. These requirements must be determined during the project planning phases of the investigation, and any required permits must be obtained before field work begins. Innovative monitoring well installation techniques, which typically are not used, will be discussed only generally in this procedure.

3.0 GLOSSARY

Monitoring Well - A well which is screened, cased, and sealed which is capable of providing a groundwater level and groundwater sample representative of the zone being monitored. Some monitoring wells may be constructed as open boreholes.

Piezometer - A pipe or tube inserted into the water bearing zone, typically open to water flow at the bottom and to the atmosphere at the top, and used to measure water level elevations. Piezometers may range in size from 1/2-inch-diameter plastic tubes to well points or monitoring wells.

Potentiometric Surface - The surface representative of the level to which water will rise in a well cased to the screened aquifer.

Well Point (Drive Point) - A screened or perforated tube (Typically 1-1/4 or 2 inches in diameter) with a solid, conical, hardened point at one end, which is attached to a riser pipe and driven into the ground with a sledge hammer, drop weight, or mechanical vibrator. Well points may be used for groundwater injection and recovery, as piezometers (i.e., to measure water levels) or to provide groundwater samples for water quality data.

4.0 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 driller may also be responsible for obtaining, in advance, any required permits for monitoring well installation and construction.

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

5.0 PROCEDURES

5.1 Equipment/Items Needed

Below is a list of items that may be needed when installing a monitoring well or piezometer:

- Health and safety equipment (hard hats, safety glasses, etc.) as required by the Site Safety Officer.
- Well drilling and installation equipment with associated materials (typically supplied by the driller).
- Hydrogeologic equipment (weighted engineer's tape, water level indicator, retractable engineers rule, electronic calculator, clipboard, mirror and flashlight - for observing downhole activities, paint and ink marker for marking monitoring wells, sample jars, well installation forms, and a field notebook).
- Drive point installation tools (sledge hammer, drop hammer, or mechanical vibrator; tripod, pipe wrenches, drive points, riser pipe, and end caps).

5.2 Well Design

The objectives and intended use for each monitoring well must be clearly defined before the monitoring system is designed. Within the monitoring system, different monitoring wells may serve different purposes and, therefore, require different types of construction. During all phases of the well design, attention must be given to clearly documenting the basis for design decisions, the details of well construction, and the materials used. The objectives for installing the monitoring wells may include:

- Determining groundwater flow directions and velocities.
- Sampling or monitoring for trace contaminants.
- Determining aquifer characteristics (e.g., hydraulic conductivity).

Siting of monitoring wells shall be performed after a preliminary estimation of the groundwater flow direction. In most cases, groundwater flow directions and potential well locations can be determined by an experienced hydrogeologist through the review of geologic data and the site terrain. In addition, data from production wells or other monitoring wells in the area may be used to determine the groundwater flow direction. If these methods cannot be used, piezometers, which are relatively inexpensive to install, may have to be installed in a preliminary investigative phase to determine groundwater flow direction.

5.2.1 Well Depth, Diameter, and Monitored Interval

The well depth, diameter, and monitored interval must be tailored to the specific monitoring needs of each investigation. Specification of these items generally depends on the purpose of the monitoring system and the characteristics of the hydrogeologic system being monitored. Wells of different depth, diameter, and monitored interval can be employed in the same groundwater monitoring system. For instance, varying the monitored interval in several wells, at the same location (cluster wells) can help to determine the vertical gradient and the depths at which contaminants are present. Conversely, a fully penetrating well is usually not used to quantify or vertically locate a contaminant plume, since groundwater samples collected in wells that are screened over the full thickness of the water-bearing zone will be representative of average conditions across the entire monitored interval. However, fully penetrating wells can be used to establish the existence of contamination in the water-bearing zone. The well diameter desired depends upon the hydraulic characteristics of the water-bearing zone, sampling requirements, drilling method and cost.

The decision concerning the monitored interval and well depth is based on the following (and possibly other) information:

- The vertical location of the contaminant source in relation to the water-bearing zone.
- The depth, thickness and uniformity of the water-bearing zone.
- The anticipated depth, thickness, and characteristics (e.g., density relative to water) of the contaminant plume.
- Fluctuation in groundwater levels (due to pumping, tidal influences, or natural recharge/discharge events).
- The presence and location of contaminants encountered during drilling.
- Whether the purpose of the installation is for determining existence or non-existence of contamination or if a particular stratigraphic zone is being investigated.
- The analysis of borehole geophysical logs.

In most situations where groundwater flow lines are horizontal, depending on the purpose of the well and the site conditions, monitored intervals are 20 feet or less. Shorter screen lengths (5 feet or less) are usually required where flow lines are not horizontal, (i.e., if the wells are to be used for accurate measurement of the potentiometric head at a specific point).

Many factors influence the diameter of a monitoring well. The diameter of the monitoring well depends on the application. In determining well diameter, the following needs must be considered:

- Adequate water volume for sampling.
- Drilling methodology.
- Type of sampling device to be used.
- Costs.

Standard monitoring well diameters are 2, 4, 6, or 8 inches. Drive points are typically 1-1/4 or 2 inches in diameter. For monitoring programs which require screened monitoring wells, either a 2-inch or 4-inch-diameter well is preferred. Typically, well diameters greater than 4 inches are used in monitoring programs in which open-hole bedrock monitoring wells are used. With smaller diameter wells, the volume of stagnant water in the well is minimized, and well construction costs are reduced; however, the sampling devices that can be used are limited.

In specifying well diameter, sampling requirements must be considered (up to a total of 4 gallons of water may be required for a single sample to account for full organic and inorganic analyses, and split samples), particularly if the monitored formation is known to be a low-yielding formation. The unit volume of water contained within a monitoring well is dependent on the well diameter as follows:

Casing Inside Diameter (Inch)	Standing Water Length to Obtain 1 Gallon Water (Feet)
2	6.13
4	1.53
6	0.68

If a well recharges quickly after purging, then well diameter may not be an important factor regarding sample volume requirements.

Pumping tests for determining aquifer characteristics may require larger diameter wells (for installation of high capacity pumps); however, in small-diameter wells in-situ permeability tests can be performed during drilling or after well installation is completed.

5.2.2 Riser Pipe and Screen Materials

Well materials are specified by diameter, type of material, and thickness of pipe. Well screens require an additional specification of slot size. Thickness of pipe is referred to as "Schedule" for polyvinyl chloride (PVC) casing and is usually Schedule 40 (thinner wall) or 80 (thicker wall). Steel pipe thickness is often referred to as "Strength". Standard Strength is usually adequate for monitoring well purposes. With larger diameter pipe, the wall thickness must be greater to maintain adequate strength. The required thickness is also dependent on the method of installation; risers for drive points require greater strength than wells installed inside drilled borings.

The selection of well screen and riser materials depends on the method of drilling, the type of subsurface materials the well penetrates, the type of contamination expected, and natural water quality and depth. Cost and the level of accuracy required are also important. The materials generally available are Teflon, stainless steel, PVC galvanized steel, and carbon steel. Each has advantages and limitations (see Attachment A of this guideline for an extensive presentation on this topic). The two most commonly used materials are PVC and stainless steel. Properties of these two materials are compared in Attachment B. Stainless steel is a good choice where trace metals or organic sampling is required; however, costs are high. Teflon materials are extremely expensive, but are relatively inert and provide the least opportunity for water contamination due to well materials. PVC has many advantages, including low cost, excellent availability, light weight, ease of manipulation, and widespread acceptance. The crushing strength of PVC may limit the depth of installation, but the use of Schedule 80 materials may overcome some of the problems associated with depth. However, the smaller inside diameter of Schedule 80 pipe may be an important factor when considering the size of bailers or pumps required for sampling or testing. Due to this problem, the minimum well pipe size recommended for Schedule 80 wells is 4-inch I.D.

Screens and risers may have to be decontaminated before use because oil-based preservatives and oil used during thread cutting and screen manufacturing may contaminate samples. Metal pipe may corrode and release metal ions or chemically react with organic constituents, but this is considered a minor issue. Galvanized steel is not recommended where samples may be collected for metals analyses, as zinc and cadmium levels in groundwater samples may become elevated from leaching of the zinc coating.

Threaded, flush-joint casing is most often preferred for monitoring well applications. PVC, Teflon, and steel can all be obtained with threaded joints. Welded-joint steel casing is also acceptable. Glued PVC may release organic contaminants into the well, and therefore, should not be used if the well is to be sampled for organic constituents.

When the water-bearing zone is in consolidated bedrock, such as limestone or fractured granite, a well screen is often not necessary (the well is simply an open hole in bedrock). Unconsolidated materials, such as sands, clay, and silts require a screen. A screen slot size of 0.010 or 0.020 inch is generally used when a screen is necessary, and the annular borehole space around the screened interval is artificially packed with an appropriately sized sand, selected based on formation grain size. The slot size controls the quantity of water entering the well and prevents entry of natural materials or sand pack. The screen shall pass no more than 10 percent of the pack material, or in-situ aquifer material. The site geologist shall specify the combination of screen slot size and sand pack which will be compatible with the water-bearing zone, to maximize groundwater inflow and minimize head losses and movement of fines into the wells. For example, as a standard procedure, a Morie No. 1 or No. 10 to No. 20 U.S. Standard Sieve size filter pack is typically appropriate for a 0.020-inch slot screen; however, a No. 20 to No. 40 U.S. Standard Sieve size filter pack is typically appropriate for a 0.010-inch slot screen.

5.2.3 Annular Materials

Materials placed in the annular space between the borehole and riser pipe and screen include a sand pack when necessary, a bentonite seal, and cement-bentonite grout. The sand pack is usually a medium- to coarse-grained poorly graded, silica sand and should relate to the grain size of the aquifer sediments. The quantity of sand placed in the annular space is dependent upon the length of the screened interval, but should always extend at least 1 foot above the top of the screen. At least 1 to 3 feet of bentonite pellets or equivalent shall be placed above the sand pack. Cement-bentonite grout (or equivalent) is then placed to extent from the top of the bentonite pellets to the ground surface.

On occasion, and with the concurrence of the involved regulatory agencies, monitoring wells may be packed naturally (i.e., no artificial sand pack installed). In this case, the natural formation material is allowed to collapse around the well screen after the well is installed. This method has been used where the formation material itself is a relatively uniform grain size, or when artificial sand packing is not possible due to borehole collapse.

Bentonite expands by absorbing water and provides a seal between the screened interval and the overlying portion of the annular space and formation. Cement-bentonite grout is placed on top of the bentonite pellets, extending to the surface. The grout effectively seals the remaining borehole annulus and eliminates the possibility for surface infiltration reaching the screened interval. Grouting also replaces material removed during drilling and prevents hole collapse and subsidence around the well. A tremie pipe should be used to introduce grout from the bottom upward, to prevent bridging, and to provide a better seal. In shallow boreholes that don't collapse, it may be more practical to pour the grout from the surface without a tremie pipe.

Grout is a general term which has several different connotations. For all practical purposes within the monitoring well installation industry, grout refers to the solidified material which is installed and occupies the annular space above the bentonite pellet seal. Grout, most of the time, is made up of one or two assemblages of material, (e.g., cement and/or bentonite). A cement-bentonite grout, which is the most common type of grout used in monitoring well completions, normally is a mixture of cement, bentonite, and water at a ratio of one 90-pound bag of Portland Type I cement, plus 3 to 5 pounds of granular or flake-type bentonite, and 6-7 gallons of water. A neat cement consists of one ninety-pound bag of Portland Type I cement and 6-7 gallons of water. A bentonite slurry (bentonite and water mixed to a thick but pumpable mixture) is sometimes used instead of grout for deep well installations where placement of bentonite pellets is difficult. Bentonite chips are also occasionally used for annular backfill in place of grout.

In certain cases, the borehole may be drilled to a depth greater than the anticipated well installation depth. For these cases, the well shall be backfilled to the desired depth with bentonite pellets/chips or sand. A short (1- to 2-foot) section of capped riser pipe sump is sometimes installed immediately below the screen, as a silt reservoir, when significant post-development silting is anticipated. This will ensure that the entire screen surface remains unobstructed.

5.2.4 Protective Casing

When the well is completed and grouted to the surface, a protective steel casing is typically placed over the top of the well. This casing generally has a hinged cap and can be locked to prevent vandalism. The protective casing has a larger diameter than the well and is set into the wet cement grout over the well upon completion. In addition, one hole is drilled just above the cement collar through the protective casing which acts as a weep hole for the flow of water which may enter the annulus during well development, purging, or sampling.

A protective casing which is level with the ground surface (flush-mounted) is used in roadway or parking lot applications where the top of a monitoring well must be below the pavement. The top of the riser pipe is placed 4 to 5 inches below the pavement, and a locking protective casing is cemented in place to 3 inches below the pavement. A large diameter, manhole-type protective collar is set into the wet cement around the well with the top set level with or slightly above the pavement. An appropriately-sized id is placed over the protective sleeve. The cement should be slightly mounded to direct pooled water away from the well head.

5.3 Monitoring Well Installation

Pertinent data regarding monitoring well installation shall be recorded on log sheets as depicted and discussed in SOP SA-6.3. Attachments to this referenced SOP illustrate terms and physical construction of various types of monitoring wells.

5.3.1 Monitoring Wells in Unconsolidated Sediments

After the borehole is drilled to the desired depth, well installation can begin. The procedure for well installation will partially be dictated by the stability of the formation in which the well is being placed. If the borehole collapses immediately after the drilling tools are withdrawn, then a temporary casing must be installed and well installation will proceed through the center of the temporary casing, and continue as the temporary casing is withdrawn from the borehole. In the case of hollow-stem auger drilling, the augers will act to stabilize the borehole during well installation.

Before the screen and riser pipe are lowered into the borehole, all pipe and screen sections should be measured with an engineer's rule to ensure proper placement. When measuring sections, the threads on one end of the pipe or screen must be excluded while measuring, since the pipe and screen sections are screwed flush together.

After the screen and riser pipe are lowered through the temporary casing, the sand pack can be installed. A weighted tape measure must be used during the installation procedure to carefully monitor installation progress. The sand is slowly poured into the annulus between the riser pipe and temporary casing, as the casing is withdrawn. Sand should always be kept within the temporary casing during withdrawal in order to ensure an adequate sand pack. However, if too much sand is within the temporary casing (greater than 1 foot above the bottom of the casing) bridging between the temporary casing and riser pipe may occur. Centralizers may be used at the geologist's discretion, one above and one below the screen, to assure enough annular space for sand pack placement.

After the sand pack is installed to the desired depth (at least 1 foot above the top of the screen), then the bentonite pellet seal (or equivalent), can be installed in the same manner as the sand pack. At least 1 to 3 feet of bentonite pellets should be installed above the sand pack. Pellets should be added slowly and their fall monitored closely to ensure that bridging does not occur.

The cement-bentonite grout is then mixed and tremied into the annulus as the temporary casing or augers are withdrawn. Finally, the protective casing can be installed as detailed in Section 5.2.4.

5.3.2 Confining Layer Monitoring Wells

When drilling and installing a well in a confined aquifer, proper well installation techniques must be applied to avoid cross contamination between aquifers. Under most conditions, this can be accomplished by installing double-cased wells. This is accomplished by drilling a large-diameter boring through the upper aquifer, 1 to 5 feet into the underlying confining layer, and setting and pressure grouting or tremie grouting a large-diameter casing into the confining layer. The grout material must fill the space between the native material and the outer casing. A smaller diameter boring is then continued through the confining layer for

installation of the monitoring well as detailed for overburden monitoring wells. Sufficient time (determined by the field geologist), must be allowed for setting of the grout prior to drilling through the confined layer.

5.3.3 Bedrock Monitoring Wells

When installing bedrock monitoring wells, a large diameter boring is drilled through the overburden and approximately 5 –10 feet into bedrock. A casing (typically steel) is installed and either pressure grouted or tremie grouted in place. After the grout has cured, a smaller diameter boring is continued into bedrock to the desired depth. If the boring does not collapse, the well can be left open, and a screen is not necessary. If the boring collapses, then a screen is required and can be installed as detailed for overburden monitoring wells. If a screen is to be used, then the casing which is installed through the overburden and into the bedrock does not require grouting and can be removed when the final well installation is completed.

5.3.4 Drive Points

Drive points can be installed with either a sledge hammer, drop hammer, or a mechanical vibrator. The screen section is threaded and tightened onto the riser pipe with pipe wrenches. The drive point is simply pounded into the subsurface to the desired depth. If a heavy drop hammer is used, then a tripod and pulley setup is required to lift the hammer. Drive points typically cannot be manually driven to depths exceeding 10 feet.

Direct push sampling/monitoring point installation methods, using a direct push rig or drilling rig, are described in SOP SA-2.5.

5.3.5 Innovative Monitoring Well Installation Techniques

Certain innovative sampling devices have proven advantageous. These devices are essentially screened samplers installed in a borehole with only small-diameter tubes extending to the surface. This reduces drilling costs, decreases the volume of stagnant water, and provides a sampling system that minimizes cross-contamination from sampling equipment. Four manufacturers of these samplers include Timco Manufacturing Company, Inc., of Prairie du Sac, Wisconsin, BARCAD Systems, Inc., of Concord, Massachusetts, Westbay Instruments Ltd. of Vancouver, British Columbia, Canada and the University of Waterloo at Waterloo, Ontario, Canada.. Each manufacturer offers various construction materials.

5.4 Well Development Methods

The purpose of well development is to stabilize and increase the permeability of the gravel pack around the well screen, and to restore the permeability of the formation which may have been reduced by drilling operations. Wells are typically developed until all fine material and drilling water is removed from the well. Sequential measurements of pH, conductivity, turbidity, and temperature taken during development may yield information (stabilized values) regarding whether sufficient development has been performed. The selection of the well development method shall be made by the field geologist and is based on the drilling methods, well construction and installation details, and the characteristics of the formation that the well is screened in. The primary methods of well development are summarized below. A more detailed discussion may be found in Driscoll (1986).

5.4.1 Overpumping and Backwashing

Wells may be developed by alternatively drawing the water level down at a high rate (by pumping or bailing) and then reversing the flow direction (backwashing) so that water is passing from the well into the formation. This back and forth movement of water through the well screen and gravel pack serves to

remove fines from the formation immediately adjacent to the well, while preventing bridging (wedging) of sand grains. Backwashing can be accomplished by several methods, including pouring water into the well and then bailing, starting and stopping a pump intermittently to change water levels, or forcing water into the well under pressure through a water-tight fitting ("rawhiding"). Care should be taken when backwashing not to apply too much pressure, which could damage or destroy the well screen.

5.4.2 Surging with a Surge Plunger

A surge plunger (also called a surge block) is approximately the same diameter as the well casing and is aggressively moved up and down within the well 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. There are two basic types of surge plungers; solid and valved surge plungers. In formations with low yields, a valved surge plunger may be preferred, as solid plungers tend to force water out of the well at a greater rate than it will flow back in. Valved plungers are designed to produce a greater inflow than outflow of water during surging.

5.4.3 Compressed Air

Compressed air can be used to develop a well by either of two methods: backwashing or surging. Backwashing is done by forcing water out through the screens, using increasing air pressure inside a sealed well, then releasing the pressurized air to allow the water to flow back into the well. Care should be taken when using this method so that the water level does not drop below the top of the screen, thus introducing air into the formation and reducing well yield. Surging, or the "open well" method, consists of alternately releasing large volumes of air suddenly into an open well below the water level to produce a strong surge by virtue of the resistance of water head, friction, and inertia. Pumping of the well is subsequently done using the air lift method.

5.4.4 High Velocity Jetting

In the high velocity jetting method, water is forced at high velocities from a plunger-type device and through the well screen to loosen fine particles from the sand pack and surrounding formation. The jetting tool is slowly rotated and raised and lowered along the length of the well screen to develop the entire screened area. Jetting using a hose lowered into the well may also be effective. The fines washed into the screen during this process can then be bailed or pumped from the well.

6.0 RECORDS

A critical part of monitoring well installation is recording of all significant details and events in the site logbook or field notebook. The geologist must record the exact depths of significant hydrogeological features, screen placement, gravel pack placement, and bentonite placement.

A Monitoring Well Sheet (see Attachments to SOP SA-6.3) shall be completed, ensuring the uniform recording of data for each installation and rapid identification of missing information. Well depth, length, materials of construction, length and openings of screen, length and type of riser, and depth and type of all backfill materials shall be recorded. Additional information shall include location, installation date, problems encountered, water levels before and after well installation, cross-reference to the geologic boring log, and methods used during the installation and development process. Documentation is very important to prevent problems involving questionable sample validity. Somewhat different information will need to be recorded, depending on whether the well is completed in overburden (single- or double-cased), as a cased well in bedrock, or as an open hole in bedrock.

The quantities of sand, bentonite, and grout placed in the well are also important. The geologist shall calculate the annular space volume and have an idea of the quantity of material needed to fill the annular

space. Volumes of backfill significantly higher than the calculated volume may indicate a problem such as a large cavity, while a smaller backfill volume may indicate a cave-in or bridging of the backfill materials. Any problems with rig operation or down-time shall be recorded and may affect the driller's final fee.

7.0 REFERENCES

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ATTACHMENT A

RELATIVE COMPATIBILITY OF RIGID WELL CASING MATERIAL (PERCENT)

Potentially-Deteriorating Substance	Type of Casing Material						
	PVC 1	Galvanized Steel	Carbon Steel	Lo-carbon Steel	Stainless Steel 304	Stainless Steel 316	Teflon*
Buffered Weak Acid	100	56	51	59	97	100	100
Weak Acid	98	59	43	47	96	100	100
Mineral Acid/ High Solids Content	100	48	57	60	80	82	100
Aqueous/Organic Mixtures	64	69	73	73	98	100	100
Percent Overall Rating	91	58	56	59	93	96	100

Preliminary Ranking of Rigid Materials:

1 Teflon®
2 Stainless Steel 316
3. Stainless Steel 304
4 PVC 1
5 Lo-Carbon Steel
6 Galvanized Steel
7 Carbon Steel

* Trademark of DuPont

RELATIVE COMPATIBILITY OF SEMI-RIGID OR ELASTOMERIC MATERIALS (PERCENT)

Potentially-Deteriorating Substance	Type of Casing Material								
	PVC Flexible	PP	PE Conv.	PE Linear	PMM	Viton®*	Silicone	Neoprene	Teflon®*
Buffered Weak Acid	97	97	100	97	90	92	87	85	100
Weak Acid	92	90	94	96	78	78	75	75	100
Mineral Acid/ High Solids Content	100	100	100	100	95	100	78	82	100
Aqueous/Organic Mixtures	62	71	40	60	49	78	49	44	100
Percent Overall Rating	88	90	84	88	78	87	72	72	100

Preliminary Ranking of Semi-Rigid or Elastomeric Materials:

1 Teflon®
2 Polypropylene (PP)
3. PVC Flexible/PE Linear
4 Viton®
5 PE Conventional
6 Plexiglas/Lucite (PMM)
7 Silicone/Neoprene

* Trademark of DuPont

Source: Barcelona et al., 1983

ATTACHMENT B

COMPARISON OF STAINLESS STEEL AND PVC FOR MONITORING WELL CONSTRUCTION

Characteristic	Stainless Steel	PVC
Strength	Use in deep wells to prevent compression and closing of screen/riser.	Use when shear and compressive strength are not critical.
Weight	Relatively heavier.	Light-weight; floats in water.
Cost	Relatively expensive.	Relatively inexpensive.
Corrosivity	Deteriorates more rapidly in corrosive water.	Non-corrosive -- may deteriorate in presence of ketones, aromatics, alkyl sulfides, or some chlorinated hydrocarbons.
Ease of Use	Difficult to adjust size or length in the field.	Easy to handle and work with in the field.
Preparation for Use	Should be steam cleaned if organics will be subsequently sampled.	Never use glue fittings -- pipes should be threaded or pressure fitted. Should be steam cleaned when used for monitoring wells.
Interaction with Contaminants*	May sorb organic or inorganic substances when oxidized.	May sorb or release organic substances.

* See also Attachment A.

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION I

LOW STRESS (low flow) PURGING AND SAMPLING PROCEDURE FOR THE COLLECTION OF GROUND WATER SAMPLES FROM MONITORING WELLS

July 30, 1996
Revision 2

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION I

LOW STRESS (low flow) PURGING AND SAMPLING PROCEDURE FOR THE COLLECTION OF GROUND WATER SAMPLES FROM MONITORING WELLS

I. SCOPE & APPLICATION

This standard operating procedure (SOP) provides a general framework for collecting ground water samples that are indicative of mobile organic and inorganic loads at ambient flow conditions (both the dissolved fraction and the fraction associated with mobile particulates). The SOP emphasizes the need to minimize stress by low water-level drawdowns, and low pumping rates (usually less than 1 liter/min) in order to collect samples with minimal alterations to water chemistry. This SOP is aimed primarily at sampling monitoring wells that can accept a submersible pump and have a screen, or open interval length of 10 feet or less (this is the most common situation). However, this procedure is flexible and can be used in a variety of well construction and ground-water yield situations. Samples thus obtained are suitable for analyses of ground water contaminants (volatile and semi-volatile organic analytes, pesticides, PCBs, metals and other inorganics), or other naturally occurring analytes.

This procedure does not address the collection of samples from wells containing light or dense non-aqueous phase liquids (LNAPLs and DNAPLs). For this the reader may wish to check: Cohen, R.M. and J.W. Mercer, 1993, DNAPL Site Evaluation; C.K. Smoley (CRC Press), Boca Raton, Florida and U.S. Environmental Protection Agency, 1992, RCRA Ground-Water Monitoring: Draft Technical Guidance; Washington, DC (EPA/530-R-93-001).

The screen, or open interval of the monitoring well should be optimally located (both laterally and vertically) to intercept existing contaminant plume(s) or along flowpaths of potential contaminant releases. It is presumed that the analytes of interest move (or potentially move) primarily through the more permeable zones within the screen, or open interval.

Use of trademark names does not imply endorsement by U.S.EPA but is intended only to assist in identification of a specific type of device.

Proper well construction and development cannot be overemphasized, since the use of installation techniques that are appropriate to the hydrogeologic setting often prevents "problem well" situations from occurring. It is also recommended that as part of development or redevelopment the well should be tested to determine the appropriate pumping rate to obtain stabilization of field indicator parameters with minimal drawdown in shortest amount of time. With this information field crews can then conduct purging and sampling in a more expeditious manner.

The mid-point of the saturated screen length (which should not exceed 10 feet) is used by convention as the location of the pump intake. However, significant chemical or permeability contrast(s) within the screen may require additional field work to determine the optimum vertical location(s) for the intake, and appropriate pumping rate(s) for purging and sampling more localized target zone(s). Primary flow zones (high(er) permealability and/or high(er) chemical concentrations) should be identified in wells with screen lengths longer than 10 feet, or in wells with open boreholes in bedrock. Targeting these zones for water sampling will help insure that the low stress procedure will not underestimate contaminant concentrations. The Sampling and Analysis Plan must provide clear instructions on how the pump intake depth(s) will be selected, and reason(s) for the depth(s) selected.

Stabilization of indicator field parameters is used to indicate that conditions are suitable for sampling to begin. Achievement of turbidity levels of less than 5 NTU and stable drawdowns of less than 0.3 feet, while desirable, are not mandatory. Sample collection may still take place provided the remaining criteria in this procedure are met. If after 4 hours of purging indicator field parameters have not stabilized, one of 3 optional courses of action may be taken: a) continue purging until stabilization is achieved, b) discontinue purging, do not collect any samples, and record in log book that stabilization could not be achieved (documentation must describe attempts to achieve stabilization) c) discontinue purging, collect samples and provide full explanation of attempts to achieve stabilization (note: there is a risk that the analytical data obtained, especially metals and strongly hydrophobic organic analytes, may not meet the sampling objectives).

Changes to this SOP should be proposed and discussed when the site Sampling and Analysis Plan is submitted for approval. Subsequent requests for modifications of an approved plan must include adequate technical justification for proposed changes. All changes and modifications must be approved before implementation in field.

II.EQUIPMENT

A. Extraction device

Adjustable rate, submersible pumps are preferred (for example, centrifugal or bladder pump constructed of stainless steel or

Teflon).

Adjustable rate, peristaltic pumps (suction) may be used with caution. Note that EPA guidance states: "Suction pumps are not recommended because they may cause degassing, pH modification, and loss of volatile compounds" (EPA/540/P-87/001, 1987, page 8.5-11).

The use of inertial pumps is discouraged. These devices frequently cause greater disturbance during purging and sampling and are less easily controlled than the pumps listed above. This can lead to sampling results that are adversely affected by purging and sampling operations, and a higher degree of data variability.

B. Tubing

Teflon or Teflon lined polyethylene tubing are preferred when sampling is to include VOCs, SVOCs, pesticides, PCBs and inorganics.

PVC, polypropylene or polyethylene tubing may be used when collecting samples for inorganics analyses. However, these materials should be used with caution when sampling for organics. If these materials are used, the equipment blank (which includes the tubing) data must show that these materials do not add contaminants to the sample.

Stainless steel tubing may be used when sampling for VOCs, SVOCs, pesticides, and PCBs. However, it should be used with caution when sampling for metals.

The use of 1/4 inch or 3/8 inch (inner diameter) tubing is preferred. This will help ensure the tubing remains liquid filled when operating at very low pumping rates.

Pharmaceutical grade (Pharmed) tubing should be used for the section around the rotor head of a peristaltic pump, to minimize gaseous diffusion.

C. Water level measuring device(s), capable of measuring to 0.01 foot accuracy (electronic “tape”, pressure transducer). Recording pressure transducers, mounted above the pump, are especially helpful in tracking water levels during pumping operations, but their use must include check measurements with a water level “tape” at the start and end of each record.

D. Flow measurement supplies (e.g., graduated cylinder and stop watch).

E. Interface probe, if needed.

F. Power source (generator, nitrogen tank, etc.). If a gasoline generator is used, it must be located downwind and at least 30 feet from the well so that the exhaust fumes do not contaminate the samples.

G. Indicator field parameter monitoring instruments - pH, Eh, dissolved oxygen (DO), turbidity, specific conductance, and temperature. Use of a flow-through-cell is required when measuring all listed parameters, except turbidity. Standards to perform field calibration of instruments. Analytical methods are listed in 40 CFR 136, 40 CFR 141, and SW-846. For Eh measurements, follow manufacturer's instructions.

H. Decontamination supplies (for example, non-phosphate detergent, distilled/deionized water, isopropyl alcohol, etc.).

I. Logbook(s), and other forms (for example, well purging forms).

J. Sample Bottles.

K. Sample preservation supplies (as required by the analytical methods).

L. Sample tags or labels.

M. Well construction data, location map, field data from last sampling event.

N. Well keys.

O. Site specific Sample and Analysis Plan/Quality Assurance Project Plan.

P. PID or FID instrument (if appropriate) to detect VOCs for health and safety purposes, and provide qualitative field evaluations.

III.PRELIMINARY SITE ACTIVITIES

Check well for security damage or evidence of tampering, record pertinent observations.

Lay out sheet of clean polyethylene for monitoring and sampling equipment.

Remove well cap and immediately measure VOCs at the rim of the well with a PID or FID instrument and record the reading in the field logbook.

If the well casing does not have a reference point (usually a V-cut or indelible mark in the well casing), make one. Describe its location and record the date of the mark in the logbook.

A synoptic water level measurement round should be performed (in the shortest possible time) before any purging and sampling activities begin. It is recommended that water level depth (to 0.01 ft.) and

total well depth (to 0.1 ft.) be measured the day before, in order to allow for re-settlement of any particulates in the water column. If measurement of total well depth is not made the day before, it should not be measured until after sampling of the well is complete. All measurements must be taken from the established referenced point. Care should be taken to minimize water column disturbance.

Check newly constructed wells for the presence of LNAPLs or DNAPLs before the initial sampling round. If none are encountered, subsequent check measurements with an interface probe are usually not needed unless analytical data or field head space information signal a worsening situation. Note: procedures for collection of LNAPL and DNAPL samples are not addressed in this SOP.

IV.PURGING AND SAMPLING PROCEDURE

Sampling wells in order of increasing chemical concentrations (known or anticipated) is preferred.

1. Install Pump

Lower pump, safety cable, tubing and electrical lines slowly (to minimize disturbance) into the well to the midpoint of the zone to be sampled. The Sampling and Analysis Plan should specify the sampling depth, or provide criteria for selection of intake depth for each well (see Section I). If possible keep the pump intake at least two feet above the bottom of the well, to minimize mobilization of particulates present in the bottom of the well. Collection of turbid free water samples may be especially difficult if there is two feet or less of standing water in the well.

2. Measure Water Level

Before starting pump, measure water level. If recording pressure transducer is used-initialize starting condition.

3. Purge Well

3a. Initial Low Stress Sampling Event

Start the pump at its lowest speed setting and slowly increase the speed until discharge occurs. Check water level. Adjust pump speed until there is little or no water level drawdown (less than 0.3 feet). If the minimal drawdown that can be achieved exceeds 0.3 feet but remains stable, continue purging until indicator field parameters stabilize.

Monitor and record water level and pumping rate every three to five minutes (or as appropriate) during purging. Record any pumping rate adjustments (both time and flow rate). Pumping rates should, as needed, be reduced to the minimum capabilities of the pump (for example, 0.1 - 0.4 l/min) to ensure stabilization of indicator

parameters. Adjustments are best made in the first fifteen minutes of pumping in order to help minimize purging time. During pump start-up, drawdown may exceed the 0.3 feet target and then "recover" as pump flow adjustments are made. Purge volume calculations should utilize stabilized drawdown value, not the initial drawdown. Do not allow the water level to fall to the intake level (if the static water level is above the well screen, avoid lowering the water level into the screen). The final purge volume must be greater than the stabilized drawdown volume plus the extraction tubing volume.

Wells with low recharge rates may require the use of special pumps capable of attaining very low pumping rates (bladder, peristaltic), and/or the use of dedicated equipment. If the recharge rate of the well is lower than extraction rate capabilities of currently manufactured pumps and the well is essentially dewatered during purging, then the well should be sampled as soon as the water level has recovered sufficiently to collect the appropriate volume needed for all anticipated samples (ideally the intake should not be moved during this recovery period). Samples may then be collected even though the indicator field parameters have not stabilized.

3b. Subsequent Low Stress Sampling Events

After synoptic water level measurement round, check intake depth and drawdown information from previous sampling event(s) for each well. Duplicate, to the extent practicable, the intake depth and extraction rate (use final pump dial setting information) from previous event(s). Perform purging operations as above.

4. Monitor Indicator Field Parameters

During well purging, monitor indicator field parameters (turbidity, temperature, specific conductance, pH, Eh, DO) every three to five minutes (or less frequently, if appropriate). Note: during the early phase of purging emphasis should be put on minimizing and stabilizing pumping stress, and recording those adjustments. Purging is considered complete and sampling may begin when all the above indicator field parameters have stabilized. Stabilization is considered to be achieved when three consecutive readings, taken at three (3) to five (5) minute intervals, are within the following limits:

turbidity (10% for values greater than 1 NTU),
DO (10%),
specific conductance (3%),
temperature (3%),
pH (± 0.1 unit),
ORP/Eh (± 10 millivolts).

All measurements, except turbidity, must be obtained using a flow-through-cell. Transparent flow-through-cells are preferred, because they allow field personnel to watch for particulate build-up within the cell. This build-up may affect indicator field parameter values

measured within the cell and may also cause an underestimation of turbidity values measured after the cell. If the cell needs to be cleaned during purging operations, continue pumping and disconnect cell for cleaning, then reconnect after cleaning and continue monitoring activities.

The flow-through-cell must be designed in a way that prevents air bubble entrapment in the cell. When the pump is turned off or cycling on/off (when using a bladder pump), water in the cell must not drain out. Monitoring probes must be submerged in water at all times. If two flow-through-cells are used in series, the one containing the dissolved oxygen probe should come first (this parameter is most susceptible to error if air leaks into the system).

5. Collect Water Samples

Water samples for laboratory analyses must be collected before water has passed through the flow-through-cell (use a by-pass assembly or disconnect cell to obtain sample).

VOC samples should be collected first and directly into pre-preserved sample containers. Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

During purging and sampling, the tubing should remain filled with water so as to minimize possible changes in water chemistry upon contact with the atmosphere. It is recommended that 1/4 inch or 3/8 inch (inside diameter) tubing be used to help insure that the sample tubing remains water filled. If the pump tubing is not completely filled to the sampling point, use one of the following procedures to collect samples: (1) add clamp, connector (Teflon or stainless steel) or valve to constrict sampling end of tubing; (2) insert small diameter Teflon tubing into water filled portion of pump tubing allowing the end to protrude beyond the end of the pump tubing, collect sample from small diameter tubing; (3) collect non-VOC samples first, then increase flow rate slightly until the water completely fills the tubing, collect sample and record new drawdown, flow rate and new indicator field parameter values.

Add preservative, as required by analytical methods, to samples immediately after they are collected if the sample containers are not pre-preserved. Check analytical methods (e.g. EPA SW-846, water supply, etc.) for additional information on preservation. Check pH for all samples requiring pH adjustment to assure proper pH value. For VOC samples, this will require that a test sample be collected during purging to determine the amount of preservative that needs to be added to the sample containers prior to sampling.

If determination of filtered metal concentrations is a sampling objective, collect filtered water samples using the same low flow procedures. The use of an in-line filter is required, and the filter

size (0.45 um is commonly used) should be based on the sampling objective. Pre-rinse the filter with approximately 25 - 50 ml of ground water prior to sample collection. Preserve filtered water sample immediately. Note: filtered water samples are not an acceptable substitute for unfiltered samples when the monitoring objective is to obtain chemical concentrations of total mobile contaminants in ground water for human health risk calculations.

Label each sample as collected. Samples requiring cooling (volatile organics, cyanide, etc.) will be placed into a cooler with ice or refrigerant for delivery to the laboratory. Metal samples after acidification to a pH less than 2 do not need to be cooled.

6. Post Sampling Activities

If recording pressure transducer is used, remeasure water level with tape.

After collection of the samples, the pump tubing may either be dedicated to the well for resampling (by hanging the tubing inside the well), decontaminated, or properly discarded.

Before securing the well, measure and record the well depth (to 0.1 ft.), if not measured the day before purging began. Note: measurement of total well depth is optional after the initial low stress sampling event. However, it is recommended if the well has a “silting” problem or if confirmation of well identity is needed.

Secure the well.

V. DECONTAMINATION

Decontaminate sampling equipment prior to use in the first well and following sampling of each subsequent well. Pumps will not be removed between purging and sampling operations. The pump and tubing (including support cable and electrical wires which are in contact with the well) will be decontaminated by one of the procedures listed below.

Procedure 1

The decontaminating solutions can be pumped from either buckets or short PVC casing sections through the pump or the pump can be disassembled and flushed with the decontaminating solutions. It is recommended that detergent and isopropyl alcohol be used sparingly in the decontamination process and water flushing steps be extended to ensure that any sediment trapped in the pump is removed. The pump exterior and electrical wires must be rinsed with the decontaminating solutions, as well. The procedure is as follows:

Flush the equipment/pump with potable water.

Flush with non-phosphate detergent solution. If the solution is recycled, the solution must be changed periodically.

Flush with potable or distilled/deionized water to remove all of the detergent solution. If the water is recycled, the water must be changed periodically.

Flush with isopropyl alcohol (pesticide grade). If equipment blank data from the previous sampling event show that the level of contaminants is insignificant, then this step may be skipped.

Flush with distilled/deionized water. The final water rinse must not be recycled.

Procedure 2

Steam clean the outside of the submersible pump.

Pump hot potable water from the steam cleaner through the inside of the pump. This can be accomplished by placing the pump inside a three or four inch diameter PVC pipe with end cap. Hot water from the steam cleaner jet will be directed inside the PVC pipe and the pump exterior will be cleaned. The hot water from the steam cleaner will then be pumped from the PVC pipe through the pump and collected into another container. Note: additives or solutions should not be added to the steam cleaner.

Pump non-phosphate detergent solution through the inside of the pump. If the solution is recycled, the solution must be changed periodically.

Pump potable water through the inside of the pump to remove all of the detergent solution. If the solution is recycled, the solution must be changed periodically.

Pump distilled/deionized water through the pump. The final water rinse must not be recycled.

VI. FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not compromised the quality of the ground water samples. All field quality control samples must be prepared the same as regular investigation samples with regard to sample volume, containers, and preservation. The following quality control samples shall be collected for each batch of samples (a batch may not exceed 20 samples). Trip blanks are required for the VOC samples at a frequency of one set per VOC sample cooler.

Field duplicate.

Matrix spike.

Matrix spike duplicate.

Equipment blank.

Trip blank (VOCs).

Temperature blank (one per sample cooler).

Equipment blank shall include the pump and the pump's tubing. If tubing is dedicated to the well, the equipment blank will only include the pump in subsequent sampling rounds.

Collect samples in order from wells with lowest contaminant concentration to highest concentration. Collect equipment blanks after sampling from contaminated wells and not after background wells.

Field duplicates are collected to determine precision of sampling procedure. For this procedure, collect duplicate for each analyte group in consecutive order (VOC original, VOC duplicate, SVOC original, SVOC duplicate, etc.).

If split samples are to be collected, collect split for each analyte group in consecutive order (VOC original, VOC split, etc.). Split sample should be as identical as possible to original sample.

All monitoring instrumentation shall be operated in accordance with EPA analytical methods and manufacturer's operating instructions. EPA analytical methods are listed in 40 CFR 136, 40 CFR 141, and SW-846 with exception of Eh, for which the manufacturer's instructions are to be followed. Instruments shall be calibrated at the beginning of each day. If a measurement falls outside the calibration range, the instrument should be re-calibrated so that all measurements fall within the calibration range. At the end of each day, check calibration to verify that instruments remained in calibration. Temperature measuring equipment, thermometers and thermistors, need not be calibrated to the above frequency. They should be checked for accuracy prior to field use according to EPA Methods and the manufacturer's instructions.

VII.FIELD LOGBOOK

A field log shall be kept to document all ground water field monitoring activities (see attached example matrix), and record all of the following:

Well identification.

Well depth, and measurement technique.

Static water level depth, date, time and measurement technique.

Presence and thickness of immiscible liquid (NAPL) layers and

detection method.

Pumping rate, drawdown, indicator parameters values, and clock time, at the appropriate time intervals; calculated or measured total volume pumped.

Well sampling sequence and time of each sample collection.

Types of sample bottles used and sample identification numbers.

Preservatives used.

Parameters requested for analysis.

Field observations during sampling event.

Name of sample collector(s).

Weather conditions.

QA/QC data for field instruments.

Any problems encountered should be highlighted.

Description of all sampling equipment used, including trade names, model number, diameters, material composition, etc.

VIII. DATA REPORT

Data reports are to include laboratory analytical results, QA/QC information, and whatever field logbook information is needed to allow for a full evaluation of data useability.

EXAMPLE (Minimum Requirements) Page____of____

Well PURGING-FIELD WATER QUALITY MEASUREMENTS FORM

Location (Site/Facility Name)_____ Depth to _____/_____ of screen
Well Number_____Date_____ (below MP) top bottom
Field Personnel_____ Pump Intake at (ft. below MP)_____
Sampling Organization_____ Purging Device; (pump type)_____
Identify MP_____

Clock Time 24 HR	Water Depth below MP ft	Pump Dial[1]	Purge Rate ml/min	Cum. Volume Purged liters	Temp. °C	Spec. Cond.[2] µS/cm	pH	ORP/ Eh[3] mv	DO mg/L	Turb-idity NTU	Comments

1. Pump dial setting (for example: hertz, cycles/min, etc).
2. µSiemens per cm(same as µmhos/cm)at 25 °C.
3. Oxidation reduction potential (stand in for Eh).

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1.0 PURPOSE

This Standard Operating Procedure (SOP) describes procedures and equipment commonly used for collecting environmental samples of surface water and aquatic sediment for either onsite examination and chemical testing or for offsite laboratory analysis.

2.0 SCOPE

The information presented in this document is applicable to all environmental sampling of surface waters (Section 5.3) and aquatic sediments (Section 5.5), except where the analyte(s) may interact with the sampling equipment. The collection of concentrated sludges or hazardous waste samples from disposal or process lagoons often requires methods, precautions, and equipment different from those described herein.

3.0 GLOSSARY

Analyte – Chemical or radiochemical material whose concentration, activity, or mass is measured.

Composite Sample – A sample representing a physical average of grab samples.

Environmental Sample – A quantity of material collected in support of an environmental investigation that does not require special handling or transport considerations as detailed in SOP SA-6.1.

Grab Sample – A portion of material collected to represent material or conditions present at a single unit of space and time.

Hazardous Waste Sample – A sample containing (or suspected to contain) concentrations of contaminants that are high enough to require special handling and/or transport considerations per SOP SA-6.1.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, initial sampling locations, and field procedures used in the collection of soil samples. The Project Manager also has the overall responsibility for seeing that all surface water and sediment sampling activities are properly conducted by appropriately trained personnel in accordance with applicable planning documents.

Field Operations Leader - This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that

custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is responsible for finalizing the locations for collection of surface water and sediment samples. The FOL is ultimately responsible for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan (HASP). This includes but is not be limited to performing air quality monitoring during sampling and boring and excavation activities, and ensuring that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO or SSO designee may also be required to advise the FOL on other safety-related matters regarding boring and sampling, such as mitigative measures to address potential hazards from hazardous objects or conditions.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP and other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, , container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

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

5.0 HEALTH AND SAFETY

Precautions to preserve the health and safety of field personnel implementing this SOP are distributed throughout. The following general hazards may also exist during field activities, and the means of avoiding them must be used to preserve the health and safety of field personnel:

Bridge/Boat Sampling – Potential hazards associated with this activity include:

- Traffic – one of the primary concerns as samplers move across a bridge because free space of travel is not often provided. Control measures should include:
  - When sampling from a bridge, if the samplers do not have at least 6 feet of free travel space or physical barriers separating them and the traffic patterns, the HASP will include a Traffic Control Plan.
  - The use of warning signs and high-visibility vests are required to warn oncoming traffic and to increase the visibility of sample personnel.
- Slips, trips, and falls from elevated surfaces are a primary concern. Fall protection shall be worn when or if samplers must lean over a rail to obtain sample material. A Fall Protection Competent

Person (in accordance with Occupational safety and Health Administration [OSHA] fall protection standards) must be assigned to ensure that fall protection is appropriately and effectively employed

- Water hazards/drowning – if someone enters the water from an elevated surface (such as a bridge or dock) and when sampling from a boat. To minimize this potential, personnel shall wear United states Coast Guard (USCG)-approved floatation devices, and the sampling crew must also have on hand a Type IV Throwable Personal Floatation Device with at least 90 feet of 3/8-inch rope. See Section 5.5.2 of this SOP.

- Within the HASP, provisions will also be provided concerning the requirement of a Safe Vessel Certification or the necessity to conduct a boat inspection prior to use. In addition, the HASP shall also specify requirements as to whether the operator must be certified as a commercial boat operator and whether members of the sampling team must have a state-specific safe boating certification.

Entering Water to Collect Samples – Several hazards are associated with this activity and can be mitigated as follows:

- Personnel must wear a USCG-approved Floatation Device (selected and identified in the HASP). The SSO shall ensure that the device selected is in acceptable condition and suitable for the individual using it. This includes consideration of the weight of the individual.

- Lifelines shall be employed from a point on the shore. This activity will always be conducted with a Buddy. See Section 6.5.2.

- Personnel shall carry a probe to monitor the bottom ahead of them for drop offs or other associated hazards.

- The person in the water shall exercise caution concerning the path traveled so that the lifeline does not become entangled in underwater obstructions such as logs, branches, stumps, etc., thereby restricting its effectiveness in extracting the person from the water.

- Personnel shall not enter waters on foot in situations where natural hazards including alligators, snakes, as well as sharks, gars, and other predators within inland waterways may exist.

- In all cases, working along and/or entering the water during high currents or flood conditions shall be prohibited.

- Personnel shall not enter bodies of water where known debris exists that could result in injuries from cuts and lacerations.

Sampling in marshes or tidal areas in some instances can be accomplished using an all-terrain vehicle (ATV). This is not the primary recommended approach because the vehicle may become disabled, or weather conditions or tidal changes could result in environmental damage as well as loss of the vehicle. The primary approach is recommended to be on foot where minimal disturbance would occur. The same precautions specified above with regard to sediment disturbance apply as well as the previously described safety concerns associated with natural hazards. The natural hazards include alligators, bees (nests in dead falls and tree trunks), snakes, etc. In addition, moving through and over this terrain is difficult and could result in muscle strain and slips, trips, and falls. Common sense dictates that the sampler selects the most open accessible route over moderate terrain. Move slowly and deliberately through challenging terrain to minimize falls. Mud boots or other supportive PPE should be considered and specified in the HASP to permit samplers to move over soft terrain with the least amount of effort. In these situations, it is also recommended, as the terrain allows, that supplies be loaded and transported in a sled over the soft ground.

Working in these areas, also recognize the following hazards and means of protection against them:

Insects are also a primary concern. These include mosquitoes, ticks, spiders, bees, ants, etc. The HASP will identify those particular to your area. Typical preventative measures include:

- Use insect repellant. Approval of various repellants should be approved by the Project Chemist or Project Manager.
- Wearing light-colored clothing to control heat load due to excessive temperatures. In addition, it makes it easier to detect crawling insects on your clothing.
- Taping pants to boots to deny access. Again, this is recommended to control access to the skin by crawling insects. Consultation with the Project Health and Safety Officer SSO/Health and Safety Manager is recommended under extreme heat loads because this will create conditions of heat stress.
- Performing a body check to remove insects. The quicker you remove ticks, the less likely they will become attached and transfer bacteria to your bloodstream. Have your Buddy check areas inaccessible to yourself. This includes areas such as the upper back and between shoulder blades where it is difficult for you to examine and even more difficult for you to remove.

Safety Reminder

If you are allergic to bee or ant stings, it is especially critical that you carry your doctor-recommended antidote with you in these remote sampling locations due to the extended time required to extract incapacitated individuals as well as the effort required to extract them. In these scenarios, instruct your Buddy in the proper administration of the antidote. In all cases, if you have received a sting, administer the antidote regardless of the immediate reaction, evacuate, and seek medical attention as necessary. The FOL and/or SSO will determine when and if you may return to the field based on the extent of the immune response and hazards or potential hazards identified in these locations. To the FOL and SSO, this is a serious decision you have to make as to whether to take someone vulnerable to these hazards into a remote location where you may not be able to carry them out. Consider it wisely.

Poisonous Plants – To minimize the potential of encountering poisonous plants in the field, at least one member of the field team needs to have basic knowledge of what these plants look like so that they can be recognized, pointed out to other field personnel, and avoided if at all possible. If the field team cannot avoid contact and must move through an area where these plants exist, the level of personal protective equipment (PPE) shall include Tyvek coveralls and enhanced decontamination procedures for the removal of oils from the tooling and/or equipment.

Temperature-Related Stress – Excessively cold temperatures may result in cold stress, especially when entering the water either intentionally or by accident. Provisions for combating this hazard should be maintained at the sample location during this activity. Excessively hot temperatures may result in heat stress especially in scenarios where equipment is packed through the marsh.

Because all of these activities are conducted outside, electrical storms are a significant concern. The following measures will be incorporated to minimize this hazard:

- Where possible, utilize commercial warning systems and weather alerts to detect storms moving into the area.

- If on or in the water, get out of the water. Move to vehicles or preferably into enclosed buildings with plumbing and wiring.

- Where warning systems are not available, follow the 30/30 Rule (i*f there are less than 30 seconds between thunder and lightning, go inside for at least 30 minutes after the last thunder).*

See Section 4.0 of the Health and Safety Guidance Manual (HSGM) for additional protective measures.

6.0 PROCEDURES

6.1 Introduction

Collecting a representative sample of surface water or sediment may be difficult because of water movement, stratification, or heterogeneous distribution of the targeted analytes. To collect representative samples, one must standardize sampling methods related to site selection, sampling frequency, sample collection, sampling devices, and sample handling, preservation, and identification. Regardless of quality control applied during laboratory analyses and subsequent scrutiny of analytical data packages, reported data are no better than the confidence that can be placed in the representativeness of the samples. Consult Appendix C for guidance on sampling that should be considered during project planning and that may be helpful to field personnel.

6.1.1 Surface Water Sampling Equipment

The selection of sampling equipment depends on the site conditions and sample type to be acquired. In general, the most representative samples are obtained from mid-channel at a stream depth of 0.5 foot in a well-mixed stream; however, project-specific planning documents will address site-specific sampling requirements including sample collection points and sampling equipment. The most frequently used samplers include the following:

- Peristaltic pump
- Bailer
- Dip sampler
- Weighted bottle
- Hand pump
- Kemmerer
- Depth-integrating sampler

The dip sampler and weighted bottle sampler are used most often, and detailed discussions for these devices and the Kemmerer sampler are addressed subsequently in this section.

The criteria for selecting a sampler include:

1. Disposability and/or easy decontamination.

2. Inexpensive cost (if the item is to be disposed).

3. Ease of operation.

4. Non-reactive/non-contaminating properties - Teflon-coated, glass, stainless-steel or polyvinyl chloride (PVC) sample chambers are preferred (in that order).

Measurements collected for each sample (grab or each aliquot collected for compositing) shall include but not be limited to:

- Specific conductance
- Temperature
- pH
- Dissolved oxygen

Sample measurements shall be conducted as soon as the sample is acquired. Measurement techniques described in SOP SA-1.1 shall be followed. All pertinent data and results shall be recorded in a field notebook or on sample log sheets (see Attachment A) or an equivalent electronic form(s). These analyses may be selected to provide information on water mixing/stratification and potential contamination. Various types of water bodies have differing potentials for mixing and stratification.

In general, the following equipment if necessary for obtaining surface water samples:

- Required sampling equipment, which may include a remote sampling pole, weighted bottle sampler, Kemmerer sampler, or other device.

- Real-time air monitoring instrument (e.g., PID, FID) as directed in the project-specific planning document.

- Required PPE as directed in the project-specific planning document, which may include:

  - Nitrile surgeon's or latex gloves (layered as necessary).

  - Safety glasses.

  - Other items identified on the Safe Work Permit that may be required based on location-specific requirements (e.g., hearing protection, steel-toed work boots, hard hat). These provisions will be listed in the HASP or addressed by the FOL and/or SSO.

> ***Safety Reminder***
> The use of latex products may elicit an allergic reaction in some people. Should this occur, remove the latex gloves, treat for an allergic reaction, and seek medical attention as necessary.

  - Required paperwork (see SOP SA-6.3 and Attachments A and B to this SOP).

  - Required decontamination equipment.

  - Required sample containers.

  - Sealable polyethylene bags (e.g., Ziploc® baggies).

  - Heavy-duty cooler.

  - Ice.

- Paper towels and garbage bags.

- Chain-of-custody records and custody seals.

Dip Sampling

Specific procedures for collecting a dip or grab sample of surface water can vary based on site-specific conditions (e.g., conditions near the shore and how closely a sampler can safely get to the shore). The general procedure for collecting a sample using a pole or directly from the water body is as follows:

1. If using a remote sampling pole, securely attach the appropriate sample container to a pole of sufficient length to reach the water to be sampled. Samples for volatile analysis should be collected first. Use PPE as described in the HASP. When sample containers are provided pre-preserved or if the pole cannot accommodate a particular sample container, use a dedicated, clean, unpreserved bottle/container for sampling and transfer to an appropriately preserved container.

2. Remove the cap. Do not place the cap on the ground or elsewhere where it might become contaminated.

3. Carefully dip the container into the water just below the surface (or as directed by project-specific planning documents), and allow the bottle to fill. Sample bottles for volatile analysis must be filled with no headspace. Avoid contacting the bottom of the water body because this will disturb sediment that may interfere with the surface water sample.

4. Retrieve the container and carefully replace the cap securely. If using a container other than the sample bottle, pour the water from that container into the sample bottle and replace the cap securely.

5. Use a clean paper towel to clean and dry the outside of the container.

6. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

7. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

Constituents measured in grab samples collected near the water surface are only indicative of conditions near the surface of the water and may not be a true representation of the total concentration distributed throughout the water column and in the cross section. Therefore, as possible based on site conditions, the sampler may be required to augment dip samples with samples that represent both dissolved and suspended constituents and both vertical and horizontal distributions.

CAUTION

In areas prone to natural hazards such as alligators and snakes, etc., always use a buddy as a watch. Always have and use a lifeline or throwable device to extract persons who could potentially fall into the water. Be attentive to the signs, possible mounds indicating nests, and possible slides into the water. Remember that although snakes are typically encountered on the ground, it is not unheard of to see them on low-hanging branches. Be attentive to your surroundings because these may indicate that hazards are nearby.

Weighted Bottle Sampling

A grab sample can also be collected using a weighted holder that allows a bottle to be lowered to any desired depth, opened for filling, closed, and returned to the surface. This allows discrete sampling with depth. Several of these samples can be combined to provide a vertical composite. Alternatively, an open bottle can be lowered to the bottom and raised to the surface at a uniform rate so that the bottle collects sample throughout the total depth and is just filled on reaching the surface. The resulting sample using either method will roughly approach what is known as a depth-integrated sample.

A closed weighted bottle sampler consists of glass or plastic bottle with a stopper, a weight and/or holding device, and lines to open the stopper and lower or raise the bottle. The general procedure for sampling with this device is as follows:

1. Gently lower the sampler to the desired depth so as not to remove the stopper prematurely (watch for bubbles).

2. When the desired depth is reached, pull out the stopper with a sharp jerk of the stopper line.

3. Allow the bottle to fill completely, as evidenced by the absence of air bubbles.

4. Raise the sampler and cap the bottle.

5. Use a paper towel to clean and dry the outside of the container. This bottle can be used as the sample container as long as the bottle is an approved container type.

6. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

7. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

Kemmerer Sampler

If samples are desired at a specific depth, and the parameters to be measured do not require a Teflon-coated sampler, a standard Kemmerer sampler may be used. The Kemmerer sampler is a brass, stainless steel or acrylic cylinder with rubber stoppers that leave the ends open while it is lowered in a vertical position (thus allowing free passage of water through the cylinder). A "messenger" is sent down the line when the sampler is at the designated depth to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill sample bottles. The general procedure for sampling with this device is as follows:

1. Gently lower the sampler to the desired depth.

2. When the desired depth is reached, send down the messenger to close the cylinder and then raise the sampler.

3. Open the sampler valve to fill each sample bottle (filling bottles for volatile analysis first).

4. Use a paper towel to clean and dry the outside of the container.

5. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

6. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

6.1.2 Surface Water Sampling Techniques

Samples collected during site investigations may be grab samples or composite samples. The following general procedures apply to various types of surface water collection techniques:

- If a clean, pre-preserved sample container is not used, rinse the sample container least once with the water to be sampled before the sample is collected. This is not applicable when sample containers are provided pre-preserved because doing so will wash some or all of the preservative out of the bottle.

- For sampling moving water, collect the farthest downstream sample first, and continue sample collection in an upstream direction. In general, work from zones suspected of low contamination to zones of high contamination.

- Take care to avoid excessive agitation of the water because loss of volatile constituents could result.

- When obtaining samples in 40 mL vials with septum-lined lids for volatile organics analysis, fill the container completely (with a meniscus) to exclude any air space in the top of the bottle and to be sure that the Teflon liner of the septum faces in after the vial is filled and capped. Turn the vial upside down and tap gently on your wrist to check for air bubbles. If air bubbles rise in the bottle, add additional sample volume to the container.

- Do not sample at the surface, unless sampling specifically for a known constituent that is immiscible and on top of the water. Instead, invert the sample container, lower it to the approximate depth, and hold it at about a 45-degree angle with the mouth of the bottle facing upstream.

6.2 Onsite Water Quality Testing

Onsite water quality testing shall be conducted as described in SOP SA-1.1.

6.3 Sediment Sampling

6.3.1 General

If composite surface water samples are collected, sediment samples are usually collected at the same locations as the associated surface water samples. If only one sediment sample is to be collected, the sampling location shall be approximately at the center of the water body, in a depositional area if possible based on sample location restraints (see below), unless the SAP states otherwise.

Generally, coarser-grained sediments are deposited near the headwaters of reservoirs. Bed sediments near the center of a water body will be composed of fine-grained materials that may, because of their lower porosity and greater surface area available for adsorption, contain greater concentrations of contaminants. The shape, flow pattern, bathymetry (i.e., depth distribution), and water circulation patterns must all be considered when selecting sediment sampling sites. In streams, areas likely to have sediment accumulation (e.g., bends, behind islands or boulders, quiet shallow areas or very deep, low-velocity areas) shall be sampled, in general, and areas likely to show net erosion (i.e., high-velocity, turbulent areas) and suspension of fine solid materials shall be generally avoided. Follow instructions in the SAP, as applicable.

Chemical constituents associated with bottom material may reflect an integration of chemical and biological processes. Bottom samples reflect the historical input to streams, lakes, and estuaries with

respect to time, application of chemicals, and land use. Bottom sediments (especially fine-grained material) may act as a sink or reservoir for adsorbed heavy metals and organic contaminants (even if water column concentrations are less than detection limits). Therefore, it is important to minimize the loss of low-density "fines" during any sampling process.

Samples collected for volatile organic compound (VOC) analysis must be collected prior to any sample homogenization. Regardless of the method used for collection, the aliquot for VOC analysis must be collected directly from the sampling device (hand auger bucket, scoop, trowel), to the extent practical. If a device such as a dredge is used, the aliquot should be collected after the sample is placed in the mixing container prior to mixing.

In some cases, the sediment may be soft and not lend itself to collection by plunging Encore™ or syringe samplers into the sample matrix. In these cases, it is appropriate to open the sampling device, (Encore™ barrel or syringe) prior to sample collection, and carefully place the sediment in the device, filling it fully with the required volume of sample.

On active or former military sites, ordnance items may be encountered in some work areas. Care should be exercised when handling site media (such as if unloading a dredge as these materials may be scooped up). If suspected ordnance items are encountered, stop work immediately, move to shore and notify the Project Manager and Health and Safety Manager.

All relevant information pertaining to sediment sampling shall be documented as applicably described in SOP SA-6.3 and Attachment B or an equivalent electronic form.

6.3.2 Sampling Equipment and Techniques for Bottom Materials

A bottom-material sample may consist of a single scoop or core, or may be a composite of several individual samples in the cross section. Sediment samples may be obtained using onshore or offshore techniques.

SAFETY REMINDER

The following health and safety provisions apply when working on/over/near water:

- At least two people are required to be present at the sampling location in situations where the water depth and/or movement deem it necessary, each wearing a USCG-approved Personal Flotation Devices

- A minimum of three people are required if *any* of the following conditions are anticipated or observed:

  - Work in a waterway that is turbulent *or* swift that could sweep a sampler down stream should he or she fall in accidentally.

  - The underwater walking surface (e.g., stream/river bed) is suspected or observed to involve conditions that increase the potential for a worker to fall into the water. Examples include large/uneven rocks or boulders, dense mud or sediment that could entrap worker's feet, etc.

  - Waterway is tidal, and conditions such as those listed above could rapidly change.

The third person in the above condition must be equipped and prepared to render emergency support [e.g., lifeline, tethered Personal Flotation Device (Throwable Type IV, life saver), skiff, means to contact external emergency response support, etc.]

The following samplers may be used to collect sediment samples:

- Scoop sampler
- Dredge samplers
- Coring samplers

Each type of sampler is discussed below.

In general, the following equipment if necessary for obtaining sediment samples:

- Required sampling equipment, which may include a scoop sampler, dredge sampler, coring sampler, or stainless steel or pre-cleaned disposable trowel.
- Stainless bowl or pre-cleaned disposable bowl to homogenize sample.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in the project-specific planning document.
- Required PPE as directed in the project-specific planning document, which may include:
  - Nitrile surgeon's or latex gloves (layered as necessary).
  - Safety glasses.
  - Other items identified on the Safe Work Permit that may be required based on location-specific requirements (e.g., hearing protection, steel-toed work boots, hard hat). These provisions will be listed in the HASP or addressed by the FOL and/or SSO.
  - Required paperwork (see SOP SA-6.3 and Attachments A and B to this SOP).
  - Required decontamination equipment.
  - Required sample containers.
  - Sealable polyethylene bags (e.g., Ziploc® baggies).
  - Heavy-duty cooler.
  - Ice.
  - Paper towels and garbage bags.
  - Chain-of-custody records and custody seals.

<u>Scoop Sampler</u>

A scoop sampler consists of a pole to which a jar or scoop is attached. The pole may be made of bamboo, wood, PVC, or aluminum and be either telescoping or of fixed length. The scoop or jar at the end of the pole is usually attached using a clamp.

If the water body can be sampled from the shore or if the sampler can safely wade to the required location, the easiest and best way to collect a sediment sample is to use a scoop sampler. Scoop sampling also reduces the potential for cross-contamination. The general scoop sampling procedure is as follows:

1. Reach over or wade into the water body.

2. While facing upstream (into the current), scoop the sampler along the bottom in an upstream direction. Although it is very difficult not to disturb fine-grained materials at the sediment-water interface when using this method, try to keep disturbances to a minimum.

Dredge Samplers

Dredges are generally used to sample sediments that cannot easily be obtained using coring devices (e.g., coarse-grained or partially cemented materials) or when large quantities of sample are required. Dredges generally consist of a clam shell arrangement of two buckets. The buckets may either close upon impact or be activated by use of a "messenger." Some dredges are heavy and may require use of a winch and crane assembly for sample retrieval. The three major types of dredges are Peterson, Eckman and Ponar.

The Peterson dredge is used when the bottom is rocky, in very deep water, or when the flow velocity is high. The Peterson dredge shall be lowered very slowly as it approaches bottom, because it can force out and miss lighter materials if allowed to drop freely.

The Eckman dredge has only limited usefulness. It performs well where bottom material is unusually soft, as when covered with organic sludge or light mud. It is unsuitable, however, for sandy, rocky, and hard bottoms and is too light for use in streams with high flow velocities.

The Ponar dredge is a Peterson dredge modified by the addition of side plates and a screen on the top of the sample compartment. The screen over the sample compartment permits water to pass through the sampler as it descends, thus reducing the "shock wave." The Ponar dredge is easily operated by one person in the same fashion as the Peterson dredge. The Ponar dredge is one of the most effective samplers for general use on all types of substrates.

The general procedure for using dredge samplers is as follows:

1. Gently lower the dredge to the desired depth.

2. When the desired depth is reached, send the messenger down to cable to close the cylinder and then carefully raise the sampler.

3. Open the sampler to retrieve the sediment.

4. Transfer the sediment to the bowl in which it will be homogenized. Fill the sample bottle(s) for volatile analysis *prior to* homogenization. Homogenize the remainder of the sediment collected.

5. Fill the containers for all analyses other and VOCs.

6. Use a paper towel to clean and dry the outside of each container.

7. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

SAFETY REMINDER
Safety concerns using these dredges include lifting hazards, pinches, and compressions (several pinch points exist within the jaws and levers). In all cases, handle the dredge by the rope to avoid capturing fingers/hands.

Coring Samplers

Coring samplers are used to sample vertical columns of sediment. Many types of coring devices have been developed depending on the depth of water from which the sample is to be obtained, the nature of the bottom material, and the length of core to be collected. They vary from hand-push tubes to electronic vibrational core tube drivers.

Coring devices are particularly useful in pollutant monitoring because turbulence created by descent through the water is minimal, thus the fines at the sediment-water interface are only minimally disturbed. The sample is withdrawn intact, permitting the removal of only those layers of interest.

In shallow, wadeable waters, the use of a core liner or tube manufactured of Teflon or plastic is recommended for the collection of sediment samples. Caution should be exercised not to disturb the bottom sediments when the sample is obtained by wading in shallow water. The general procedure to collecting a sediment sample with a core tube is as follows:

1. Push the tube into the substrate until 4 inches or less of the tube is above the sediment-water interface. When sampling hard or coarse substrates, a gentle rotation of the tube while it is being pushed will facilitate greater penetration and decrease core compaction.

2. Cop the top of the tube to provide suction and reduce the chance of losing the sample.

3. Slowly extract the tube so as not to lose sediment from the bottom of the tube. Cap the bottom of the tube before removing it from the water. This will also help to minimize loss of sample.

4. Transfer the sediment to the bowl in which it will be homogenized. Fill the sample bottle(s) for volatile analysis prior to homogenization. Homogenize the remainder of the sediment collected.

5. Fill the containers for all analyses other and VOCs.

6. Use a paper towel to clean and dry the outside of each container.

7. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

In deeper, non-wadeable water bodies, sediment cores may be collected from a bridge or boat using different coring devices such as Ogeechee Sand Pounders, gravity cores, and vibrating coring devices. All three devices utilize a core barrel with a core liner tube system. The core liners can be removed from the core barrel and replaced with a clean core liner after each sample. Before extracting the sediment

from the coring tubes, the clear supernatant above the sediment-water interface in the core should be decanted from the tube. This is accomplished by turning the core tube to its side and gently pouring the liquid out until fine sediment particles appear in the waste liquid. Post-retrieval processing of samples is the same as above.

7.0 REFERENCES

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Feltz, H. R., 1980. Significance of Bottom Material Data in Evaluating Water Quality in Contaminants and Sediments. Ann Arbor, Michigan, Ann Arbor Science Publishers, Inc., V. 1, p. 271-287.

Kittrell, F. W., 1969. A Practical Guide to Water Quality Studies of Streams. U.S. Federal Water Pollution Control Administration, Washington, D.C., 135 p.

U.S. EPA, 1984. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-84-017.

U.S. EPA, 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Water Surveillance Branch, USEPA Surveillance and Analytical Division, Athens, Georgia.

U.S. Geological Survey, 1977. National Handbook of Recommended Methods for Water-Data Acquisition. Office of Water Data Coordination, USGS, Reston, Virginia.

ATTACHMENT A
SURFACE WATER SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SURFACE WATER SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Stream
[] Spring
[] Pond
[] Lake
[] Other: _____
[] QA Sample Type: _____

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

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

SAMPLING DATA:

	Color	pH	S.C.	Temp.	Turbidity	DO	Salinity	Other
Date:	Visual	Standard	mS/cm	Degrees C	NTU	mg/l	%	NA
Time:								
Depth:								
Method:								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

ATTACHMENT B
SOIL & SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

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

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

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

GRAB SAMPLE DATA:

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

COMPOSITE SAMPLE DATA:

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

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

APPENDIX C
GUIDANCE ON SAMPLING DESIGN AND SAMPLE COLLECTION

C.1 Defining the Sampling Program

Many factors are considered in developing a sampling program for surface water and/or sediment, including study objectives, accessibility, site topography, physical characteristics of the water body (e.g., flow and mixing), point and diffuse sources of contamination, and personnel and equipment available to conduct the study. For waterborne constituents, dispersion depends on vertical and lateral mixing within the body of water. For sediment, dispersion depends on bottom current or flow characteristics, sediment characteristics (e.g., density, size), and geochemical properties (that affect adsorption/desorption). The hydrogeologist developing the sampling plan must therefore know not only the mixing characteristics of streams and lakes but must also understand the role of fluvial-sediment transport, deposition, and chemical sorption.

C.1.1 Sampling Program Objectives

The scope of the sampling program must consider the sources and potential pathways for transport of contamination to or within a surface water body. Sources may include point sources (leaky tanks, outfalls, etc.) or nonpoint sources (e.g., contaminated runoff). The major pathways for surface water contamination (not including airborne deposition) are overland runoff, leachate influx to the water body, direct waste disposal (solid or liquid) into the water body, and groundwater flow influx from upgradient. The relative importance of these pathways, and therefore the design of the sampling program, is controlled by the physiographic and hydrologic features of the site, the drainage basin(s) that encompasses the site, and the history of site activities.

Physiographic and hydrologic features to be considered include slopes and runoff direction, areas of temporary flooding or pooling, tidal effects, artificial surface runoff controls such as berms or drainage ditches (and when they were constructed relative to site operation), and locations of springs, seeps, marshes, etc. In addition, the obvious considerations such as the locations of man-made discharge points to the nearest stream (intermittent or flowing), pond, lake, estuary, etc. shall be considered.

A more subtle consideration in designing the sampling program is the potential for dispersion of dissolved or sediment-associated contaminants away from the source. The dispersion could lead to a more homogeneous distribution of contamination at low or possibly non-detectable concentrations. Such dispersion does not, however, always readily occur. For example, obtaining a representative sample of contamination from a main stream immediately below an outfall or a tributary is difficult because the inflow frequently follows a stream bank with little lateral mixing for some distance. Sampling alternatives to overcome this situation include: (1) moving the sampling location far enough downstream to allow for adequate mixing, or (2) collecting integrated samples in a cross section. Also, non-homogeneous distribution is a particular problem with regard to sediment-associated contaminants, which may accumulate in low-energy environments (coves, river bends, deep spots, or even behind boulders) near or distant from the source while higher-energy areas (main stream channels) near the source may show no contaminant accumulation.

The distribution of particulates within a sample itself is an important consideration. Many organic compounds are only slightly water soluble and tend to adsorb onto particulate matter. Nitrogen, phosphorus, and heavy metals may also be transported by particulates. Samples must be collected with a representative amount of suspended material; transfer from the sampling device shall include transferring a proportionate amount of the suspended material.

C.1.2 Location of Sampling Stations

Accessibility is the primary factor affecting sampling costs. The desirability and utility of a sample for analysis and consideration of site conditions must be balanced against the costs of collection as controlled by accessibility. Bridges or piers are the first choice for locating a sampling station on a stream because bridges provide ready access and also permit the sampling technician to sample any point across the stream. A boat or pontoon (with an associated increase in cost) may be needed to sample locations on lakes, reservoirs, or larger rivers. Frequently, however, a boat will take longer to cross a water body and will hinder manipulation of the sampling equipment. Wading for samples is not recommended unless it is known that contaminant levels are low so that skin contact will not produce adverse health effects. This provides a built in margin of safety in the event that wading boots or other protective equipment should fail to function properly. If it is necessary to wade into the water body to obtain a sample, the sampler shall be careful to minimize disturbance of bottom sediments and must enter the water body downstream of the sampling location. If necessary, the sampling technician shall wait for the sediments to settle before taking a sample.

Under ideal and uniform contaminant dispersion conditions in a flowing stream, the same concentrations of each contaminant would occur at all points along the cross section. This situation is most likely downstream of areas of high turbulence. Careful site selection is needed to ensure, as nearly as possible, that samples are taken where uniform flow or deposition and good mixing conditions exist.

The availability of stream flow and sediment discharge records can be an important consideration in choosing sampling sites in streams. Stream flow data in association with contaminant concentration data are essential for estimating the total contaminant loads carried by the stream. If a gaging station is not conveniently located on a selected stream, the project hydrogeologist shall explore the possibility of obtaining stream flow data by direct or indirect methods. Remember these locations are also where you may encounter natural hazards as these are areas where they hunt. Always exercise extreme caution.

C.1.3 Frequency of Sampling

The sampling frequency and objectives of the sampling event will be defined by the project planning documents. For single-event site or area characterization sampling, both bottom material and overlying water samples shall be collected at the specified sampling stations. If valid data are available on the distribution of a contaminant between the solid and aqueous phases, it may be appropriate to sample only one phase, although this is not often recommended. If samples are collected primarily for monitoring purposes (i.e., consisting of repetitive, continuing measurements to define variations and trends at a given location), water samples should be collected at a pre-established and constant interval as specified in the project plans (often monthly or quarterly and during droughts and floods). Samples of bottom material should generally be collected from fresh deposits at least yearly, and preferably seasonally, during both spring and fall.

The variability in available water quality data shall be evaluated before determining the number and collection frequency of samples required to maintain an effective monitoring program.

C.2 <u>Surface Water Sample Collection</u>

C.2.1 Streams, Rivers, Outfalls and Drainage Features

Methods for sampling streams, rivers, outfalls, and drainage features (ditches, culverts) at a single point vary from the simplest of hand-sampling procedures to the more sophisticated multi-point sampling techniques known as the equal-width-increment (EWI) method or the equal-discharge-increment (EDI) methods (see below).

Samples from different depths or cross-sectional locations in the watercourse taken during the same sampling episode shall be composited. However, samples collected along the length of the watercourse or at different times may reflect differing inputs or dilutions and therefore shall not be composited. Generally, the number and type of samples to be taken depend on the river's width, depth, and discharge and on the suspended sediment the stream or river transports. The greater the number of individual points that are sampled, the more likely that the composite sample will truly represent the overall characteristics of the water.

In small streams less than about 20 feet wide, a sampling site can generally be found where the water is well mixed. In such cases, a single grab sample taken at mid-depth in the center of the channel is adequate to represent the entire cross section.

For larger streams, at least one vertical composite shall be taken with one sample each from just below the surface, at mid-depth, and just above the bottom. The measurement of dissolved oxygen (DO), pH, temperature, conductivity, etc., shall be made on each aliquot of the vertical composite and on the composite itself. For rivers, several vertical composites shall be collected, as directed in the project planning documents.

C.2.2 Lakes, Ponds and Reservoirs

Lakes, ponds, and reservoirs have a much greater tendency to stratify than rivers and streams. The relative lack of mixing requires that more samples be obtained. The number of water sampling sites on a lake, pond, or impoundment will vary with the size and shape of the basin. In ponds and small lakes, a single vertical composite at the deepest point may be sufficient. Similarly, measurement of DO, pH, temperature, etc. is to be conducted on each aliquot of the vertical composite and on the composite itself. In naturally formed ponds, the deepest point may have to be determined empirically; in impoundments, the deepest point is usually near the dam.

In lakes and larger reservoirs, several vertical composites shall be composited to form a single sample if a sample representative of the water column is required. These vertical composites are often collected along a transect or grid. In some cases, it may be of interest to form separate composites of epilimnetic and hypolimnetic zones. In a stratified lake, the epilimnion is the thermocline that is exposed to the atmosphere. The hypolimnion is the lower, "confined" layer that is only mixed with the epilimnion and vented to the atmosphere during seasonal "overturn" (when density stratification disappears). These two zones may thus have very different concentrations of contaminants if input is only to one zone, if the contaminants are volatile (and therefore vented from the epilimnion but not the hypolimnion), or if the epilimnion only is involved in short-term flushing (i.e., inflow from or outflow to shallow streams). Normally, however, a composite consists of several vertical composites with samples collected at various depths.

In lakes with irregular shape and with bays and coves that are protected from the wind, separate composite samples may be needed to adequately represent water quality because it is likely that only poor mixing will occur. Similarly, additional samples are recommended where discharges, tributaries, land use characteristics, and other such factors are suspected of influencing water quality.

Many lake measurements are now made in situ using sensors and automatic readout or recording devices. Single and multi-parameter instruments are available for measuring temperature, depth, pH, oxidation-reduction potential (ORP), specific conductance, DO, some cations and anions, and light penetration.

C.2.3 Estuaries

Estuarine areas are, by definition, zones where inland freshwaters (both surface and ground) mix with oceanic saline waters. Knowledge of the estuary type may be necessary to determine sampling locations. Estuaries are generally categorized into one of the following three types dependent on freshwater inflow and mixing properties:

- Mixed Estuary - characterized by the absence of a vertical halocline (gradual or no marked increase in salinity in the water column) and a gradual increase in salinity seaward. Typically, this type of estuary is shallow and is found in major freshwater sheet flow areas. Because this type of estuary is well mixed, sampling locations are not critical.

- Salt Wedge Estuary - characterized by a sharp vertical increase in salinity and stratified freshwater flow along the surface. In these estuaries, the vertical mixing forces cannot override the density differential between fresh and saline waters. In effect, a salt wedge tapering inland moves horizontally back and forth with the tidal phase. If contamination is being introduced into the estuary from upstream, water sampling from the salt wedge may miss it entirely.

- Oceanic Estuary - characterized by salinities approaching full-strength oceanic waters. Seasonally, freshwater inflow is small, with the preponderance of the fresh-saline water mixing occurring near or at the shore line.

Sampling in estuarine areas is normally based on the tidal phase, with samples collected on successive slack tides (i.e., when the tide turns). Estuarine sampling programs shall include vertical salinity measurements at 1- to 5-foot increments, coupled with vertical DO and temperature profiles.

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1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures to be used to collect surface, near-surface, and subsurface soil samples. Additionally, it describes the methods for sampling of test pits and trenches to determine subsurface soil and rock conditions and for recovery of small-volume or bulk samples from pits.

2.0 SCOPE

This document applies to the collection of surface, near-surface, and subsurface soil samples exposed through hand digging, hand augering, drilling, or machine excavating at hazardous substance sites for laboratory testing, onsite visual examination, and onsite testing.

3.0 GLOSSARY

Composite Sample - A composite sample is a combination of more than one grab sample from various locations and/or depths and times that is homogenized and treated as one sample. This type of sample is usually collected when determination of an average waste concentration for a specific area is required. Composite samples shall not be collected for volatile organics analysis.

Confined Space - As stipulated in 29 Code of Federal Regulations (CFR) 1910.146, a confined space means a space that: (1) is large enough and so configured that an employee can bodily enter and perform assigned work; (2) has limited or restricted means for entry or exit (e.g., tanks, vessels, silos, storage bins, hoppers, vaults, pits, and excavations); and (3) is not designed for continuous employee occupancy. TtNUS considers all confined space as permit-required confined spaces.

Grab Sample - One sample collected at one location and at one specific time.

Hand Auger - A sampling device used to extract soil from the ground.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

Sample for Non-Volatile Analyses - Includes all chemical parameters other than volatile organics (e.g., semivolatiles, pesticides/PCBs, metals, etc.) and those engineering parameters that do not require undisturbed soil for their analysis.

Split-Barrel Sampler - A steel tube, split in half lengthwise, with the halves held together by threaded collars at either end of the tube. Also called a split-spoon sampler, this device can be driven into resistant materials using a drive weight mounted in the drilling string. A standard split-barrel sampler is typically available in two common lengths, providing either 20-inch or 26-inch longitudinal clearance for obtaining 18-inch or 24-inch-long samples, respectively. These split-barrel samplers commonly range in size from 2 to 3.5 inches OD. The larger sizes are commonly used when a larger volume of sample material is required (see Attachment B).

Test Pit and Trench - Open, shallow excavations, typically rectangular (if a test pit) or longitudinal (if a trench), excavated to determine shallow subsurface conditions for engineering, geological, and soil chemistry exploration and/or sampling purposes. These pits are excavated manually or by machine (e.g., backhoe, clamshell, trencher, excavator, or bulldozer).

Thin-Walled Tube Sampler - A thin-walled metal tube (also called a Shelby tube) used to recover relatively undisturbed soil samples. These tubes are available in various sizes, ranging from 2 to 5 inches outside diameter (OD) and from 18 to 54 inches in length.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, selecting proposed sampling locations, and selecting field procedures used in the collection of soil samples. Additionally, in consultation with other project personnel (geologist, hydrogeologist, etc.), the Project Manager establishes the need for test pits or trenches and determines their approximate locations and dimensions.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan. This will include (but not be limited to) performing air quality monitoring during sampling, boring, and excavation activities and to ensure that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO/designee may also be required to advise the FOL on other safety-related matters regarding boring, excavation, and sampling, such as mitigative measures to address potential hazards from unstable trench walls, puncturing of drums or other hazardous objects, etc.

Field Operations Leader (FOL) - This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is responsible for finalizing the locations for collection of surface, near-surface, and subsurface (hand and machine borings, test pits/trenches) soil samples. He/she is ultimately responsible for the sampling and backfilling of boreholes, test pits, and trenches and for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP and/or other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, boring logs, test pit logs, container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

Competent Person - A Competent Person, as defined in 29 CFR 1929.650 of Subpart P - Excavations, means one who is capable of identifying existing and predictable hazards in the surroundings, or working conditions that are unsanitary, hazardous, or dangerous to employees, and who has authorization to take prompt corrective measures to eliminate them.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

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

- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

5.0 HEALTH AND SAFETY

Health and safety precautions are identified for individual sample collection procedures throughout this SOP. In addition to those precautions, the following general hazards may be incurred during sampling activities:

- Knee injuries from kneeling on hard or uneven surfaces
- Slips, trips, and falls
- Cuts and lacerations
- Traffic hazards associated with sampling in parking areas, along roadways and highways.

Methods of avoiding these hazards are provided below.

Knee injuries – If kneeling is required during soil sampling, this could result in knee injuries from stones/foreign objects and general damage due to stress on the joints. To minimize this hazard:

- Clear any foreign objects from the work area.
- Wear hard-sided knee pads.
- Stretch ligaments, tendons and muscles before, during and after. Take breaks as frequently as necessary.
- Report pre-existing conditions to the SSO if you feel this activity will aggravate an existing condition.

Slips, Trips, and Falls – These hazards exist while traversing varying terrains carrying equipment to sample locations. To minimize these hazards:

- Pre-survey sampling locations. Eliminate, barricade, or otherwise mark physical hazards leading to the locations.
- Carry small loads that do not restrict the field of vision.
- Travel the safest and clearest route (not necessarily the shortest).

Cuts and Lacerations - To prevent cuts and lacerations associated with soil sampling, the following provisions are required:

- Always cut away from yourself and others when cutting tubing or rope. This will prevent injury to yourself and others if the knife slips.
- Do not place items to be cut in your hand or on your knee.
- Change blades as necessary to maintain a sharp cutting edge. Many accidents result from struggling with dull cutting attachments.

- Whenever practical, wear cut-resistant gloves (e.g., leather or heavy cotton work gloves) at least on the hand not using the knife.
- Keep cutting surfaces clean and smooth.
- Secure items to be cut – do not hold them against the opposing hand, a leg, or other body part.
- When transporting glassware, keep it in a hard-sided container such as a cooler so that if there is a fall, you will be less likely to get cut by broken glass.
- DO NOT throw broken sample jars or glass ampoules into garbage bags. Place broken glass and glass ampoules in hard-sided containers such as a cardboard box or directly into a dumpster. DO NOT reach into garbage bags to retrieve any item accidentally thrown away. Empty the contents onto a flat surface to avoid punctures and lacerations from reaching where you cannot see.

Vehicular and Foot Traffic Hazards – When sampling along the roadway or near traffic patterns, follow the following precautions:

- Motorists may be distracted by onsite activities – ASSUME THEY DO NOT SEE YOU OR MEMBERS OF YOUR FIELD CREW.
- DO NOT place obstructions (such as vehicles) along the sides of the road that may cause site personnel to move into the flow of traffic to avoid your activities or equipment or that will create a blind spot.
- **Provide a required free space of travel**. Maintain at least 6 feet of space between you and moving traffic. Where this is not possible, use flaggers and/or signs to warn oncoming traffic of activities near or within the travel lanes.
- Face Traffic. Whenever feasible, if you must move within the 6 feet of the required free space or into traffic, attempt to face moving traffic at all times. Always leave yourself an escape route.
- Wear high-visibility vests to increase visual recognition by motorists.
- Do not rely on the vehicle operator's visibility, judgment, or ability. Make eye contact with the driver. Carefully and deliberately use hand signals so they will not startle or confuse motorists or be mistaken for a flagger's direction before moving into traffic.
- Your movements may startle a motorist and cause an accident, so move deliberately. Do not make sudden movements that might confuse a motorist.

6.0 PROCEDURES

The following procedures address surface and subsurface sampling.

CAUTION

Each situation must be evaluated individually to determine the applicability and necessity for obtaining a utility clearance ticket/dig permit. Common sense dictates, prior to digging or boring with power equipment, no matter what the depth, or digging by hand in a manner that could damage unprotected underground utilities, that a dig permit is required. See SOP HS-1.0, Utility Locating and Excavation Clearance, for additional

clarification. If you do not know or are unsure as to whether a ticket is necessary – **Get the Ticket**.

6.1 Overview

Soil sampling is an important adjunct to groundwater monitoring. Sampling of the soil horizons above the groundwater table can detect contaminants before they migrate to the water table, and can establish the amount of contamination absorbed or adsorbed on aquifer solids that have the potential of contributing to groundwater contamination.

Soil types can vary considerably on a hazardous waste site. These variations, along with vegetation, can affect the rate of contaminant migration through the soil. It is important, therefore, that a detailed record be maintained during sampling operations, particularly noting sampling locations, depths, and such characteristics as grain size, color, and odor. Subsurface conditions are often stable on a daily basis and may demonstrate only slight seasonal variation especially with respect to temperature, available oxygen and light penetration. Changes in any of these conditions can radically alter the rate of chemical reactions or the associated microbiological community, thus further altering specific site conditions. Certain vegetation species can create degradation products that can alter contaminant concentrations in soil. This is why vegetation types and extent of degradation of this foliage must be recorded. To prevent degradation, samples must be kept at their at-depth temperature or lower, protected from direct light, sealed tightly in approved glass containers, and be analyzed as soon as possible after collection. In addition, to the extent possible, vegetation should be removed from the sample.

The physical properties of the soil, its grain size, cohesiveness, associated moisture, and such factors as depth to bedrock and water table, will limit the depth from which samples can be collected and the method required to collect them. It is the intent of this document to present the most commonly employed soil sampling methods used at hazardous waste sites.

6.2 Soil Sample Collection

6.2.1 Procedure for Preserving and Collecting Soil Samples for Volatile Organic Compound Analysis

Samples collected using traditional methods such as collection in a jar with no preservation have been known to yield non-representative samples due to loss of volatile organic compounds (VOCs). To prevent such losses, preservation of samples with methanol or sodium bisulfate may be used to minimize volatilization and biodegradation. This preservation may be performed either in the field or laboratory, depending on the sampling methodology employed. Because of the large number of sampling methods and associated equipment required, careful coordination between field and laboratory personnel is needed.

Soil samples to be preserved by the laboratory are currently being collected using Method SW-846, 5035. For samples preserved in the field, laboratories are currently performing low-level analyses (sodium bisulfate preservation) and high- to medium-level analyses (methanol preservation) depending on the needs of the end user.

The following procedures outline the necessary steps for collecting soil samples to be preserved at the laboratory, and for collecting soil samples to be preserved in the field with methanol or sodium bisulfate.

6.2.1.1 Soil Samples to be Preserved at the Laboratory

Soil samples collected for volatile organic analysis that are to be preserved at the laboratory shall be obtained using a hermetically sealed sample vial such as an EnCore™ sampler. Each sample shall be obtained using a reusable sampling handle (T-handle) that can be provided with the EnCore™ sampler when requested and purchased. Collect the sample in the following manner for each EnCore™ sampler:

1. Scene Safety - Evaluate the area where sampling will occur. Ensure that the area is safe from physical, chemical, and natural hazards. Clear or barricade those hazards that have been identified.

2. Wear the appropriate personal protective equipment (PPE). This will include, at a minimum, safety glasses and nitrile surgeon's gloves. If you must kneel on the ground or place equipment on the surface being sampled, cover the ground surface with plastic to minimize surface contamination of your equipment and clothing. Wear knee pads to protect your knees from kneeling on hard or uneven surfaces.

3. Load the Encore™ sampler into the T-handle with the plunger fully depressed.

4. Expose the area to be sampled using a hand trowel or similar device to remove surface debris.

5. Press the T-handle against the freshly exposed soil surface, forcing soil into the sampler. The plunger will be forced upward as the cavity fills with soil.

6. When the sampler is full, rotate the plunger and lock it into place. If the plunger does not lock, the sampler is not full. This method ensures there is no headspace. Soft soil may require several plunges or forcing soil against a hard surface such as a sample trowel to ensure that headspace is eliminated.

7. Use a paper towel to remove soil from the side of the sampler so a tight seal can be made between the sample cap and the rubber O-ring.

8. With soil slightly piled above the rim of the sampler, force the cap on until the catches hook the side of the sampler.

9. Remove any surface soil from the outside of the sampler and place in the foil bag provided with the sampler. Good work hygiene practices and diligent decontamination procedures prevents the spread of contamination even on the outside of the containers.

10. Label the bag with appropriate information in accordance with SOP SA-6.3.

11. Place the full sampler inside a lined cooler with ice and cool to 4˚C ± 2 ˚C. Make sure any required trip blanks and temperature blanks are also in the cooler. Secure custody of the cooler in accordance with SOP SA-6.3.

12. Typically, collect three Encore™ samplers at each location. Consult the SAP or laboratory to determine the required number of Encore™ samplers to be collected.

13. The T-handle shall be decontaminated before moving to the next interval or location using a soap and water wash and rinse, and where applicable, the selected solvent as defined in the project planning documents.

Using this type of sampling device eliminates the need for field preservation and the shipping restrictions associated with preservatives. A complete set of instructions is included with each Encore™ sampler.

After the Encore™samples are collected, they should be placed on ice immediately and delivered to the laboratory within 48 hours (following the chain-of-custody and documentation procedures outlined in SOP SA-6.1). Samples must be preserved by the laboratory within 48 hours of sample collection.

6.2.1.2 Soil Samples to be Preserved in the Field

Soil samples preserved in the field may be prepared for analyses using both the low-level (sodium bisulfate preservation) and high- to medium-level (methanol preservation) methods.

Safety Reminder

When using chemicals in the field to preserve samples, the FOL and/or SSO must ensure that Materials Safety Data Sheets (MSDSs) have been provided with the chemicals to be used. They also must ensure that these chemicals have been added to the Chemical Inventory List contained within Section 5.0, Hazard Communication, of your Health and Safety Guidance Manual (HSGM). Lastly, but most importantly, the FOL and/or SSO must review the hazards with personnel using these chemicals and ensure that provisions are available for recommended PPE and emergency measures (e.g., eyewash, etc.).

Methanol Preservation (High to Medium Level):

Bottles may be pre-spiked with methanol in the laboratory or prepared in the field. Soil samples to be preserved in the field with methanol shall utilize 40 to 60 mL glass vials with septum-lined lids. Each sample bottle shall be filled with 25 mL of demonstrated analyte-free purge-and-trap grade methanol. The preferred method for adding methanol to the sample bottle is by removing the lid and using a pipette or scaled syringe to add the methanol directly to the bottle.

CAUTION

NEVER attempt to pipette by mouth

In situations where personnel are required to spike the septum using a hypodermic needle, the following provisions for handling sharps must be in place:

- Training of personnel regarding methods for handling of sharps
- Hard-sided containers for the disposal of sharps
- Provisions for treatment in cases where persons have received a puncture wound

Soil shall be collected with the use of a decontaminated (or disposable), small-diameter coring device such as a disposable tube/plunger-type syringe with the tip cut off. The outside diameter of the coring device must be smaller than the inside diameter of the sample bottle neck.

A small electronic balance or manual scale will be necessary for measuring the volume of soil to be added to the methanol-preserved sample bottle. Calibration of the scale shall be performed prior to use and intermittently throughout the day according to the manufacturer's requirements.

The sample should be collected as follows:

1. Weigh the unused syringe and plunger to the nearest 0.01 gram.

2. Pull the plunger back and insert the syringe into the soil to be sampled.

3. Collect 8 to 12 grams of soil by pushing the syringe barrel into the soil.

4. Weigh the sample and adjust until obtaining the required amount of sample.

5. Record the sample weight to the nearest 0.01 gram in the field logbook and/or on the sample log sheet.

6. Extrude the weighed soil sample into the methanol-preserved sample bottle taking care not to contact the sample container with the syringe.

7. If dirty, wipe soil particles from the threads of the bottle and cap. Cap the bottle tightly.

8. After capping the bottle, swirl the sample (do not shake) in the methanol and break up the soil such that all of the soil is covered with methanol.

9. Place the sample on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

Sodium Bisulfate Preservation (Low Level):

CAUTION
Care should be taken when adding the soil to the sodium bisulfate solution. A chemical reaction of soil containing carbonates (limestone) may cause the sample to effervesce or the vial to possibly explode. To avoid this hazard or hazards of this type, a small sample aliquot should be subjected to the sample preservative. If it effervesces in an open air environment, utilize an alternative method such as Encore™ or 2-ounce jar.

Bottles may be prepared in the laboratory or in the field with sodium bisulfate solution. Samples to be preserved in the field using the sodium bisulfate method are to be prepared and collected as follows:

1. Add 1 gram of sodium bisulfate to 5 mL of laboratory-grade deionized water in a 40 to 60 mL glass vial with septum-lined lid.

2. Collect the soil sample and record the sample weight to the nearest 0.01 gram in the field logbook or on the sample log sheet as described for methanol preservation

3. Add the weighed sample to the sample vial.

4. Collect duplicate samples using the methanol preservation method on a one-for-one sample basis because it is necessary for the laboratory to perform both low-level and medium-level analyses.

5. Place the samples on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

NOTE
If lower detection limits are necessary, an option to field preserving with sodium bisulfate may be to collect EnCore™ samplers at a given sample location. Consult the planning documents to determine whether this is required. If it is, collect samples in accordance with the Encore™ sampling procedure above and then send all samplers to the laboratory to perform the required preservation and analyses.

6.2.2 Procedure for Collecting Soil Samples for Non-Volatile Analyses

Samples collected for non-volatile analyses may be collected as either grab or composite samples as follows:

1. With a stainless steel trowel or other approved tool, transfer a portion of soil to be sampled to a stainless steel bowl or disposable inert plastic tray.

2. Remove roots, vegetation, sticks, and stones larger than the size of a green pea.

3. Thoroughly mix the soil in the bowl or tray to obtain as uniform a texture and color as practicable. The soil type, moisture content, amount of vegetation, and other factors may affect the amount of time required to obtain a properly mixed sample. In some cases, it may be impossible to obtain a uniform sample appearance. Use the field logbook to describe any significant difficulties encountered in obtaining a uniform mixture.

4. Transfer the mixed soil to the appropriate sample containers and close the containers.

5. Label the sample containers in accordance with SOP SA-6.3.

6. Place the containers in a cooler of ice as soon after collection as possible.

7. Prepare the sample shipment and ship the samples in accordance with SOP SA-6.1.

> ***NOTE***
> Cooling may not be required for some samples depending on the scheduled analyses. Consult the planning documents if in doubt regarding correct sample preservation conditions. When in doubt – Cool to 4˚C.

> ***NOTE***
> Head space is permitted in soil sample containers for non-volatile analyses to allow for sample expansion.

6.2.3 Procedure for Collecting Undisturbed Soil Samples

> ***NOTE***
> Use of thin-walled undisturbed tube samplers is restricted by the consistency of the soil to be sampled. Often, very loose and/or wet samples cannot be retrieved by the samplers, and soil with a consistency in excess of very stiff cannot be penetrated by the sampler. Devices such as Dennison or Pitcher core samplers can be used to obtain undisturbed samples of stiff soil. Using these devices normally increases sampling costs, and therefore their use should be weighed against the need for acquiring an undisturbed sample. These devices are not discussed in this SOP because they are not commonly used.

When it is necessary to acquire undisturbed samples of soil for purposes of engineering parameter analysis (e.g., permeability), a thin-walled, seamless tube sampler (Shelby tube) shall be employed using the following collection procedure:

1. In preparation for sampling utilizing a drill rig, field personnel must complete the following activities:

- Ensure that all subsurface drilling activities are preceded by a utility clearance for the area to be investigated. This includes activities described in SOP HS-1.0, Utility Location and Excavation Clearance, as well as any location-specific procedures that may apply.

> ***REMEMBER***
> If you are digging near a marked utility (within the diameter of an underground utility that has been marked plus 18 inches), you must first locate the utility through vacuum extraction or hand digging to ensure that your activities will not damage the utility.

- Complete an Equipment Inspection Checklist for the drill rig or direct-push technology (DPT) rig. This checklist will be provided in the HASP.
- Review the Safe Work Permit prior to conducting the activity.
- Review the activity to be conducted.

2. Remove all surface debris (e.g., vegetation, roots, twigs, etc.) from the specific sampling location and drill and/or clean out the borehole to the desired sampling depth. Be careful to minimize potential disturbance of the material to be sampled. In saturated material, withdraw the drill bit slowly to prevent loosening of the soil around the borehole and to maintain the water level in the hole at or above groundwater level.

> ***CAUTION***
> The use of bottom-discharge bits or jetting through an open-tube sampler to clean out the borehole shall not be allowed. Only the use of side-discharge bits is permitted.

3. Determine whether a stationary piston-type sampler is required to limit sample disturbance and aid in retaining the sample. Either the hydraulically operated or control rod activated-type of stationary piston sampler may be used.

4. Prior to inserting the tube sampler into the borehole, check to ensure that the sampler head contains a check valve. The check valve is necessary to keep water in the rods from pushing the sample out the tube sampler during sample withdrawal. In addition, the check valve maintains a positive suction within the tube to help retain the sample.

5. A stainless steel tube sampler is typically used to minimize chemical reaction between the sample and the sampling tube.

6. With the sampling tube resting on the bottom of the hole and the water level in the boring at groundwater level or above, push the tube into the soil with a continuous and rapid motion, without impacting or twisting. If the soil is too hard to penetrate by pushing alone, careful hammering may be used by minimizing drop distance (tapping) of the hammer. Before pulling the tube, turn it at least one revolution to shear the sample off at the bottom. In no case shall the tube be pushed farther than the length provided for the soil sample. Allow about 3 inches in the tube for cuttings and sludge.

7. Upon removal of the sampling tube from the hole, measure the length of sample in the tube and also the length penetrated.

8. Remove disturbed material in the upper end of the tube and measure the length of sample again.

9. After removing at least 1 inch of soil from the lower end, place enough packing material (clean inert material such as paper or cloth) tightly in each end of the Shelby tube and then pour melted wax into each end to make at least a ½-inch wax plug and then add more packing material to fill the voids at both ends.

10. Place plastic caps on the ends, tape the caps in place, and dip the ends in wax to prevent loss of soil.

11. Affix label(s) to the tube as required and record sample number, depth, penetration, and recovery length on the label.

12. Mark the "up" direction on the side and upper end of the tube with indelible ink.

13. Complete a chain-of-custody form (see SOP SA-6.3) and other required forms (including Attachment A of this SOP).

14. Ship samples protected with suitable resilient packing material to reduce shock, vibration, and disturbance.

CAUTION

To preserve sample integrity do not allow tubes to freeze, and store the samples vertically with the same orientation they had in the ground, (i.e., top of sample is up) in a cool place out of the sun at all times.

CAUTION

A primary concern in the preparation of the wax plugs is the potential for the heat source and melted wax to cause a fire and/or burns. Follow the directions below to prevent injury or fire.

Electrical Heating

Using hot plates to melt the wax is acceptable. In an outdoor setting, make sure a Ground Fault Circuit Interrupter (GFCI) is employed within the electrical circuit. If a portable generator is used, ensure that the generator is an adequate distance from the sampling operation (at least 50 feet). Ensure that the extension cord is rated for the intended load and for outdoor use and is free from recognizable damage. Ensure flammable preservatives are not employed or stored near the hot plate. Although a Hot Work Permit is not required, scene safety evaluation by site personnel of the above elements is. As always, if a fire potential exists, the provisions for extinguishing must be immediately accessible as well as any provisions for first aid measures.

Open Flame

If an open flame is used, the following provisions are necessary:

- Complete a Hot Work Permit and any local permit required for elevated temperature applications. The Hot Work Permit, provided in your HASP, will aid the FOL and/or the SSO in ensuring that fire protection provisions (extinguishers, fire watches, etc.) are in place as well as ensuring that local requirements have been addressed.

- Ensure that water is available to address any wax splashes or contact. If possible, immerse the contacted area. Where this is not possible, run water over the area and apply cold compresses. The need for medical attention or first aid shall be determined on site under the direction of the SSO.

6.3 Surface Soil Sampling

The simplest, most direct method of collecting surface soil samples for subsequent analysis is by use of a stainless steel shovel, hand auger, soil corer, or stainless steel or disposable plastic trowel.

NOTE

Multiple depth intervals are used to describe surface soil. Sometimes surface soil is defined as soil from 0 to 2 inches below ground surface (bgs), and sometimes it is defined as soil from other depths such as 0 to 2 feet bgs. Ensure that the definition of surface soil depth is clear before collecting surface soil samples.

For the purposes of instruction, the terms “surface soil” and “near-surface soil” are used in this SOP as follows:

- Surface soil - 0 to 6 inches bgs
- Near-surface soil - 6 to 18 inches bgs

If these intervals are defined differently in the planning documents, substitute the appropriate depth ranges.

In general, the following equipment is necessary for obtaining surface soil samples:

- Stainless steel or pre-cleaned disposable trowel.
- Stainless steel hand auger, soil corer, or shovel.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in project planning document.
- Required PPE.
  - Nitrile surgeon’s or latex gloves may be used, layered as necessary.
  - Safety glasses
  - Other – Items identified on the Safe Work Permit may be required based on location-specific requirements such as hearing protection, steel-toed work boots, and a hard hat when working near a drill rig. These provisions will be listed in the HASP or directed by the FOL and/or SSO.

Safety Reminder

The use of latex products may elicit an allergic reaction in some people. Should this occur, remove the latex gloves, treat for an allergic reaction, and seek medical attention as necessary.

- Required paperwork (see SOP SA-6.3 and Attachment A of this SOP)

- Required decontamination equipment
- Required sample container(s)
- Wooden stakes or pin flags
- Sealable polyethylene bags (e.g., Ziploc® baggies)
- Heavy duty cooler
- Ice
- Chain-of-custody records and custody seals

When acquiring surface soil samples, use the following procedure:

1. Place padding or use knee pads when kneeling near the sample location. If necessary, place plastic sheeting to provide a clean surface for sample equipment to avoid possible cross- contamination.
2. Carefully remove vegetation, roots, twigs, litter, etc. to expose an adequate soil surface area to accommodate sample volume requirements.
3. Using a precleaned syringe or EnCore™ samplers, follow the procedure in Section 6.2.1 for collecting surface soil samples for volatile analysis. Surface soil samples for volatile organic analysis should be collected deeper than 6 inches bgs because shallower material has usually lost most of the volatiles through evaporation. Ensure that the appropriate surface soil depth is being analyzed in accordance with the planning document.
4. Using decontaminated sampling tools, thoroughly mix in place a sufficient amount of soil to fill the remaining sample containers. See Section 6.5 of this procedure for hand auger instruction, as needed.
5. Transfer the sample into those containers utilizing a stainless steel trowel.
6. Cap and securely tighten all sample containers.
7. Affix a sample label to each container. Be sure to fill out each label carefully and clearly, addressing all the categories described in SOP SA-6.3.
8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.
9. Site restoration – Whenever removing sample materials, always restore the surface. It is our intent to leave the area better than we found it. Do NOT create trip hazards in areas when pedestrian traffic may exist.

6.4 Near-Surface Soil Sampling

Collection of samples from near the surface (depth of 6 to 18 inches) can be accomplished with tools such as shovels, hand auger, soil corers, and stainless steel or pre-cleaned disposable trowels and the equipment listed under Section 6.5 of this procedure.

To obtain near-surface soil samples, the following protocol shall be used:

1. With a clean shovel, make a series of vertical cuts in the soil to the depth required to form a square approximately 1 foot by 1 foot.

2. Lever out the formed plug and scrape the bottom of the freshly dug hole with a decontaminated stainless steel or pre-cleaned disposable trowel to remove any loose soil.

3. Follow steps 1 through 9 of Section 6.3.

6.5 Subsurface Soil Sampling With a Hand Auger

A hand augering system generally consists of a variety of stainless steel bucket bits (approximately 6.5 inches long and 2, 2.75, 3.25, and 4 inches in diameter), series of extension rods (available in 2-, 3-, 4- and 5-inch lengths), and a T-handle connected to extension rods and to the auger bucket. A larger-diameter bucket bit is commonly used to bore a hole to the desired sampling depth and then it is withdrawn. The larger-diameter bit is then replaced with a smaller-diameter bit, lowered down the hole, and slowly turned into the soil to the completion depth (approximately 6 inches). The apparatus is then withdrawn and the soil sample collected.

The hand auger can be used in a wide variety of soil conditions. It can be used to sample soil either from the surface, or to depths in excess of 12 feet. However, the presence of subsurface rocks and landfill material and collapse of the borehole normally limit sampling depth.

To accomplish soil sampling using a hand augering system, the following equipment is required:

- Complete hand auger assembly (variety of bucket bit sizes)
- Stainless steel mixing bowls
- The equipment listed in Section 6.3
- Miscellaneous hand tools as required to assemble and disassemble the hand auger units

CAUTION

Potential hazards associated with hand augering include:

- Muscle strain and sprain due to over twisting and/or over compromising yourself.
- Equipment failure due to excessive stress on the T-handle or rods through twisting. Failure of any of these components will result in a sudden release and potential injury due to that failure.

As in all situations, any intrusive activities that could damage underground utilities shall be proceeded by a Dig/Excavation permit/ticket. Call the Utility Locating service in the area or your Project Health and Safety Officer for more information. When in doubt – **Get the Ticket!**

To obtain soil samples using a hand auger, use the following procedure:

1. Wearing designated PPE, attach a properly decontaminated bucket bit to a clean extension rod and attach the T-handle to the extension rod.

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

3. Twist the bucket into the ground while pushing vertically downward on the auger. The cutting shoes fill the bucket as it is advanced into the ground.

4. As the auger bucket fills with soil, periodically remove any unneeded soil.

5. Add rod extensions as necessary to extend the reach of the auger. Also, note (in a field notebook, boring log, and/or on a standardized data sheet) any changes in the color, texture or odor of the soil as a function of depth. The project-specific planning document (SAP, HASP, etc.) describe requirements for scanning the soil with a real-time air monitoring instrument (e.g., PID, FID, etc.) and recording the measurements.

6. After reaching the desired depth (e.g., the top of the interval to be sampled), slowly and carefully withdraw the apparatus from the borehole to prevent or minimize movement of soil from shallower intervals to the bottom of the hole.

7. Remove the soiled bucket bit from the rod extension and replace it with another properly decontaminated bucket bit. The bucket bit used for sampling is to be smaller in diameter than the bucket bit employed to initiate the borehole.

8. Carefully lower the apparatus down the borehole. Care must be taken to avoid scraping the borehole sides.

9. Slowly turn the apparatus until the bucket bit is advanced approximately 6 inches.

10. Discard the top of the core (approximately 1 inch), which represents any loose material collected by the bucket bit before penetrating the sample material.

11. Using a precleaned syringe or EnCore™ samplers, follow the procedure in Section 6.2.1 for collecting a soil sample for volatile compound analysis directly from the bucket bit.
12. Utilizing a properly decontaminated stainless steel trowel or dedicated disposable trowel, remove the remaining sample material from the bucket bit and place into a properly decontaminated stainless steel mixing bowl.

13. Homogenize the sample material as thoroughly as practicable then fill the remaining sample containers. Refer to Section 6.2.2.

14. Follow steps 4 through 7 listed in Section 6.3.

6.5.1 Sampling Using Stainless Steel Soil Corers

A soil corer is a stainless steel tube equipped with a cutting shoe and sample window in the side. The soil corer is advanced into the soil by applying downward pressure (body weight). The soil is unloaded by then forcing a ram towards the cutting shoe, which results in the discharge of the soil core through a window in the sleeve.

Use, application, and sample protocol is the same as for hand augering provided above, but without necessarily rotating the corer while advancing it.

SAFETY REMINDER

Hand augering and soil corer sampling can be physically demanding based on the type of geology and subsurface encumbrances encountered. Soil coring has some added

hazards such the corer collapsing under your weight. To reduce the potential for muscle strain and damage, the following measures will be incorporated:

- Stretch and limber your muscles before heavy exertion. This hazard becomes more predominant in the early morning hours (prior to muscles becoming limber) and later in the day (as a result of fatigue).
- Job rotation – Share the duties so that repetitive actions do not result in fatigue and injury.
- Increase break frequencies as needed, especially as ambient conditions of heat and/or cold stress may dictate.
- Do not force the hand tools or use cheater pipes or similar devices to bypass an obstruction. Move to another location near the sampling point. Exerting additional forces on the sampling devices can result in damage and/or failure that could potentially injure someone in the immediate vicinity.
- Do not over compromise yourself when applying force to the soil corer or hand auger. If there is a sudden release, it could result in a fall or muscle injury due to strain.

6.6 Subsurface Soil Sampling with a Split-Barrel Sampler

A split-barrel (split-spoon) sampler consists of a heavy carbon steel or stainless steel sampling tube that can be split into two equal halves to reveal the soil sample (see Attachment B). A drive head is attached to the upper end of the tube and serves as a point of attachment for the drill rod. A removable tapered nosepiece/drive shoe attaches to the lower end of the tube and facilitates cutting. A basket-like sample retainer can be fitted to the lower end of the split tube to hold loose, dry soil samples in the tube when the sampler is removed from the drill hole. This split-barrel sampler is made to be attached to a drill rod and forced into the ground by means of a 140-pound or larger casing driver.

Safety Reminder

It is intended through the Equipment Inspection for Drill Rigs form provided in the HASP that the hammer and hemp rope, where applicable, associated with this activity will be inspected (no physical damage is obvious), properly attached to the hammer (suitable knots or sufficient mechanical devices), and is in overall good condition.

Split-barrel samplers are used to collect soil samples from a wide variety of soil types and from depths greater than those attainable with other soil sampling equipment.

The following equipment is used for obtaining split-barrel samples:

- Drilling equipment (provided by subcontractor).
- Split-barrel samplers (2-inch OD, 1-3/8-inch ID, either 20 inches or 26 inches long); Larger OD samplers are available if a larger volume of sample is needed.
- Drive weight assembly, 140-pound weight, driving head, and guide permitting free fall of 30 inches.
- Stainless steel mixing bowls.

- Equipment listed in Section 6.3.

The following steps shall be followed to obtain split-barrel samples (Steps 1 through 4 are typically performed by the drilling subcontractor):

1. Attach the split-barrel sampler to the sampling rods.

2. Lower the sampler into the borehole inside the hollow stem auger bits.

3. Advance the split-barrel sampler by hammering the length (typically 18 or 24 inches) of the split-barrel sampler into the soil using 140-pound or larger hammer.

4. When the desired depth is achieved, extract the drill rods and sampler from the augers and/or borehole.

5. Detach the sampler from the drill rods.

6. Place the sampler securely in a vise so it can be opened using pipe wrenches.

> ***CAUTION***
> Pipe wrenches are used to separate the split spoon into several components. The driller's helper should not apply excessive force through the use of cheater pipes or push or pull in the direction where, if the wrench slips, hands or fingers will be trapped against an immovable object.

7. Remove the drive head and nosepiece with the wrenches, and open the sampler to reveal the soil sample.

8. Immediately scan the sample core with a real-time air monitoring instrument (e.g., FID, PID, etc.) (as project-specific planning documents dictate). Carefully separate (or cut) the soil core, with a decontaminated stainless steel knife or trowel, at about 6-inch intervals while scanning the center of the core for elevated readings. Also scan stained soil, soil lenses, and anomalies (if present), and record readings.

9. If elevated vapor readings were observed, collect the sample scheduled for volatile analysis from the center of the core where elevated readings occurred. If no elevated readings where encountered, the sample material should be collected from the core's center (this area represents the least disturbed area with minimal atmospheric contact) (refer to Section 6.2.1).

10. Using the same trowel, remove remaining sample material from the split-barrel sampler (except for the small portion of disturbed soil usually found at the top of the core sample) and place the soil into a decontaminated stainless steel mixing bowl.

11. Homogenize the sample material as thoroughly as practicable then fill the remaining sample containers (refer to Section 6.2.2).

12. Follow steps 4 through 7 in Section 6.3.

6.7 Subsurface Soil Sampling Using Direct-Push Technology

Subsurface soil samples can be collected to depths of 40+ feet using DPT. DPT equipment,

responsibilities, and procedures are described in SOP SA-2.5.

6.8 Excavation and Sampling of Test Pits and Trenches

6.8.1 Applicability

This subsection presents routine test pit or trench excavation techniques and specialized techniques that are applicable under certain conditions.

CAUTION

During the excavation of trenches or pits at hazardous waste sites, several health and safety concerns arise from the method of excavation. No personnel shall enter any test pit or excavation over 4 feet deep except as a last resort, and then only under direct supervision of a Competent Person (as defined in 29 CFR 1929.650 of Subpart P - Excavations). Whenever possible, all required chemical and lithological samples should be collected using the excavator bucket or other remote sampling apparatus. If entrance is required, all test pits or excavations must be stabilized by bracing the pit sides using specifically designed wooden, steel, or aluminum support structures or through sloping and benching. Personnel entering the excavation may be exposed to toxic or explosive gases and oxygen-deficient environments; therefore, monitoring will be conducted by the Competent Person to determine if it is safe to enter. Any entry into a trench greater than 4 feet deep will constitute a Confined Space Entry and must be conducted in conformance with OSHA standard 29 CFR 1910.146. In all cases involving entry, substantial air monitoring, before entry, appropriate respiratory gear and protective clothing determination, and rescue provisions are mandatory. There must be at least three people present at the immediate site before entry by one of the field team members. This minimum number of people will increase based on the potential hazards or complexity of the work to be performed. The reader shall refer to OSHA regulations 29 CFR 1926.650, 29 CFR 1910.120, 29 CFR 1910.134, and 29 CFR 1910.146. High-hazard entries such as this will be supported by members of the Health Sciences Group professionally trained in these activities.

Excavations are generally not practical where a depth of more than about 15 to 20-feet is desired, and they are usually limited to a few feet below the water table. In some cases, a pumping system may be required to control water levels within the pit, providing that pumped water can be adequately stored or disposed. If soil data at depths greater than 15-feet are required, the data are usually obtained through test borings instead of test pits.

In addition, hazardous wastes may be brought to the surface by excavation equipment. This material, whether removed from the site or returned to the subsurface, must be properly handled according to any and all applicable federal, state, and local regulations.

6.8.2 Test Pit and Trench Excavation

Test pits or trench excavations are constructed with the intent that they will provide an open view of subsurface lithology and/or disposal conditions that a boring will not provide. These procedures describe the methods for excavating and logging test pits and trenches installed to determine subsurface soil and rock conditions. Test pit operations shall be logged and documented (see Attachment C).

Test pits and trenches may be excavated by hand or power equipment to permit detailed descriptions of the nature and contamination of the in-situ materials. The size of the excavation will depend primarily on the following:

- The purpose and extent of the exploration
- The space required for efficient excavation
- The chemicals of concern
- The economics and efficiency of available equipment

Test pits normally have a cross section that is 4 to 10 feet square; test trenches are usually 3 to 6 feet wide and may be extended for any length required to reveal conditions along a specific line. The following table provides guidelines for design consideration based on equipment efficiencies.

Equipment	Typical Widths, in Feet
Trenching machine	0.25 to 1.0
Backhoe/Track Hoe	2 to 6

The lateral limits of excavation of trenches and the position of test pits shall be carefully marked on area base maps. If precise positioning is required to indicate the location of highly hazardous materials, nearby utilities, or dangerous conditions, the limits of the excavation shall be surveyed. Also, if precise determination of the depth of buried materials is needed for design or environmental assessment purposes, the elevation of the ground surface at the test pit or trench location shall also be determined by survey. If the test pit/trench will not be surveyed immediately, it shall be backfilled and its position identified with stakes placed in the ground at the margin of the excavation for later surveying.

The construction of test pits and trenches shall be planned and designed in advance as much as possible. However, the following field conditions may necessitate revisions to the initial plans:

- Subsurface utilities
- Surface and subsurface encumbrances
- Vehicle and pedestrian traffic patterns
- Purpose for excavation (e.g., the excavation of potential ordnance items)

The final depth and construction method shall be collectively determined by the FOL and designated Competent Person. The actual layout of each test pit, temporary staging area, and spoils pile may further be predicated based on site conditions and wind direction at the time the test pit is excavated. Prior to excavation, the area may be surveyed by magnetometer or metal detector or other passive methods specified in SOP HS1.0, Utility Location and Excavation Clearance, to identify the presence of underground utilities or drums. Where possible, the excavator should be positioned upwind and preferably within an enclosed cab.

No personnel shall enter any test pit or excavation except as a last resort, and then only under direct supervision of a Competent Person. If entrance is required, OSHA requirements must be met (e.g., walls must be braced with wooden or steel braces, ladders must be placed for every 25 feet of lateral travel and extended 3 feet above ground surface). A temporary guard rail or vehicle stop must be placed along the surface of the hole before entry in situations where the excavation may be approached by traffic. Spoils will be stockpiled no closer than 2 feet from the sidewall of the excavation. The excavation equipment operator shall be careful not to undercut sidewalls and will, where necessary, bench back to

increase stability. The top cover, when considered clean, will be placed separately from the subsurface materials to permit clean cover. It is emphasized that the project data needs should be structured such that required samples can be collected without requiring entrance into the excavation. For example, samples of leachate, groundwater, or sidewall soil can be collected with telescoping poles or similar equipment.

Dewatering and watering may be required to ensure the stability of the side walls, to prevent the bottom of the pit from heaving, and to keep the excavation stable. This is an important consideration for excavations in cohesionless material below the groundwater table and for excavations left open greater than a day. Liquids removed as a result of dewatering operations must be handled as potentially contaminated materials. Procedures for the collection and disposal of such materials should be discussed in the site-specific project plans.

Where possible excavations and test pits shall be opened and closed within the same working day. Where this is not possible, the following engineering controls shall be put in place to control access:

- Trench covers/street plates
- Fences encompassing the entire excavation intended to control access
- Warning signs warning personnel of the hazards
- Amber flashing lights to demarcate boundaries of the excavation at night

Excavations left open will have emergency means to exit should someone accidentally enter.

6.8.3 Sampling in Test Pits and Trenches

6.8.3.1 General

Log test pits and trenches as they are excavated in accordance with the Test Pit Log presented in Attachment C. These records include plan and profile sketches of the test pit/trench showing materials encountered, their depth and distribution in the pit/trench, and sample locations. These records also include safety and sample screening information.

Entry of test pits by personnel is extremely dangerous, shall be avoided unless absolutely necessary, and can occur only after all applicable health and safety and OSHA requirements have been met as stated above. These provisions will be reiterated as appropriate in the project-specific HASP.

The final depth and type of samples obtained from each test pit will be determined at the time the test pit is excavated. Sufficient samples are usually obtained and analyzed to quantify contaminant distribution as a function of depth for each test pit. Additional samples of each waste phase and any fluids encountered in each test pit may also be collected.

In some cases, samples of soil may be extracted from the test pit for reasons other than waste sampling and chemical analysis, for instance, to obtain geotechnical information. Such information includes soil types, stratigraphy, strength, etc., and could therefore entail the collection of disturbed (grab or bulk) or relatively undisturbed (hand-carved or pushed/driven) samples that can be tested for geotechnical properties. The purposes of such explorations are very similar to those of shallow exploratory or test borings, but often test pits offer a faster, more cost-effective method of sampling than installing borings.

6.8.3.2 Sampling Equipment

The following equipment is needed for obtaining samples for chemical or geotechnical analysis from test pits and trenches:

- Backhoe or other excavating machinery.
- Shovels, picks, hand augers, and stainless steel trowels/disposable trowels.
- Sample container - bucket with locking lid for large samples; appropriate bottle ware for chemical or geotechnical analysis samples.
- Polyethylene bags for enclosing sample containers; buckets.
- Remote sampler consisting of 10-foot sections of steel conduit (1-inch-diameter), hose clamps, and right angle adapter for conduit (see Attachment D).

6.8.3.3 Sampling Methods

The methods discussed in this section refer to test pit sampling from grade level. If test pit entry is required, see Section 6.8.3.4.

- Excavate the trench or pit in several 0.5- to 1.0-foot depth increments. Where soil types support the use of a sand bar cutting plate, use of this device is recommended to avoid potentially snagging utilities with the excavator teeth. It is recommended that soil probes or similar devices be employed where buried items or utilities may be encountered. This permits the trench floor to be probed prior to the next cut.
- After each increment:
  - the operator shall wait while the sampler inspects the test pit from grade level
  - the sampler shall probe the next interval where this is considered necessary. Practical depth increments for lithological evaluations may range from 2 to 4 feet i or where lithological changes are noted.
- The backhoe operator, who will have the best view of the test pit, shall immediately cease digging if:
  - Any fluid phase, including groundwater seepage, is encountered in the test pit
  - Any drums, other potential waste containers, obstructions, or utility lines are encountered
  - Distinct changes of material being excavated are encountered

This action is necessary to permit proper sampling of the test pit and to prevent a breach of safety protocol. Depending on the conditions encountered, it may be required to excavate more slowly and carefully with the backhoe.

For obtaining test pit samples from grade level, the following procedure shall be followed:

- Use the backhoe to remove loose material from the excavation walls and floor to the greatest extent possible.

- Secure the walls of the pit, if necessary. (There is seldom any need to enter a pit or trench that would justify the expense of shoring the walls. All observations and samples should be taken from the ground surface.)

- Samples of the test pit material are to be obtained either directly from the backhoe bucket or from the material after it has been deposited on the ground, as follows:

  a. The sampler or FOL shall direct the backhoe operator to remove material from the selected depth or location within the test pit/trench.

  b. The backhoe operator shall bring the bucket over to a designated location on the sidewall a sufficient distance from the pit (at least 5 feet) to allow the sampler to work around the bucket.

  c. After the bucket has been set on the ground, the backhoe operator shall either disengage the controls or shut the machine down.

  d. When signaled by the operator that it is safe to do, the sampler will approach the bucket.

  e. The soil shall be monitored with a photoionization or flame ionization detector (PID or FID) as directed in the project -specific planning documents.

  f. The sampler shall collect the sample from the center of the bucket or pile in accordance with surface soil sampling procedures of Section 6.3 or 6.4, as applicable. Collecting samples from the center of a pile or bucket eliminates cross-contamination from the bucket or other depth intervals.

- If a composite sample is desired, several depths or locations within the pit/trench will be selected, and the bucket will be filled from each area. It is preferable to send individual sample bottles filled from each bucket to the laboratory for compositing under the more controlled laboratory conditions. However, if compositing in the field is required, each sample container shall be filled from materials that have been transferred into a mixing bucket and homogenized. Note that homogenization/compositing is not applicable for samples to be subjected to volatile organic analysis.

> ***CAUTION***
> Care must be exercised when using the remote sampler described in the next step because of potential instability of trench walls. In situations where someone must move closer than 2 feet to the excavation edge, a board or platform should be used to displace the sampler's weight to minimize the chance of collapse of the excavation edge. Fall protection should also be employed when working near the edges or trenches greater than 6 feet deep. An immediate means to extract people who have fallen into the trench will be immediately available. These means may include ladders or rope anchor points.

- Using the remote sampler shown in Attachment D, samples can be taken at the desired depth from the sidewall or bottom of the pit as follows:

  a. Scrape the face of the pit/trench using a long-handled shovel or hoe to remove the smeared zone that has contacted the backhoe bucket.

  b. Collect the sample directly into the sample jar, by scraping with the jar edge, eliminating the need for sample handling equipment and minimizing the likelihood of cross-contamination.

c. Cap the sample jar, remove it from the remote sampler assembly, and package the sample for shipment in accordance with SOP SA-6.3.

- Complete documentation as described in SOP SA-6.3 and Attachment C of this SOP.

6.8.3.4 In-Pit Sampling

Under rare conditions, personnel may be required to enter the test pit/trench. This is necessary only when soil conditions preclude obtaining suitable samples from the backhoe bucket (e.g., excessive mixing of soil or wastes within the test pit/trench) or when samples from relatively small discrete zones within the test pit are required. This approach may also be necessary to sample any seepage occurring at discrete levels or zones in the test pit that are not accessible with remote samplers.

In general, personnel shall sample and log pits and trenches from the ground surface, except as provided for by the following criteria:

- There are no practical alternative means of obtaining such data.

- The SSO and Competent Person determine that such action can be accomplished without breaching site safety protocol. This determination will be based on actual monitoring of the pit/trench after it is dug (including, at a minimum, measurements of oxygen concentration, flammable gases, and toxic compounds, in that order). Action levels will be provided in project-specific planning documents.

- A company-designated Competent Person determines that the pit/trench is stable trough soil classification evaluation/inspections or is made stable (by cutting/grading the sidewalls or using shoring) prior to entrance of any personnel. OSHA requirements shall be strictly observed.

If these conditions are satisfied, only one person may enter the pit/trench. On potentially hazardous waste sites, this individual shall be dressed in selected PPE as required by the conditions in the pit. He/she shall be affixed to a harness and lifeline and continuously monitored while in the pit.

A second and possible third individual shall be fully dressed in protective clothing including a self-contained breathing device and on standby during all pit entry operations to support self rescue or assisted self rescue. The individual entering the pit shall remain therein for as brief a period as practical, commensurate with performance of his/her work. After removing the smeared zone, samples shall be obtained with a decontaminated trowel or spoon.

6.8.3.5 Geotechnical Sampling

In addition to the equipment described in Section 6.8.3.2, the following equipment is needed for geotechnical sampling:

- Soil sampling equipment, similar to that used in shallow drilled boring (i.e., thin-walled tube samplers), that can be pushed or driven into the floor of the test pit.

- Suitable driving (e,g., sledge hammer) or pushing (e.g., backhoe bucket) equipment used to advance the sampler into the soil.

- Knives, spatulas, and other suitable devices for trimming hand-carved samples.

- Suitable containers (bags, jars, tubes, boxes, etc.), labels, wax, etc. for holding and safely transporting collected soil samples.

- Geotechnical equipment (pocket penetrometer, torvane, etc.) for field testing collected soil samples for classification and strength properties.

Disturbed grab or bulk geotechnical soil samples may be collected for most soil in the same manner as comparable soil samples for chemical analysis. These collected samples may be stored in jars or plastic-lined sacks (larger samples), which will preserve their moisture content. Smaller samples of this type are usually tested for their index properties to aid in soil identification and classification: larger bulk samples are usually required to perform compaction tests.

Relatively undisturbed samples are usually extracted in cohesive soil using thin-walled tube samplers, and such samples are then tested in a geotechnical laboratory for their strength, permeability, and/or compressibility. The techniques for extracting and preserving such samples are similar to those used in performing Shelby tube sampling in borings, except that the sampler is advanced by hand or backhoe, rather than by a drill rig. Also, the sampler may be extracted from the test pit by excavation around the tube when it is difficult to pull it out of the ground. If this excavation requires entry of the test pit, the requirements described in Section 6.8.3.4 shall be followed. The thin-walled tube sampler shall be pushed or driven vertically into the floor or steps excavated in the test pit at the desired sampling elevations. Extracting tube samples horizontally from the walls of the test pit is not appropriate because the sample will not have the correct orientation.

A sledge hammer or backhoe may be used to drive or push the tube into the ground. Place a piece of wood over the top of the sampler or sampling tube to prevent damage during driving/pushing of the sample. Pushing the sampler with a constant thrust is always preferable to driving it with repeated blows, thus minimizing disturbance to the sample. When using a sledge hammer, it is recommended that the sampler be stabilized using a rope/strap wrench or pipe wrench to remove the person's hands holding the sampler from the strike zone. If the sample cannot be extracted by rotating it at least two revolutions (to shear off the sample at the bottom), hook the sampler to the excavator or backhoe and extract. This means an alternative head will be used as a connection point or that multiple choke hitches will be applied to extract the sampler. If this fails and the excavator can dig deeper without potentially impacting subsurface utilities, excavate the sampler. If this fails or if the excavator cannot be used due to subsurface utilities, hand-excavate to remove the soil from around the sides of the sampler. If hand-excavation requires entry into the test pit, the requirements in Section 6.8.3.4 must be followed. Prepare the sample as described in Steps 9 through 13 in Section 6.2.3, and label, pack and transport the sample in the required manner, as described in SOPs SA-6.3 and SA-6.1.

6.8.4 Backfilling of Trenches and Test Pits

All test pits and excavations must be either backfilled, covered, or otherwise protected at the end of each day. No excavations shall remain open during non-working hours unless adequately covered or otherwise protected.

Before backfilling, the onsite crew may photograph, if required by the project-specific work plan, all significant features exposed by the test pit and trench and shall include in the photograph a scale to show dimensions. Photographs of test pits shall be marked to include site number, test pit number, depth, description of feature, and date of photograph. In addition, a geologic description of each photograph shall be entered in the site logbook. All photographs shall be indexed and maintained as part of the project file for future reference.

After inspection, backfill material shall be returned to the pit under the direction of the FOL. Backfill should be returned to the trench or test pit in 6-inch to 1-foot lifts and compacted with the bucket. Remote controlled tampers or rollers may be lowered into the trench and operated from top side. This procedure will continue to the grade surface. It is recommended that the trench be tracked or rolled in. During

excavation, clean soil from the top 2 feet may have been separated to be used to cover the last segments. Where these materials are not clean, it is recommended that clean fill be used for the top cover.

If a low-permeability layer is penetrated (resulting in groundwater flow from an upper contaminated flow zone into a lower uncontaminated flow zone), backfill material must represent original conditions or be impermeable. Backfill could consist of a soil-bentonite mix prepared in a proportion specified by the FOL (representing a permeability equal to or less than original conditions). Backfill can be covered by "clean" soil and graded to the original land contour. Revegetation of the disturbed area may also be required.

6.9 Records

The appropriate sample log sheet (see Attachment A of this SOP) must be completed by the site geologist/sampler for all samples collected. All soil sampling locations should be documented by tying in the location of two or more nearby permanent landmarks (building, telephone pole, fence, etc.) or obtaining GPS coordinates; and shall be noted on the appropriate sample log sheet, site map, or field notebook. Surveying may also be necessary, depending on the project requirements.

Test pit logs (see Attachment C of this SOP) shall contain a sketch of pit conditions. If the project-specific work plan requires photographs, at least one photograph with a scale for comparison shall be taken of each pit. Included in the photograph shall be a card showing the test pit number. Boreholes, test pits, and trenches shall be logged by the field geologist in accordance with SOP GH-1.5.

Other data to be recorded in the field logbook include the following:

- Name and location of job
- Date of boring and excavation
- Approximate surface elevation
- Total depth of boring and excavation
- Dimensions of pit
- Method of sample acquisition
- Type and size of samples
- Soil and rock descriptions
- Photographs if required
- Groundwater levels
- PID/FID/LEL/O_2 meter readings
- Other pertinent information, such as waste material encountered

In addition, site-specific documentation to be maintained by the SSO and/or Competent Person will be required including:

- Calibration logs
- Excavation inspection checklists
- Soil type classification

7.0 REFERENCES

American Society for Testing and Materials, 1987. ASTM Standards D1587-83 and D1586-84. ASTM Annual Book of Standards. ASTM. Philadelphia, Pennsylvania. Volume 4.08.

NUS Corporation, 1986. Hazardous Material Handling Training Manual.

NUS Corporation and CH2M Hill, August, 1987. Compendium of Field Operation Methods. Prepared for the U.S. EPA.

OSHA, Excavation, Trenching and Shoring 29 CFR 1926.650-653.

OSHA, Confined Space Entry 29 CFR 1910.146.

USEPA, November 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual.

ATTACHMENT A
SOIL & SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

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

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

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

GRAB SAMPLE DATA:

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

COMPOSITE SAMPLE DATA:

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

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

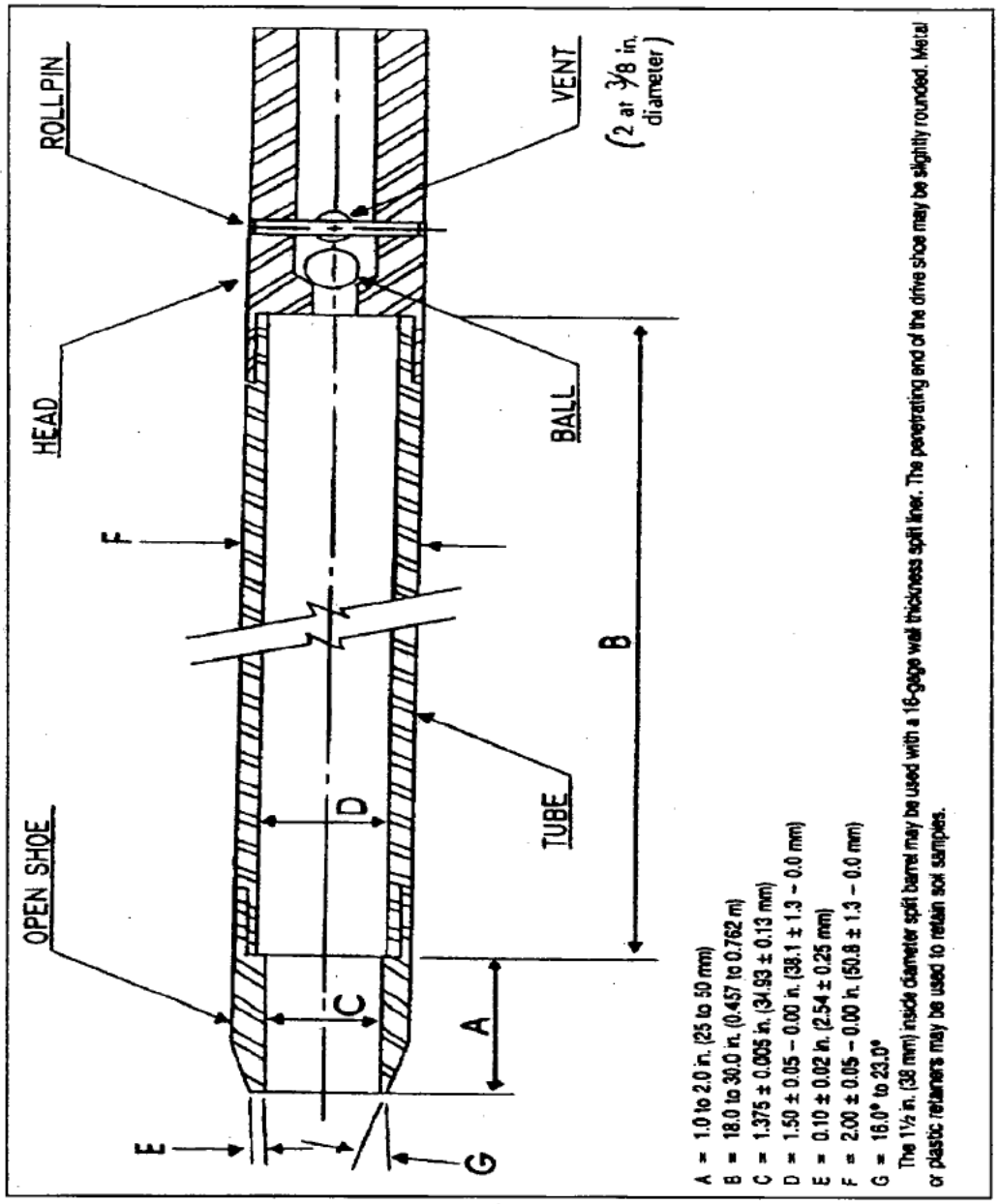
MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

ATTACHMENT B
SPLIT-SPOON SAMPLER



A = 1.0 to 2.0 in. (25 to 50 mm)
B = 18.0 to 30.0 in. (0.457 to 0.762 m)
C = 1.375 ± 0.005 in. (34.93 ± 0.13 mm)
D = 1.50 ± 0.05 – 0.00 in. (38.1 ± 1.3 – 0.0 mm)
E = 0.10 ± 0.02 in. (2.54 ± 0.25 mm)
F = 2.00 ± 0.05 – 0.00 in. (50.8 ± 1.3 – 0.0 mm)
G = 16.0° to 23.0°

The 1½ in. (38 mm) inside diameter split barrel may be used with a 16-gage wall thickness split liner. The penetrating end of the drive shoe may be slightly rounded. Metal or plastic retainers may be used to retain soil samples.

ATTACHMENT C
TEST PIT LOG

Tetra Tech NUS, Inc.

TEST PIT LOG

Page ___ of ___

PROJECT NAME: _____ TEST PIT No.: _____
PROJECT NUMBER: _____ DATE: _____
LOCATION: _____ GEOLOGIST: _____

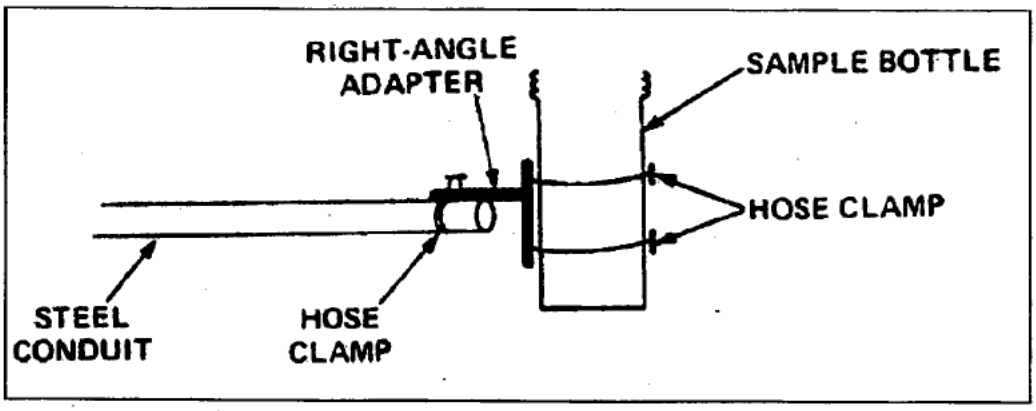
Depth (Ft.)	Lithology Change (Depth/Ft.)	MATERIAL DESCRIPTION Soil/Waste Characteristics (lithology, density, color, etc.)	USCS	Remarks	PID/FID READING Source (ppm)	BZ (ppm)

TEST PIT CROSS SECTION AND / OR PLAN VIEW

REMARKS: _____

PHOTO LOG: _____

ATTACHMENT D
REMOTE SAMPLE HOLDER FOR TEST PIT/TRENCH SAMPLING



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1.0 PURPOSE

The purpose of this procedure is to provide general reference information on Direct Push Technology (DPT). DPT is designed to collect soil, groundwater, and soil gas samples without using conventional drilling techniques. The advantage of using DPT over conventional drilling includes the generation of little or no drill cuttings, sampling in locations with difficult accessibility, reduced overhead clearance requirements, no fluid introduction during probing, and typical lower costs per sample than with conventional techniques. Disadvantages include a maximum penetration depth of approximately 15 to 40 feet in dense soils (although it may be as much as 60 to 80 feet in certain types of geological environments), reduced capability of obtaining accurate water-level measurements, and the inability to install permanent groundwater monitoring wells. The methods and equipment described herein are for collection of surface and subsurface soil samples and groundwater samples. Soil gas sampling is discussed in SOP SA-2.4.

2.0 SCOPE

This procedure provides information on proper sampling equipment and techniques for DPT. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require adjustments in methodology.

3.0 GLOSSARY

Direct Push Technology (DPT) - DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools. A primary advantage of DPT over conventional drilling techniques is that DPT results in the generation of little or no investigation derived waste.

Geoprobe® - Geoprobe® is a manufacturer of a hydraulically-powered, percussion/probing machines utilizing DPT to collect subsurface environmental samples. Geoprobe® relies on a relatively small amount of static weight (vehicle) combined with percussion as the energy for advancement of a tool string. The Geoprobe® equipment can be mounted in a multitude of vehicles for access to all types of environmental sites.

HydroPunch™ - HydroPunch™ is a manufacturer of stainless steel and Teflon® sampling tools that are capable of collecting representative groundwater and/or soil samples without requiring the installation of a groundwater monitoring well or conventional soil boring. HydroPunch™ is an example of DPT sampling equipment.

Flame Ionization Detector (FID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing a flame as the energizing source.

Photo Ionization Detector (PID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing ultraviolet radiation as the energizing source.

4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for selecting and/or reviewing the appropriate DPT drilling procedure required to support the project objectives.

Field Operations Leader (FOL)- The FOL is primarily responsible for performing the DPT in accordance with the project-specific plan.

5.0 SOIL SAMPLING PROCEDURES

5.1 General

The common methodology for the investigation of the vadose zone is soil boring drilling and soil sampling. However, drilling soil borings can be very expensive. Generally the advantage of DPT for subsurface soil sampling is the reduced cost of disposal of drilling cuttings and shorter sampling times.

5.2 Sampling Equipment

Equipment needed for conducting DPT drilling for subsurface soil sampling includes, but is not limited to, the following:

- Geoprobe® Sampling Kit
- Cut-resistant gloves
- 4-foot x 1.5-inch diameter macrocore sampler
- Probe sampling adapters
- Roto-hammer with 1.5-inch bit
- Disposable acetate liners for soil macrocore sampler
- Cast aluminum or steel drive points
- Geoprobe® AT-660 Series Large Bore Soil Sampler, or equivalent
- Standard decontamination equipment and solutions

For health and safety equipment and procedures, follow the direction provided in the Safe Work Permit in Attachment 1, or the more detailed directions provided in the project's Health and Safety Plan.

5.3 DPT Sampling Methodology

There are several methods for the collection of soil samples using DPT drilling. The most common method is discussed in the following section. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project-specific plan.

- Macrocore samplers fitted with detachable aluminum or steel drive points are driven into the ground using hydraulic pressure. If there is concrete or pavement over a sampling location, a Roto-hammer is used to drill a minimum 1.5-inch diameter hole through the surface material. A Roto-hammer may also be used if very dense soils are encountered.

- The sampler is advanced continuously in 4-foot intervals or less if desired. No soil cuttings are generated because the soil which is not collected in the sampler is displaced within the formation.

- The sampler is retracted from the hole, and the 4-foot continuous sample is removed from the outer coring tube. The sample is contained within an inner acetate liner.

- Attach the metal trough from the Geoprobe® Sampling Kit firmly to the tail gate of a vehicle. If a vehicle with a tail gate is not available, secure the trough on another suitable surface.

- Place the acetate liner containing the soils in the trough.

- While wearing cut-resistant gloves (constructed of leather or other suitable material), cut the acetate liner through its entire length using the double-bladed knife that accompanies the Geoprobe® Sampling Kit. Then remove the strip of acetate from the trough to gain access to the collected soils. Do not attempt to cut the acetate liner while holding it in your hand.

- Field screen the sample with an FID or PID, and observe/examine the sample (according to SOP GH-1.3). If appropriate, transfer the sample to sample bottles for laboratory analysis. If additional volume is required, push an additional boring adjacent to the first and composite/mix the same interval. Field compositing is usually not acceptable for sample requiring volatile organics analysis.

- Once sampling has been completed, the hole is backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch is used to cap holes through paved or concrete areas. All holes should be finished smooth to existing grade.

- In the event the direct push van/truck cannot be driven to a remote location or a sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric operated equipment (e.g., jack hammer).

- Sampling equipment is decontaminated prior to collecting the next sample.

6.0 GROUNDWATER SAMPLING PROCEDURES

6.1 General

The most common methodology for the investigation of groundwater is the installation and sampling of permanent monitoring wells. If only groundwater screening is required, the installation and sampling of temporary well points may be performed. The advantage of temporary well point installation using DPT is reduced cost due to no or minimal disposal of drilling cuttings and well construction materials, and shorter installation/times sampling.

Two disadvantages of DPT drilling for well point installation are:

- In aquifers with low yields, well points may have to be sampled without purging or development.
- If volume requirements are high, this method can be time consuming for low yield aquifers.

6.2 Sampling Equipment

Equipment needed for temporary well installation and sampling using DPT includes, but is not limited, to the following:

- 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point
- Connecting rods
- Roto-hammer with 1.5-inch bit
- Mechanical jack
- 1/4-inch OD polyethylene tubing
- 3/8-inch OD polyethylene tubing
- Peristaltic pump
- Standard decontamination equipment and solutions

6.3 DPT Temporary Well Point Installation and Sampling Methodology

There are several methods for the installation and sampling of temporary well points using DPT. The most common methodology is discussed below. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project specific plan.

- A 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point attached to connecting rods is driven into the ground to the desired depth using a rotary electric hammer or other direct push drill rig. If there is concrete or pavement over a sampling location, a Roto-hammer or electric coring machine is used to drill a hole through the surface material.

- The well point will be allowed to equilibrate for at least 15 minutes, after which a measurement of the static water level will be taken. The initial measurement of the water level will be used to assess the amount of water which is present in the well point and to determine the amount of silt and sand infiltration that may have occurred.

- The well point will be developed using a peristaltic pump and polyethylene tubing to remove silt and sand which may have entered the well point. The well point is developed by inserting polyethylene tubing to the bottom of the well point and lifting and lowering the tubing slightly while the pump is operating. The pump will be operated at a maximum rate of approximately 2 liters per minute. After removal of sediment from the bottom of the well point, the well point will be vigorously pumped at maximum capacity until discharge water is visibly clear and no further sediments are being generated. Measurements of pH, specific conductance, temperature, and turbidity shall be recorded every 5 to 10 minutes during the purging process. After two consistent readings of pH, specific conductance, temperature and turbidity ($\pm$10 percent), the well may be sampled.

- A sample will be collected using the peristaltic pump set at the same or reduced speed as during well development. Samples (with the exception of the samples to be analyzed for volatile organic compounds, VOCs) will be collected directly from the pump discharge. Sample containers for VOCs will be filled by (first shutting off the pump) crimping the discharge end of the sample tubing when filled, removing the inlet end of the sample tubing from the well, suspending the inlet tubing above the vial, and allowing water to fill each vial by gravity flow.

- Once the groundwater sample has been collected, the connecting rods and well point will be removed from the hole with the direct push rig hydraulics. The hole will be backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch will be used to cap holes through paved or concrete areas. All holes will be finished smooth to existing grade.

- In the event the direct push van/truck cannot be driven to a remote location or sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric-operated equipment (e.g., jack hammer).

- Decontaminate the equipment before moving to the next location.

7.0 RECORDS

A record of all field procedures, tests, and observations must be recorded in the field logbook, boring logs, and sample log sheets, as needed. Entries should include all pertinent data regarding the investigation. The use of sketches and field landmarks will help to supplement the investigation and evaluation.

ATTACHMENT 1
SAFE WORK PERMIT FOR DPT OPERATIONS

Permit No. _____ Date: _____ Time: From _____ to _____

SECTION I: General Job Scope

I. Work limited to the following (description, area, equipment used): **Monitoring well drilling and installation through direct push technology**

II. Required Monitoring Instruments: _____

III. Field Crew: _____

IV. On-site Inspection conducted ☐ Yes ☐ No Initials of Inspector _____ _____
TtNUS

SECTION II: General Safety Requirements (To be filled in by permit issuer)

V. Protective equipment required
Level D ☒ Level B ☐
Level C ☐ Level A ☐
Detailed on Reverse

Respiratory equipment required
Full face APR ☐ Escape Pack ☐
Half face APR ☐ SCBA ☐
SKA-PAC SAR ☐ Bottle Trailer ☐
Skid Rig ☐ None ☒

Level D Minimum Requirements: Sleeved shirt and long pants, safety footwear, and work gloves. Safety glasses, hard hats, and hearing protection will be worn when working near or sampling in the vicinity of the DPT rig.

Modifications/Exceptions.

VI. Chemicals of Concern | Action Level(s) | Response Measures

VII. Additional Safety Equipment/Procedures

Hard-hat	☒ Yes	☐ No	Hearing Protection (Plugs/Muffs)	☒ Yes	☐ No
Safety Glasses	☒ Yes	☐ No	Safety belt/harness	☐ Yes	☒ No
Chemical/splash goggles	☐ Yes	☒ No	Radio	☐ Yes	☒ No
Splash Shield	☐ Yes	☒ No	Barricades	☒ Yes	☐ No
Splash suits/coveralls	☐ Yes	☐ No	Gloves (Type - _____)	☐ Yes	☐ No
Steel toe Work shoes or boots	☒ Yes	☐ No	Work/warming regimen	☐ Yes	☐ No

Modifications/Exceptions: Reflective vests for high traffic areas.

VIII. Procedure review with permit acceptors

	Yes	NA		Yes	NA
Safety shower/eyewash (Location & Use)	☐	☒	Emergency alarms	☐	☐
Daily tail gate meetings	☒	☐	Evacuation routes	☐	☐
Contractor tools/equipment/PPE inspected	☐	☐	Assembly points	☐	☐

IX. Site Preparation

Utility Clearances obtained for areas of subsurface investigation	☐ Yes	☐ No
Physical hazards removed or blockaded	☐ Yes	☐ No
Site control boundaries demarcated/signage	☐ Yes	☐ No

X. Equipment Preparation

	Yes	NA
Equipment drained/depressurized	☐	☒
Equipment purged/cleaned	☐	☒
Isolation checklist completed	☐	☒
Electrical lockout required/field switch tested	☐	☒
Blinds/misalignments/blocks & bleeds in place	☐	☒
Hazardous materials on walls/behind liners considered	☐	☒

XI. Additional Permits required (Hot work, confined space entry). ☐ Yes ☐ No
If yes, complete permit required or contact Health Sciences, Pittsburgh Office

XII. Special instructions, precautions:

Permit Issued by: _____ Permit Accepted by: _____

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1.0 PURPOSE

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

2.0 SCOPE

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

3.0 GLOSSARY

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

Hazardous Waste - Any substance listed in 40 CFR, Subpart D (~~y~~261.30 et seq.), or otherwise characterized as ignitable, corrosive, reactive, or toxic (as defined by Toxicity Characteristic Leaching Procedure, TCLP, analysis) as specified under 40 CFR, Subpart C (~~y~~261.20 et seq.), that would be subject to manifest requirements specified in 40 CFR 262. Such substances are defined and regulated by EPA.

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

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

n.o.s. - Not otherwise specified.

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

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

Common Preservatives:

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

Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - $Na_2S_2O_3$

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

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

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

4.0 RESPONSIBILITIES

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

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

5.0 PROCEDURES

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

5.1 Sample Containers

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

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

5.2 Sample Preservation

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

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

5.2.1 Overview

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

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

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

5.2.2 Preparation and Addition of Reagents

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

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

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

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

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

- Cap sample bottle and seal securely.

Additional considerations are discussed below:

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

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

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

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

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

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

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

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

  Continue with proper acidification of the sample as described above.

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

5.3 Field Filtration

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

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

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

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

5.4 Sample Packaging and Shipping

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

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

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

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

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

5.4.1 Environmental Samples

Environmental samples are packaged as follows:

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

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

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

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

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

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

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

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

6.0 REFERENCES

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

International Air Transport Association (latest issue). Dangerous Goods Regulations, Montreal, Quebec, Canada.

U.S. Department of Transportation (latest issue). Hazardous Materials Regulations, 49 CFR 171-177.

U.S. EPA, 1984. "Guidelines Establishing Test Procedures for the Analysis of Pollutants under Clean Water Act." Federal Register, Volume 49 (209), October 26, 1984, p. 43234.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020, U.S. EPA-EMSL, Cincinnati, Ohio.

ATTACHMENT A

GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

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

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

ATTACHMENT B

ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

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

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

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

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

(1) Polyethylene (P): generally 500 ml or Glass (G): generally 1L.
(2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
(3) When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172).
(4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer periods, and has received a variance from the Regional Administrator.
(5) Should only be used in the presence of residual chlorine.
(6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments are made to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
(7) Samples should be filtered immediately on site before adding preservative for dissolved metals.
(8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
(9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.
(10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
(11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).
(12) If 1,2-diphenylthydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.
(13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
(14) For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
(15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

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1.0 PURPOSE

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

2.0 SCOPE

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

3.0 GLOSSARY

None

4.0 RESPONSIBILITIES

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

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

5.0 PROCEDURES

5.1 Site Logbook

5.1.1 General

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

- All field personnel present
- Arrival/departure of site visitors
- Time and date of H&S training
- Arrival/departure of equipment
- Time and date of equipment calibration
- Start and/or completion of borehole, trench, monitoring well installation, etc.
- Daily onsite activities performed each day
- Sample pickup information
- Health and Safety issues (level of protection observed, etc.)
- Weather conditions

A site logbook shall be maintained for each project. The site logbook shall be initiated at the start of the first onsite activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day

that onsite activities take place which involve Tetra Tech NUS or subcontractor personnel. Upon completion of the fieldwork, the site logbook must become part of the project's central file.

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

- Project name
- Tetra Tech NUS project number
- Sequential book number
- Start date
- End date

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

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

All logbook, notebook, and log sheet entries shall be made in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, the entry shall be crossed out with a single strike mark, and initialed and dated. At the completion of entries by any individual, the logbook pages used must be signed and dated. The site logbook must also be signed by the Field Operations Leader at the end of each day.

5.1.2 Photographs

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

5.2 Field Notebooks

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

5.3 Field Forms

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

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

5.3.1.1 Sample Log Sheet

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

5.3.1.2 Sample Label

A typical sample label is illustrated in Attachment B. Adhesive labels must be completed and applied to every sample container. Sample labels can usually be obtained from the appropriate Program source electronically generated in-house, or are supplied from the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record Form

The Chain-of-Custody (COC) Record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site. One carbonless copy of the completed COC form is retained by the field crew, one copy is sent to the Project Manager (or designee), while the original is sent to the laboratory. The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing vials for VOC analysis or the cooler with the air bill attached. The air bill should then state how many coolers are included with that shipment. An example of a Chain-of-Custody Record form is provided as Attachment C. Once the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed COC form (any discrepancies between the sample labels and COC form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the Tetra Tech NUS Project Manager). The COC form is signed and copied. The laboratory will retain the copy while the original becomes part of the samples' corresponding analytical data package.

5.3.1.4 Chain-of-Custody Seal

Attachment D is an example of a custody seal. The Custody seal is an adhesive-backed label. It is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. The COC seals are signed and dated by the sampler(s) and affixed across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). COC seals may be available from the laboratory; these seals may also be purchased from a supplier.

5.3.1.5 Geochemical Parameters Log Sheets

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

5.3.2 Hydrogeological and Geotechnical Forms

5.3.2.1 Groundwater Level Measurement Sheet

A Groundwater Level Measurement Sheet must be filled out for each round of water level measurements made at a site.

5.3.2.2 Data Sheet for Pumping Test

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

5.3.2.3 Packer Test Report Form

A Packer Test Report Form must be completed for each well upon which a packer test is conducted.

5.3.2.4 Boring Log

During the progress of each boring, a log of the materials encountered, operation and driving of casing, and location of samples must be kept. The Summary Log of Boring, or Boring Log is used for this purpose and must be completed for each soil boring performed. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a PID or FID), these readings must be entered on the boring log at the appropriate depth. The "Remarks" column can be used to subsequently enter the laboratory sample number, the concentration of key analytical results, or other pertinent information. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 Monitoring Well Construction Details Form

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

5.3.2.6 Test Pit Log

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

5.3.2.7 Miscellaneous Monitoring Well Forms

Monitoring Well Materials Certificate of Conformance should be used as the project directs to document all materials utilized during each monitoring well installation.

The Monitoring Well Development Record should be used as the project directs to document all well development activities.

5.3.2.8 Miscellaneous Field Forms - QA and Checklists

Container Sample and Inspection Sheet should be used as the project directs each time a container (drum, tank, etc.) is sampled and/or inspected.

QA Sample Log Sheet should be used at the project directs each time a QA sample is colleted, such as Rinsate Blank, Source Blank, etc.

Field Task Modification Request (FTMR) will be prepared for all deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Copies of all FTMRs will be maintained with the onsite planning documents and originals will be placed in the final evidence file.

The Field Project Daily Activities Check List and Field Project Pre-Mobilization Checklist should be used during both the planning and field effort to assure that all necessary tasks are planned for and completed. These two forms are not a requirement but a useful tool for most field work.

5.3.3 Equipment Calibration and Maintenance Form

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

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

5.4 Field Reports

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

5.4.1 Daily Activities Report

To provide timely oversight of onsite contractors, Daily Activities Reports are completed and submitted as described below.

5.4.1.1 Description

The Daily Activities Report (DAR) documents the activities and progress for each day's field work. This report must be filled out on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring which involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

5.4.1.2 Responsibilities

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

5.4.1.3 Submittal and Approval

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

5.4.2 Weekly Status Reports

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

It should be noted that in addition to summaries described herein, other summary reports may also be contractually required.

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

6.0 LISTING OF TETRA TECH NUS FIELD FORMS FOUND ON THE TTNUS INTRANET SITE. HTTP://INTRANET.TTNUS.COM CLICK ON FIELD LOG SHEETS

Groundwater Sample Log Sheet
Surface Water Sample Log Sheet
Soil/Sediment Sample Log Sheet
Container Sample and Inspection Sheet
Geochemical Parameters (Natural Attenuation)
Groundwater Level Measurement Sheet
Pumping Test Data Sheet
Packer Test Report Form
Boring Log
Monitoring Well Construction Bedrock Flush Mount
Monitoring Well Construction Bedrock Open Hole
Monitoring Well Construction Bedrock Stick Up
Monitoring Well Construction Confining Layer
Monitoring Well Construction Overburden Flush Mount
Monitoring Well Construction Overburden Stick Up
Test Pit Log
Monitoring Well Materials Certificate of Conformance
Monitoring Well Development Record

Daily Activities Record
Field Task Modification Request
Hydraulic Conductivity Test Data Sheet
Low Flow Purge Data Sheet
QA Sample Log Sheet
Equipment Calibration Log
Field Project Daily Activities Checklist
Field Project Pre-Mobilization Checklist

ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: _____ DATE: _____

SITE LEADER: _____

PERSONNEL:

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

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

ACTIVITIES:

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

_____

Field Operations Leader

ATTACHMENT B

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

ATTACHMENT C

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

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

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

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

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

COMMENTS

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

4/02R
FORM NO. TtNUS-001

ATTACHMENT D

CHAIN-OF-CUSTODY SEAL

CUSTODY SEAL	**CUSTODY SEAL**
Date	**Date**
Signature	**Signature**

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1.0 PURPOSE

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

2.0 SCOPE AND APPLICABILITY

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

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

3.0 GLOSSARY

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

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

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

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

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

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

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

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

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

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

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

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

General personnel qualifications for decontamination activities include the following:

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

5.0 HEALTH AND SAFETY

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

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

6.0 EQUIPMENT LIST

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

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

7.0 PROCEDURES

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

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

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

- Combination of some or all of the above

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

7.1 Decontamination Pad Design/Construction Considerations

7.1.1 Temporary Decontamination Pads

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

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

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

  - Well removed from pedestrian/vehicle thoroughfares.

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

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

  - Avoidance of potentially contaminated areas.

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

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

  - Areas where potable water and electricity are provided.

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

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

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

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

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

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

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

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

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

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

- Maintenance – Maintain the decontamination area by:

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

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

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

7.1.2 Decontamination Activities at Drill Rigs/DPT Units

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

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

7.1.3 Decontamination Activities at Remote Sample Locations

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

7.2 Equipment Decontamination Procedures

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

7.2.1 Monitoring Well Sampling Equipment

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

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

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

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

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

5. Rinse external pump components using tap water.

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

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

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

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

9. Drain residual deionized water to the extent possible.

10. Allow components of the equipment to air dry.

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

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

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

7.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

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

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

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

7.2.1.3 Miscellaneous Equipment

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

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

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

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

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

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

7.2.2 Downhole Drilling Equipment

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

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

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

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

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

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

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

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

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

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

SAFETY REMINDER

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

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

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

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

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

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

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

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

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

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

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

7.2.3 Soil/Sediment Sampling Equipment

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

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

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

3. Rinse the equipment with tap water.

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

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

5. Rinse the equipment with deionized water.

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

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

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

Dredges employed in sediment sampling are typically decontaminated as follows:

- Remove the sediment sample from the sampling device

- If sufficient associated surface water is available at the sampling site, place the dredge in the water and flush to remove visible sediment.

- Extract the dredge and wash it in soap and water per the project-specific planning documents.

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

7.3 Contact Waste/Materials

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

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

7.3.1 Investigation-Derived Wastes - Decontamination Wash Waters and Sediments

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

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

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

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

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

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

   - Enclose areas accessible by the general public using construction fencing and signs.

   - Stored materials in 55-gallon drums on pallets with four (or fewer) drums per pallet.

   - Maintain the retaining bolt and label on the outside of storage containers where readily visible.

   - Provide at least 4 feet of room between each row of pallets to allow access to containers for sampling, drum removal, and spill response.

   - As directed in project-specific planning documents, maintain an IDW Inventory List and provide the list to the site Point of Contact at the termination of each shift.

   - Maintain spill response equipment at the IDW storage area in case it is required for immediate access.

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

CAUTION

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

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

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

7.4 Decontamination Evaluation

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

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

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

NOTE

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

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

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

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

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

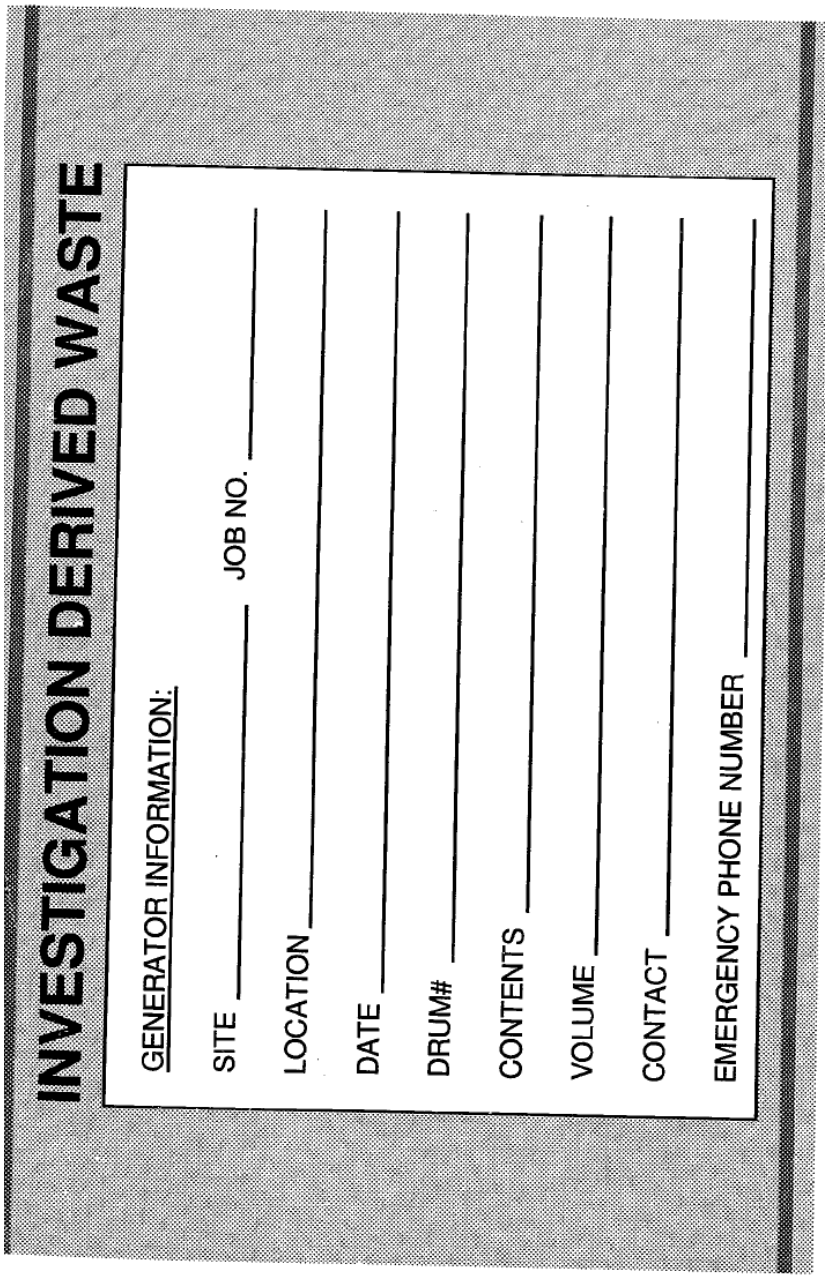
    - Per decontamination method
    - Per disposable article/batch number of disposable articles

NOTE

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

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

Attachment A
DW Label



APPENDIX B

FIELD DOCUMENTATION FORMS

Tetra Tech NUS, Inc.

BORING LOG

PROJECT NAME: _____ BORING No.: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION			USCS *	Remarks	PID/FID Reading (ppm)			
					Soil Density/ Consistency or Rock Hardness	Color	Material Classification			Sample	Sampler BZ	Borehole**	Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated reponse read.

Drilling Area Background (ppm): _____

Remarks: _____

Converted to Well: Yes _____ No _____ Well I.D. #: _____

Tetra Tech NUS, Inc.

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

DAILY ACTIVITIES CHECKLIST

Startup Checklist

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

Exit Checklist

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

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

TETRA TECH NUS, INC.

FIELD INSTRUMENT CALIBRATION LOG

INSTRUMENT NAME: _____ MODEL No.: _____

SERIAL No.: _____ DECAL No.: _____ TETRA TECH NUS CHARGE No. _____

CALIBRATION DATE	INITIAL READING	PROCEDURE	FINAL READING	SIGNATURE	COMMENTS

TtNUS Form 0007

TETRA TECH NUS, INC.

FIELD MODIFICATION RECORD

Site Name: _____ Location: _____

Project Number: _____ Task Assignment: _____

To: _____ Location: _____ Date: _____

Description:_____

_____

_____

_____

Reason for Change: _____

_____

_____

Recommended Action: _____

_____

_____

Field Operations Leader (Signature): _____ Date: _____

Disposition/Action:_____

_____

_____

Project Manager (Signature): _____ Date: _____

Distribution:

		Others as Required:
Program Manager:	_____	
Project Manager:	_____	_____
Quality Assurance Officer:	_____	_____
Field Operations Leader:	_____	_____
Project File:	_____	_____

TtNUS Form 0003

Tetra Tech NUS, Inc.

GROUNDWATER LEVEL MEASUREMENT SHEET

Project Name: _____ **Project No.:** _____

Location: _____ **Personnel:** _____

Weather Conditions: _____ **Measuring Device:** _____

Tidally Influenced: Yes ____ No ____ **Remarks:** _____

Well or Piezometer Number	Date	Time	Elevation of Reference Point (feet)*	Total Well Depth (feet)*	Water Level Indicator Reading (feet)*	Thickness of Free Product (feet)*	Groundwater Elevation (feet)*	Comments

* All measurements to the nearest 0.01 foot

Tetra Tech NUS, Inc.

GROUNDWATER SAMPLE LOG SHEET

Project Site Name: _____
Project No.: _____

[] Domestic Well Data
[] Monitoring Well Data
[] Other Well Type: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____
Type of Sample:
[] Low Concentration
[] High Concentration

SAMPLING DATA:

Date: / Time:	Color (Visual)	pH (S.U.)	S.C. (mS/cm)	Temp. (^{0}C)	Turbidity (NTU)	DO (mg/l)	Salinity (%)	Other
Method:								

PURGE DATA:

Date:	Volume	pH	S.C.	Temp.	Turbidity	DO	Salinity	Other
Method:								
Monitor Reading (ppm):								
Well Casing Diameter & Material								
Type:								
Total Well Depth (TD):								
Static Water Level (WL):								
One Casing Volume(gal/L):								
Start Purge (hrs):								
End Purge (hrs):								
Total Purge Time (min):								
Total Vol. Purged (gal/L):								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

Tetra Tech NUS, Inc.

MONITORING WELL DEVELOPMENT RECORD

Site: _____ Depth to Bottom (ft.): _____ Project Name: _____
Well: _____ Static Water Level Before (ft.): _____ Project Number: _____
Date Installed: _____ Static Water Level After (ft.): _____ Site Geologist: _____
Date Developed: _____ Screen Length (ft.): _____ Drilling Co.: _____
Dev. Method: _____ Specific Capacity: _____
Pump Type: _____ Casing ID (in.): _____

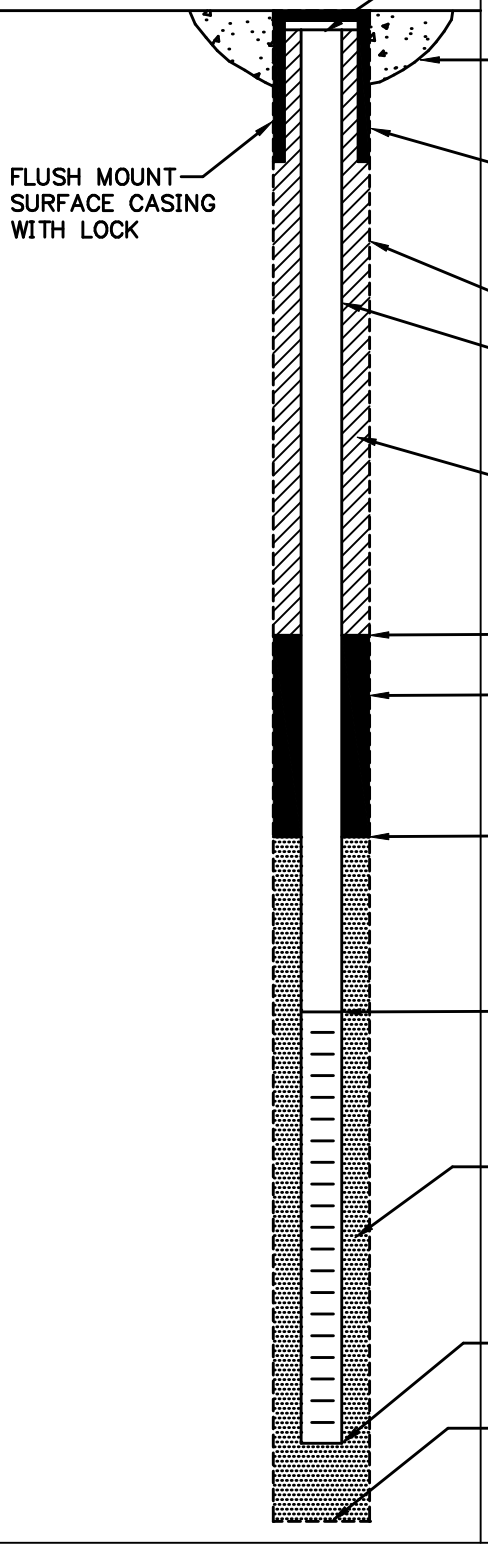
Time	Estimated Sediment Thickness (Ft.)	Cumulative Water Volume (Gal.)	Water Level Readings (Ft. below TOC)	Temperature (Degrees C)	pH	Specific Conductance (Units _____)	Turbidity (NTU)	Remarks (odor, color, etc.)

WELL NO.: _____

Tetra Tech NUS, Inc.

OVERBURDEN
MONITORING WELL SHEET
FLUSH - MOUNT

PROJECT _____	LOCATION _____	DRILLER _____
PROJECT NO. _____	BORING _____	DRILLING METHOD _____
DATE BEGUN _____	DATE COMPLETED _____	
FIELD GEOLOGIST _____		DEVELOPMENT METHOD _____
GROUND ELEVATION _____	DATUM _____	



ELEVATION TOP OF RISER: _____

TYPE OF SURFACE SEAL: _____

TYPE OF PROTECTIVE CASING: _____

I.D. OF PROTECTIVE CASING: _____

DIAMETER OF HOLE: _____

TYPE OF RISER PIPE: _____

RISER PIPE I.D.: _____

TYPE OF BACKFILL/SEAL: _____

ELEVATION/DEPTH TOP OF SEAL: ____/____

TYPE OF SEAL: _____

ELEVATION/DEPTH TOP OF SAND: ____/____

ELEVATION/DEPTH TOP OF SCREEN: ____/____

TYPE OF SCREEN: _____

SLOT SIZE x LENGTH: _____

TYPE OF SAND PACK: _____

DIAMETER OF HOLE IN BEDROCK: _____

ELEVATION / DEPTH BOTTOM OF SCREEN: ____/____

ELEVATION / DEPTH BOTTOM OF SAND: ____/____

ELEVATION/DEPTH BOTTOM OF HOLE: ____/____

BACKFILL MATERIAL BELOW SAND: _____

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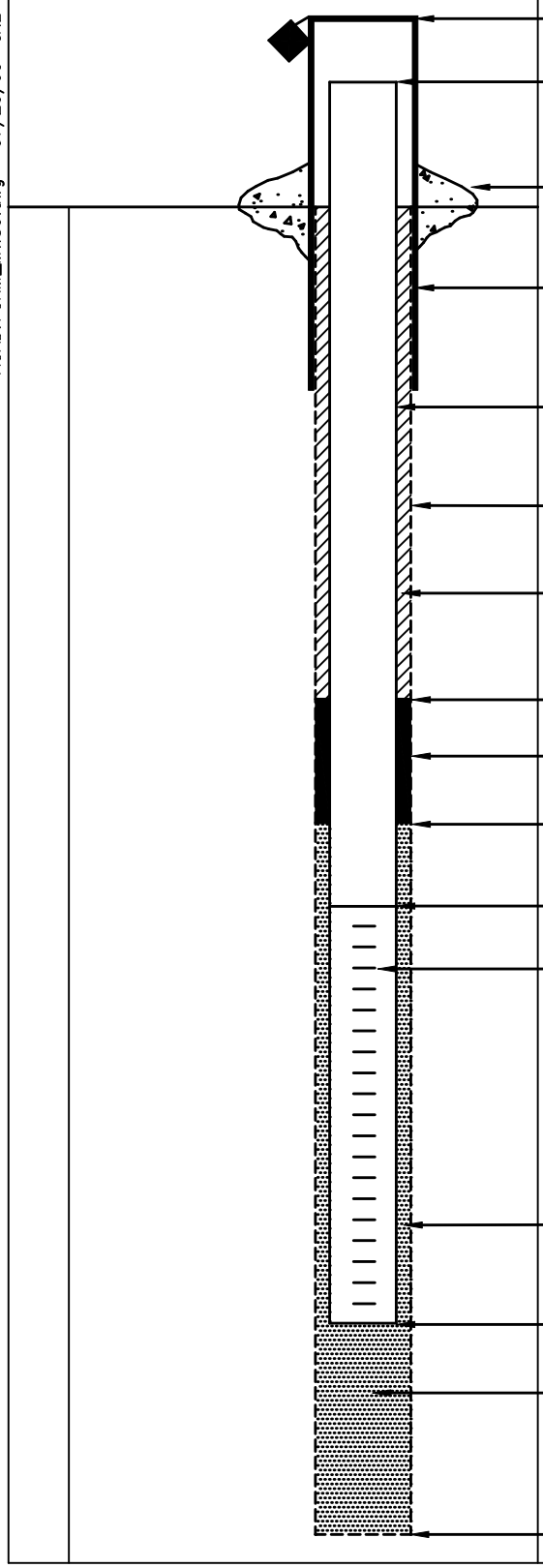
WELL NO.: _____

Tetra Tech NUS, Inc.

OVERBURDEN MONITORING WELL SHEET STICK-UP

PROJECT _____	LOCATION _____	DRILLER _____
PROJECT NO. _____	BORING _____	DRILLING METHOD _____
DATE BEGUN _____	DATE COMPLETED _____	
FIELD GEOLOGIST _____		DEVELOPMENT METHOD _____
GROUND ELEVATION _____	DATUM _____	



ELEVATION/HEIGHT OF TOP OF SURFACE CASING: ____/____

ELEVATION/HEIGHT OF TOP OF RISER PIPE: ____/____

TYPE OF SURFACE SEAL: _____

I.D. OF SURFACE CASING: _____
TYPE OF SURFACE CASING: _____

RISER PIPE I.D.: _____
TYPE OF RISER PIPE: _____

BOREHOLE DIAMETER: _____

TYPE OF BACKFILL: _____

ELEVATION/DEPTH TOP OF SEAL: ____/____

TYPE OF SEAL: _____

DEPTH TOP OF SAND PACK: _____

ELEVATION/DEPTH TOP OF SCREEN: ____/____

TYPE OF SCREEN: _____

SLOT SIZE x LENGTH: _____

I.D. OF SCREEN: _____

TYPE OF SAND PACK: _____

ELEVATION/DEPTH BOTTOM OF SCREEN: ____/____

ELEVATION/DEPTH BOTTOM OF SAND PACK: ____/____
BACKFILL MATERIAL BELOW SAND: _____

ELEVATION/DEPTH OF HOLE: ____/____

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TETRA TECH NUS, INC.

SAMPLE COLLECTION SUMMARY RECORD

PROJECT NAME: _____ TETRA TECH NUS CHARGE NUMBER: _____

SAMPLING EVENT: _____ CASE NO.: _____ DAS NO.: _____

DATE	TIME	SAMPLE LOCATION	FIELD QC							COMMENTS

TtNUS Form 0012

Tetra Tech NUS, Inc.

QA SAMPLE LOG SHEET

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

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

SAMPLING DATA:

Date: _____
Time: _____
Method: _____

WATER SOURCE:

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

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

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

RINSATE INFORMATION (If Applicable):

Media Type: _____
Equipment Used: _____
Equipment Type:

[] Dedicated
[] Reusable

SAMPLE COLLECTION INFORMATION:

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

OBSERVATIONS / NOTES:

Signature(s):

TETRA TECH NUS, INC.

SITE ENTRY LOG

Site Name: _____ Date: _____

Location: _____ Project Number: _____

NAME	REPRESENTING	TIME IN (HOURS)	TIME OUT (HOURS)	INITIALS

TtNUS Form 0002

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Project Site Name: _____
Project No.: _____

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

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

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

GRAB SAMPLE DATA:

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

COMPOSITE SAMPLE DATA:

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

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

Tetra Tech NUS, Inc.

SURFACE WATER SAMPLE LOG SHEET

Project Site Name: _____
Project No.: _____

[] Stream
[] Spring
[] Pond
[] Lake
[] Other: _____
[] QA Sample Type: _____

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

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

SAMPLING DATA:

Date:	Color	pH	S.C.	Temp.	Turbidity	DO	Salinity	Other
Time:	(Visual)	(S.U.)	(mS/cm)	(^{0}C)	(NTU)	(mg/l)	(%)	
Depth:								
Method:								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

APPENDIX C

ANALTYICAL SPECIFICATION

ATTACHMENT NO. 2

STATEMENT OF WORK/PRICE TABLES

MODIFICATION OF TECHNICAL SPECIFICATION FOR LABORATORY SERVICES
BACKGROUND STUDY NAVAL AIR STATION BRUNSWICK
BRUNSWICK, MAINE

COMPREHENSIVE LONG-TERM ENVIRONMENTAL ACTION - NAVY (CLEAN)
CONTRACT N62467-04-D-0055, CONTRACT TASK ORDER (CTO) No. 432

1.0 INTRODUCTION

Tetra Tech NUS, Inc. (TtNUS) under CLEAN Contract N62467-04-D-0055, is modifying this statement of work for laboratory analytical services to support a background study at NAS Brunswick. Requested analyses include polycyclic aromatic hydrocarbons (PAHs), pesticides, herbicides, metals, chloride and bromide, total organic carbon (TOC), and grain size analysis.

The laboratory performing these analyses must be certified by the State of Maine.

The laboratory may be required to submit updates for method detection limits (MDLs), Standard Operating Procedures (SOPs), and relevant precision and accuracy limits for all preparation and analytical methods required under this scope of work after the subcontract has been awarded.

2.0 SAMPLE INFORMATION

The approximate number of samples to be submitted for each sampling event, the type of analyses to be conducted, and the analytical methods to be used are summarized in Table 1. This background study includes analysis of soil, sediment, groundwater, and surface water.

The first round of sampling is expected to be conducted in August of 2009 to collect samples for all of the media. A second round of groundwater, sediment, and surface water sampling is anticipated for late in 2009 or in 2010. The exact date of sample collection will be communicated to the laboratory a few weeks in advance.

All samples are expected to be of low or moderate contaminant concentration since this is a background study.

Field duplicate samples will be submitted to the laboratory with "blinded" identification. The field crew will designate samples (one per twenty samples of like matrices) for matrix spike/matrix spike duplicate (MS/MSD) (organic, chloride, and bromide analyses) and matrix spike/laboratory duplicate (metal analysis); additional volumes of these samples will be provided as necessary.

Sediment Samples

For sediment sample chemical analyses (PAH, pesticide, herbicide, TOC, and metals) the laboratory must decant any standing water, homogenize the sample, and determine the percent moisture before sample analysis. The laboratory must centrifuge the sample to remove the excess of water or they must increase the sample aliquot to compensate for the high moisture. The project quantitation limits must be met regardless of the moisture content of the sediment samples.

If the percent moisture content is too high and the sediment samples contain noticeable organic material, further dewatering must be performed at the laboratory prior to sample analysis. This could be accomplished by freeze-drying under controlled conditions proven to recover the analysis-specific surrogates; centrifugation and decanting free water; low temperature oven drying (below 60°C); or other procedure proposed by the laboratory and approved by TtNUS.

Metal Analysis

For the ICP and mercury metal analyses, perform a matrix spike and a laboratory duplicate analysis instead of matrix spike/matrix spike duplicate. If the matrix spike recovery falls outside the control limits and the sample result does not exceed four times the spike added, a post-digestion spike analysis must be performed. In addition, a five-fold dilution analysis (dilution test) must be performed on the field-designated QC sample. Results for these QC analyses must be summarized in the corresponding CLP-equivalent forms (Attachment B).

GC/ECD Analyses

Positive identifications for pesticides and herbicides by gas chromatography (GC) with electron capture detector (ECD) MUST be confirmed on a second column that possesses retention characteristics different from those exhibited by the primary column. Identification using a single column with dual detectors does not meet second column confirmation requirements. Confirmed positive results less than the laboratory quantitation limit (QL) but greater than the method detection limit (MDL) must be reported by the laboratory; the laboratory must "J" flag these results. The higher of two results generated from dual column analyses must be reported as the primary result by the laboratory unless relative percent differences (RPDs) between columns are greater than 40% and interference is found to be present or there is some other justifiable reason to select the lower of the two results.The laboratory must narrate the reason for selection of the lower result..

3.0 ANALYSIS/REPORTING INFORMATION

One hard copy data package deliverable and two PDF CD copies must be submitted, in addition to the electronic data deliverables to be provided in the format described in Modified Attachment C (see accompanying Word file; original Attachment C has been deleted). The original chain-of-custody form received with the samples and signed by the laboratory sample custodian must be returned with the hard copy data package.

The analytical requirements and approximate number of samples to be submitted for analysis are detailed in Table 1. Analysis and reporting requirements addressed in the DOD Quality Systems Manual (January 2006 or April 2009 depending on current NFESC approval letter) and the requested methods must be followed. The laboratory-derived recovery limits for matrix spike, LCSs, and surrogates must be met. Additionally, it is a requirement of TtNUS that the associated PDF and hard copy data packages for PAH, pesticide, and metals analyses must meet Contract Laboratory Program (CLP) format, reporting, and PDF/hard copy data package deliverable requirements. The PDF and hard copy data packages for the remaining analyses must be in a CLP-modified format and must include the appropriate summary forms and raw data for all samples and laboratory quality control samples. The summary forms should include the method-specific quality control limits (recoveries, relative percent differences, relative standard deviations, and/or percent differences, etc.).

Additionally, each hard copy and PDF data package must contain a summary data package. This summary data package shall consist of only the summary forms (i.e., for CLP, Forms 1 through 15; for non-CLP it shall be the CLP-like equivalent of Forms 1 through 15). Attachment B details the required summary forms for CLP-like data packages and requirements for organization/bookmarking of hard copy/PDF data packages.

Modified Attachment A details the required target compound list and required project quantitation limit (PQL) goals that must be met (see accompanying Word file; original Attachment A has been deleted).

Non-detected organic and inorganic results must be reported down to the laboratory MDLs. Positive results above the MDL but below the laboratory's QL must be reported as estimated values qualified with a "J" for both organic and inorganic (metals, chloride, and bromide) analyses. Soil and sediment samples must be reported on a dry-weight basis.

The hard copy/PDF data package deliverable must contain a detailed case narrative for all analytical fractions. This case narrative must also include the Contract Task Order (CTO) number, the site name, and the TtNUS Project Manager's name. Data from all analytical runs (i.e., original, dilution, re-analysis) must be reported in the raw data and Form Is for organic analyses. For metals analysis, only the final sample results should be reported in the Form Is, and data from all analytical runs must be included in the raw data.

As part of the laboratory case narrative, it is required that the Laboratory Quality Assurance Manager sign an attestation statement verifying that all electronic diskette deliverables exactly match the data summary forms (i.e. Form Is).

As stipulated in the CLEAN Master Services Agreement (MSA), Sample Delivery Group (SDG) and fractionally-specific text (TXT) files containing all environmental sample and field quality control blank analysis results must be generated in accordance with the requirements outlined in Modified Attachment C of this specification.

Maximum holding time allowances, as defined in the following table, are to be strictly observed. Calculation of holding time is in calendar days and is to begin from the time of sample collection. The holding times are as follows:

Analyses	**Holding Time**
PAHs, Pesticides, Herbicides	7 days to extraction (aqueous); 14 days to extraction (soil/sediment); 40 days to analysis
Metals	6 months to analysis for ICP metals 28 days for mercury
TOC	28 days
Chloride and Bromide	28 days
Grain Size	Not applicable

These holding times are based on 40 CFR 136, data validation criteria, and method specific requirements, and are measured from date of collection for samples preserved as requested in the analytical methods. The holding time criteria depicted apply to all analyses necessary to successfully determine the contaminant level contained in the sample. Hence, **the holding time criteria apply to any/all subsequent sample dilutions and re-analyses**.

The TtNUS Project Manager for this project is Mr. Jeff Orient and he must be contacted in the event of any laboratory problems that could impact project deadlines (i.e., late deliverables, technical problems in the lab that could lead to late deliverables.) To insure good communication it is required that the laboratory's appointed project manager contact Mr. Orient once a week for the entire project duration.

Contact information for Mr. Orient is as follows:

Tetra Tech NUS
661 Andersen Drive, Foster Plaza 7
Pittsburgh, PA 15220
Phone: 412-921-8778
Fax: 412-921-4040
Email: jeff.orient@tetratech.com

Analytical data turnaround times are to be measured from receipt of each sample shipment. All hard copy/PDF (2 CDs) analytical data packages and associated electronic (TXT) deliverables are due within the standard MSA turnaround term of 21 calendar days from receipt of the last sample in a Sample Delivery Group (SDG).

The SDGs must contain up to 20 samples. The frequency with which SDGs contain less than 20 samples should be minimal. All hard copy/PDF data packages and electronic deliverables must be received at the same time or the deliverable will be considered incomplete and payment deductions may be imposed.

The hard copy/PDF (2 CDs) analytical data package and the original chain-of-custody form (received with the samples and signed by the laboratory sample custodian) should be sent to Ms. Lucy Guzman. Contact information for Ms. Guzman is as follows:

Tetra Tech NUS
55 Jonspin Road
Wilmington, MA 01887
Phone: 978-474-8416
Fax: 978-474-8499
Email: lucy.guzman@tetratech.com

The electronic (TXT) deliverables, in addition to a copy of the chain-of-custody form, should be sent to Ms. Tobrena Skeen. Contact information for Ms. Skeen is as follows:

Tetra Tech NUS, Inc.
661 Andersen Drive, Foster Plaza 7
Pittsburgh, PA 15220-2745
Phone: 412-921-8582
Fax: 412-921-4040
e-mail: tobrena.skeen@tetratech.com

4.0 PERIOD OF PERFORMANCE/BOTTLEWARE INFORMATION

All samples will be shipped to the laboratory via express carrier within 48 hours of collection. Please circle the Yes or No at the bottom of Table 1 which will indicate if the laboratory will provide courier service at no extra charge. The laboratory will be notified at least 2 weeks prior to sample collection.

Bottleware shipments will be coordinated by the field operation leader.

The laboratory is to provide all necessary sample containers **(plus approximately 10% extra for breakage)**. All sample containers must meet ICHEM series 300 cleanliness criteria (or equivalent), and documentation of certified cleanliness must be provided. All of the appropriate sample bottleware must be pre-preserved. The bottleware must be shipped to the designated location in Coleman-like coolers. Each cooler must include a "temperature blank" vial. **The laboratory must also provide any extra coolers needed for return shipment of samples to the laboratory for analysis.** The laboratory is also requested to provide a packing slip indicating the analytical parameters for which each container type is designated, sample labels, and chain-of-custody forms.

The laboratory must provide Material Safety Data Sheets (MSDSs) for all preservatives sent with each bottleware shipment to the field. MSDSs must be representative of the chemicals provided as preservatives with regard to mixtures and/or purity of the chemicals. For example if a 35% sulfuric acid solution is the preservative, the MSDS provided should be for 35% sulfuric acid solution not 96% sulfuric acid.

5.0 ADDITIONAL COMMENTS/CONTACTS

The internal transfers of samples, extracts, and digestates within the laboratory, must be accomplished and documented as controlled custody transfers. The laboratory must provide documentation that supports an unbroken chain of custody for samples, digestates and extracts from time of receipt or production in the laboratory until disposal.

The laboratory is to provide a minimum of 60 days storage of sample extracts and 60 intact leftover samples, as stipulated in the MSA. **Additionally, the laboratory must store PDF data packages for 5 years.**

All analyses conducted under this subcontract assignment are to be performed at the solicited facility only. The laboratory is not permitted to lower-tier subcontract these analyses, or analyze these samples at a corporate facility other than the facility stipulated without prior notification and consent from the CLEAN Subcontracting Officer.

The unit cost for analysis is to include compensation for containers, preservatives, coolers, shipping costs, storage, disposal, and laboratory quality control analyses (such as matrix spike, matrix spike duplicate, laboratory duplicate, and laboratory control sample analyses.) These items are not to be billed as separate line items.

Technical, quality assurance, and data format concerns are to be directed to Ms. Lucy Guzman at 978-474-8416 or via e-mail lucy.guzman@tetratech.com. **Ms. Guzman must be contacted and informed of any difficulties encountered during the conduct of the requested analyses.**

Contract concerns, and response to this solicitation, are to be directed to:

Ms. Meg Price
CLEAN Subcontracting Officer
Tetra Tech NUS, Inc.
234 Mall Boulevard, Suite 260
King of Prussia, PA 19406
Phone: 610-491-9688
Fax: 610-491-9645

e-mail: meg.price@tetratech.com

Triplicate copies of invoices associated with the analyses contracted herein are to be submitted to the attention of the Accounting Supervisor:

Tetra Tech NUS, Inc.
661 Andersen Drive, Foster Plaza 7
Pittsburgh, PA 15220
Phone: 412-921-8506
Fax: 412-921-4040

Please confirm the laboratory's ability to perform the methodologies requested at the analyte quantitation limits indicated. Also confirm available laboratory capacity, and complete/confirm the costing information indicated in Table 1. All costing information must reflect the terms and conditions established by the 2009 CLEAN MSA.

TABLE 1
NUMBER OF SAMPLES/ANALYTICAL METHODS
BACKGROUND STUDY, NAVAL AIR STATION BRUNSWICK
BRUNSWICK, MAINE

Matrix	Parameter	Method	# Samples	Unit Price	Total Cost
Soil (Sampling Event 1 only)					
	PAHs	3550B/8270 SIM	66	$	$
	TCL Pesticides	3550B/8081A	33	$	$
	Herbicides	8151A	33	$	$
	TOC	9060A	66	$	$
	TAL Metals	3050/6010B/6020/7471A	66	$	$
Sediment – Sampling Event 1					
	PAHs	3540C/8270 SIM	18	$	$
	TCL Pesticides	3540C/8081A	18	$	$
	Herbicides	8151A	18	$	$
	TOC	9060A	18	$	$
	TAL Metals	3050/6010B/6020/7471A	18	$	$
	Grain Size (sieve analysis only)	ASTM D422-63	18	$	$
Sediment – Sampling Event 2	PAHs	3540C/8270 SIM	18	$	$
	TCL Pesticides	3540C/8081A	18	$	$
	Herbicides	8151A	18	$	$
	TOC	9060A	18	$	$
	TAL Metals	3050/6010B/6020/7471A	18	$	$
	Grain Size (sieve analysis only)	ASTM D422-63	18	$	$
Groundwater – Sampling Event 1					
	TAL Metals (Total)	3010/6010B/6020/7470A	58	$	$
	TAL Metals (Dissolved*)	3010/6010B/6020/7470A	57	$	$

Matrix	Parameter	Method	# Samples	Unit Price	Total Cost
	Chloride and Bromide	EPA Method 300.0	58	$	$
Groundwater – Sampling Event 2					
	TAL Metals (Total)	3010/6010B/6020/7470A	58	$	$
	TAL Metals (Dissolved*)	3010/6010B/6020/7470A	57	$	$
Surface Water – Sampling Event 1					
	PAHs	3520C/8270 SIM	20	$	$
	TAL Metals (Total)	3010/6010B/6020/7470A	20	$	$
	TAL Metals (Dissolved*)	3010/6010B/6020/7470A	19	$	$
Surface Water – Sampling Event 2	PAHs	3520C/8270 SIM	20	$	$
	TAL Metals (Total)	3010/6010B/6020/7470A	20	$	$
	TAL Metals (Dissolved*)	3010/6010B/6020/7470A	19	$	$
Aqueous Rinsate and Field Blanks associated with soil and sediment samples – Sampling Event 1					
	PAHs	3520C/8270 SIM	7	$	$
	TCL Pesticides	3520C/8081A	5	$	$
	Herbicides	8151A	5	$	$
	TAL Metals	3010/6010B/6020/7470A	7	$	$
Aqueous Rinsate and Field Blanks	PAHs	3520C/8270 SIM	2	$	$
	TCL Pesticides	3520C/8081A	2	$	$

Matrix	Parameter	Method	# Samples	Unit Price	Total Cost
associated with soil and sediment samples – Sampling Event 2	Herbicides	8151A	2	$	$
	TAL Metals	3010/6010B/6020/7470A	2	$	$

* Dissolved metals samples will be filtered in the field.

TOTAL $

Can the laboratory provide sample pick-up on site? YES or NO (circle one)
If yes is there a charge and what is that charge?_____

Name of Laboratory_____

Signature_____

MODIFIED ATTACHMENT A
PROJECT REQUIRED QUANTITATION LIMITS
(See accompanying Word file)

SAP Worksheet #15a -- Reference Limits and Evaluation Table, Soil Samples

(UFP-QAPP Manual Section 2.8.1)

Matrix: Soil

Analyte	CAS Number	Project Action Limit (mg/Kg)	Analytical Method	PQL Goal (mg/Kg)	Laboratory-specific	
					QLs (mg/Kg)	MDLs (mg/Kg)
PAHs						
2-Methylnaphthalene	91-57-6	29	SW846 8270C SIM	0.33		
Acenaphthene	83-32-9	29	SW846 8270C SIM	0.33		
Acenaphthylene	208-96-8	29	SW846 8270C SIM	0.33		
Anthracene	120-12-7	29	SW846 8270C SIM	0.33		
Benzo(a)anthracene	56-55-3	0.15	SW846 8270C SIM	0.020		
Benzo(a)pyrene	50-32-8	0.015	SW846 8270C SIM	0.020		
Benzo(b)fluoranthene	205-99-2	0.15	SW846 8270C SIM	0.020		
Benzo(g,h,i)perylene	191-24-2	1.1	SW846 8270C SIM	0.33		
Benzo(k)fluoranthene	207-08-9	1.1	SW846 8270C SIM	0.33		
Chrysene	218-01-9	1.1	SW846 8270C SIM	0.33		
Dibenzo(a,h)anthracene	53-70-3	0.015	SW846 8270C SIM	0.020		
Fluoranthene	206-44-0	29	SW846 8270C SIM	0.33		
Fluorene	86-73-7	29	SW846 8270C SIM	0.33		
Indeno(1,2,3-c,d)pyrene	193-39-5	0.15	SW846 8270C SIM	0.020		

Analyte	CAS Number	Project Action Limit (mg/Kg)	Analytical Method	PQL Goal (mg/Kg)	Laboratory-specific	
					QLs (mg/Kg)	MDLs (mg/Kg)
Naphthalene	91-20-3	3.9	SW846 8270C SIM	0.33		
Phenanthrene	85-01-8	29	SW846 8270C SIM	0.33		
Pyrene	129-00-0	1.1	SW846 8270C SIM	0.33		
Pesticides						
4,4' DDD	72-54-8	0.021	SW846 8081A	0.0033		
4,4'-DDE	72-55-9	0.021	SW846 8081A	0.0033		
4,4'-DDT	50-29-3	0.021	SW846 8081A	0.0033		
Aldrin	309-00-2	0.00006	SW846 8081A	0.0017		
alpha-BHC	319-84-6	0.00072	SW846 8081A	0.0017		
alpha-Chlordane	5103-71-9	0.00003	SW846 8081A	0.0017		
beta-BHC	319-85-7	0.0026	SW846 8081A	0.0017		
delta-BHC	319-86-8	0.00005	SW846 8081A	0.0017		
Dieldrin	60-57-1	0.004	SW846 8081A	0.0033		
Endosulfan I	959-98-8	0.0001	SW846 8081A	0.0017		
Endosulfan II	33213-65-9	0.0001	SW846 8081A	0.0033		
Endosulfan sulfate	1031-07-8	0.0001	SW846 8081A	0.0033		
Endrin	72-20-8	0.00004	SW846 8081A	0.0033		
Endrin aldehyde	7421-93-4	0.00004	SW846 8081A	0.0033		
Endrin ketone	53494-70-5	0.00004	SW846 8081A	0.0033		
gamma-BHC (Lindane)	58-89-9	0.00005	SW846 8081A	0.0017		
gamma-Chlordane	5103-74-2	0.00003	SW846 8081A	0.0017		

Analyte	CAS Number	Project Action Limit (mg/Kg)	Analytical Method	PQL Goal (mg/Kg)	Laboratory-specific	
					QLs (mg/Kg)	MDLs (mg/Kg)
Heptachlor	76-44-8	0.0007	SW846 8081A	0.0017		
Heptachlor epoxide	1024-57-3	0.0000002	SW846 8081A	0.0017		
Methoxychlor	72-43-5	31	SW846 8081A	0.017		
Toxaphene	8001-35-2	0.44	SW846 8081A	0.17		
Herbicides						
2,4-D	94-75-7	1.5	SW846 8151A	0.033		
2,4-DB	94-82-6	36	SW846 8151A	0.033		
2,4,5-TP (Silvex)	93-72-1	20	SW846 8151A	0.033		
2,4,5-T	93-76-5	11	SW846 8151A	0.033		
Dalapon	75-99-0	180	SW846 8151A	0.170		
Dicamba	1918-00-9	13	SW846 8151A	0.033		
Dichloroprop	120-36-5	--	SW846 8151A	0.067		
Dinoseb	88-85-7	0.37	SW846 8151A	0.170		
MCPA	94-74-6	3.1	SW846 8151A	5		
MCPP	93-65-2	6.1	SW846 8151A	3.3		
Metals						
Aluminum	7429-90-5	170	SW846 6010B	30		
Antimony	7440-36-0	0.27	SW846 6020	0.1		
Arsenic	7440-38-2	0.39	SW846 6020	0.5		
Barium	7440-39-3	330	SW846 6010B	10		
Beryllium	7440-41-7	4	SW846 6010B	0.5		

Analyte	CAS Number	Project Action Limit (mg/Kg)	Analytical Method	PQL Goal (mg/Kg)	Laboratory-specific	
					QLs (mg/Kg)	MDLs (mg/Kg)
Cadmium	7440-43-9	0.36	SW846 6020	0.25		
Calcium	7440-70-2	--	SW846 6010B	500		
Chromium	7440-47-3	23	SW846 6010B	4		
Cobalt	7440-48-4	3.3	SW846 6020	2.5		
Copper	7440-50-8	28	SW846 6010B	2.5		
Iron	7439-89-6	5500	SW846 6010B	10		
Lead	7439-92-1	11	SW846 6010B	1		
Magnesium	7439-95-4	--	SW846 6010B	500		
Manganese	7439-96-5	180	SW846 6010B	5		
Mercury	7439-97-6	0.1	SW846 7471A	0.04		
Nickel	7440-02-0	38	SW846 6010B	4		
Potassium	7440-09-7	--	SW846 6010B	500		
Selenium	7782-49-2	0.52	SW846 6020	0.5		
Silver	7440-22-4	4.2	SW846 6010B	1.5		
Sodium	7440-23-5	--	SW846 6010B	500		
Thallium	7440-28-0	0.51	SW846 6020	0.2		
Vanadium	7440-62-2	7.8	SW846 6010B	2.5		
Zinc	7440-66-6	46	SW846 6010B	6		
TOC	--	--	SW846 9060A	400		

SAP Worksheet #15b -- Reference Limits and Evaluation Table, Sediment Samples

(UFP-QAPP Manual Section 2.8.1)

Matrix: Sediment

Analyte	CAS Number	Project Action Limit (mg/Kg)	Analytical Method	PQL Goal (mg/Kg)	Laboratory-specific	
					QLs (mg/Kg)	MDLs (mg/Kg)
PAHs						
2-Methylnaphthalene	91-57-6	0.02	SW846 8270C SIM	0.020		
Acenaphthene	83-32-9	0.29	SW846 8270C SIM	0.020		
Acenaphthylene	208-96-8	0.16	SW846 8270C SIM	0.020		
Anthracene	120-12-7	0.0572	SW846 8270C SIM	0.020		
Benzo(a)anthracene	56-55-3	0.108	SW846 8270C SIM	0.020		
Benzo(a)pyrene	50-32-8	0.15	SW846 8270C SIM	0.020		
Benzo(b)fluoranthene	205-99-2	1.5	SW846 8270C SIM	0.33		
Benzo(g,h,i)perylene	191-24-2	0.17	SW846 8270C SIM	0.020		
Benzo(k)fluoranthene	207-08-9	0.24	SW846 8270C SIM	0.020		
Chrysene	218-01-9	0.166	SW846 8270C SIM	0.020		
Dibenzo(a,h)anthracene	53-70-3	0.033	SW846 8270C SIM	0.020		
Fluoranthene	206-44-0	0.423	SW846 8270C SIM	0.33		
Fluorene	86-73-7	0.0774	SW846 8270C SIM	0.020		
Indeno(1,2,3-c,d)pyrene	193-39-5	0.2	SW846 8270C SIM	0.020		
Naphthalene	91-20-3	0.176	SW846 8270C SIM	0.020		
Phenanthrene	85-01-8	0.204	SW846 8270C SIM	0.020		
Pyrene	129-00-0	0.195	SW846 8270C SIM	0.020		

Analyte	CAS Number	Project Action Limit (mg/Kg)	Analytical Method	PQL Goal (mg/Kg)	Laboratory-specific	
					QLs (mg/Kg)	MDLs (mg/Kg)
Pesticides						
4,4' DDD	72-54-8	0.00488	SW845 8081A	0.0033		
4,4'-DDE	72-55-9	0.00316	SW845 8081A	0.0033		
4,4'-DDT	50-29-3	0.00416	SW845 8081A	0.0033		
Aldrin	309-00-2	0.002	SW845 8081A	0.0017		
alpha-BHC	319-84-6	0.006	SW845 8081A	0.0017		
alpha-Chlordane	5103-71-9	3.24	SW845 8081A	0.0017		
beta-BHC	319-85-7	0.005	SW845 8081A	0.0017		
delta-BHC	319-86-8	0.005	SW845 8081A	0.0017		
Dieldrin	60-57-1	0.0019	SW845 8081A	0.0033		
Endosulfan I	959-98-8	0.0055	SW845 8081A	0.0017		
Endosulfan II	33213-65-9	0.0055	SW845 8081A	0.0033		
Endosulfan sulfate	1031-07-8	0.0055	SW845 8081A	0.0033		
Endrin	72-20-8	0.00222	SW845 8081A	0.0033		
Endrin aldehyde	7421-93-4	0.00222	SW845 8081A	0.0033		
Endrin ketone	53494-70-5	0.00222	SW845 8081A	0.0033		
gamma-BHC (Lindane)	58-89-9	0.00237	SW845 8081A	0.0017		
gamma-Chlordane	5103-74-2	0.00324	SW845 8081A	0.0017		
Heptachlor	76-44-8	0.068	SW845 8081A	0.0017		
Heptachlor epoxide	1024-57-3	0.00247	SW845 8081A	0.0017		
Methoxychlor	72-43-5	0.019	SW845 8081A	0.017		
Toxaphene	8001-35-2	0.028	SW845 8081A	0.033		

Analyte	CAS Number	Project Action Limit (mg/Kg)	Analytical Method	PQL Goal (mg/Kg)	Laboratory-specific	
					QLs (mg/Kg)	MDLs (mg/Kg)
Herbicides						
2,4-D	94-75-7	690	SW846 8151A	0.033		
2,4-DB	94-82-6	490	SW846 8151A	0.033		
2,4,5-TP (Silvex)	93-72-1	490	SW846 8151A	0.033		
2,4,5-T	93-76-5	610	SW846 8151A	0.033		
Dalapon	75-99-0	1800	SW846 8151A	0.17		
Dicamba	1918-00-9	1800	SW846 8151A	0.033		
Dichloroprop	120-36-5	--	SW846 8151A	0.067		
Dinoseb	88-85-7	61	SW846 8151A	0.17		
MCPA	94-74-6	31	SW846 8151A	5		
MCPP	93-65-2	61	SW846 8151A	3.3		
Metals						
Aluminum	7429-90-5	25500	SW846 6010B	30		
Antimony	7440-36-0	3	SW846 6010B	1		
Arsenic	7440-38-2	3.9	SW846 6010B	1		
Barium	7440-39-3	48	SW846 6010B	10		
Beryllium	7440-41-7	160	SW846 6010B	0.5		
Cadmium	7440-43-9	0.99	SW846 6020	0.5		
Calcium	7440-70-2	--	SW846 6010B	500		
Chromium	7440-47-3	43.4	SW846 6010B	4		
Cobalt	7440-48-4	50	SW846 6010B	5		
Copper	7440-50-8	31.6	SW846 6010B	2.5		

Analyte	CAS Number	Project Action Limit (mg/Kg)	Analytical Method	PQL Goal (mg/Kg)	Laboratory-specific	
					QLs (mg/Kg)	MDLs (mg/Kg)
Iron	7439-89-6	20000	SW846 6010B	10		
Lead	7439-92-1	35.8	SW846 6010B	1		
Magnesium	7439-95-4	--	SW846 6010B	500		
Manganese	7439-96-5	460	SW846 6010B	5		
Mercury	7439-97-6	0.18	SW846 7471A	0.04		
Nickel	7440-02-0	22.7	SW846 6010B	4		
Potassium	7440-09-7	--	SW846 6010B	500		
Selenium	7782-49-2	1	SW846 6020	0.5		
Silver	7440-22-4	0.5	SW846 6020	0.2		
Sodium	7440-23-5	--	SW846 6010B	500		
Thallium	7440-28-0	5.1	SW846 6010B	2.5		
Vanadium	7440-62-2	57	SW846 6010B	5		
Zinc	7440-66-6	121	SW846 6010B	6		
TOC	--	--	SW846 9060A	400		

SAP Worksheet #15c -- Reference Limits and Evaluation Table, Groundwater Samples

(UFP-QAPP Manual Section 2.8.1)

Matrix: Groundwater

Analyte	CAS Number	Project Action Limit (µg/L)	Analytical Method	PQL Goal (µg/L)	Laboratory-specific	
					QLs (µg/L)	MDLs (µg/L)
Metals (Total and Dissolved)						
Aluminum	7429-90-5	1430	SW846 6020	200		
Antimony	7440-36-0	1.5	SW846 6020	1		
Arsenic	7440-38-2	0.045	SW846 6020	5		
Barium	7440-39-3	730	SW846 6010B	200		
Beryllium	7440-41-7	7.3	SW846 6020	1		
Cadmium	7440-43-9	1.8	SW846 6020	1		
Calcium	7440-70-2	--	SW846 6010B	5000		
Chromium	7440-47-3	11	SW846 6020	3		
Cobalt	7440-48-4	--	SW846 6020	1		
Copper	7440-50-8	150	SW846 6010B	30		
Iron	7439-89-6	2600	SW846 6010B	100		
Lead	7439-92-1	10	SW846 6020	1		
Magnesium	7439-95-4	--	SW846 6010B	5000		
Manganese	7439-96-5	88	SW846 6010B	15		
Mercury	7439-97-6	0.063	SW846 7470A	0.2		
Nickel	7440-02-0	73	SW846 6020	10		
Potassium	7440-09-7	--	SW846 6010B	5000		

Analyte	CAS Number	Project Action Limit (µg/L)	Analytical Method	PQL Goal (µg/L)	Laboratory-specific	
					QLs (µg/L)	MDLs (µg/L)
Selenium	7782-49-2	18	SW846 6020	5		
Silver	7440-22-4	18	SW846 6020	1		
Sodium	7440-23-5	20000	SW846 6010B	5000		
Thallium	7440-28-0	0.24	SW846 6020	2		
Vanadium	7440-62-2	18	SW846 6020	5		
Zinc	7440-66-6	1100	SW846 6020	20		
Anions						
Bromide	--	--	EPA Method 300.0	2000		
Chloride	--	--	EPA Method 300.0	500		

SAP Worksheet #15d -- Reference Limits and Evaluation Table, Surface Water Samples

(UFP-QAPP Manual Section 2.8.1)

Matrix: Surface Water

Analyte	CAS Number	Project Action Limit (µg/L)	Analytical Method	PQL Goal (µg/L)	Laboratory-specific	
					QLs (µg/L)	MDLs (µg/L)
PAHs						
2-Methylnaphthalene	91-57-6	72.16	SW846 8270C SIM	0.2		
Acenaphthene	83-32-9	20	SW846 8270C SIM	0.2		
Acenaphthylene	208-96-8	307	SW846 8270C SIM	0.2		
Anthracene	120-12-7	20.73	SW846 8270C SIM	0.2		
Benzo(a)anthracene	56-55-3	0.0038	SW846 8270C SIM	0.2		
Benzo(a)pyrene	50-32-8	0.0038	SW846 8270C SIM	0.2		
Benzo(b)fluoranthene	205-99-2	0.0038	SW846 8270C SIM	0.2		
Benzo(g,h,i)perylene	191-24-2	0.4391	SW846 8270C SIM	0.2		
Benzo(k)fluoranthene	207-08-9	0.0038	SW846 8270C SIM	0.2		
Chrysene	218-01-9	0.0038	SW846 8270C SIM	0.2		
Dibenzo(a,h)anthracene	53-70-3	0.0038	SW846 8270C SIM	0.2		
Fluoranthene	206-44-0	7.109	SW846 8270C SIM	0.2		
Fluorene	86-73-7	39.3	SW846 8270C SIM	0.2		
Indeno(1,2,3-c,d)pyrene	193-39-5	0.0038	SW846 8270C SIM	0.2		
Naphthalene	91-20-3	1.4	SW846 8270C SIM	0.2		
Phenanthrene	85-01-8	19.13	SW846 8270C SIM	0.2		
Pyrene	129-00-0	10.11	SW846 8270C SIM	0.2		
Metals (Total and Dissolved)						
Aluminum	7429-90-5	87	SW846 6020	50		

Analyte	CAS Number	Project Action Limit (µg/L)	Analytical Method	PQL Goal (µg/L)	Laboratory-specific	
					QLs (µg/L)	MDLs (µg/L)
Antimony	7440-36-0	5.6	SW846 6020	1		
Arsenic	7440-38-2	0.018	SW846 6020	5		
Barium	7440-39-3	4	SW846 6020	1		
Beryllium	7440-41-7	*0.66*	SW846 6020	1		
Cadmium	7440-43-9	0.25	SW846 6020	1		
Calcium	7440-70-2	--	SW846 6010B	5000		
Chromium	7440-47-3	74	SW846 6020	10		
Cobalt	7440-48-4	23	SW846 6020	1		
Copper	7440-50-8	9	SW846 6020	1		
Iron	7439-89-6	300	SW846 6010B	100		
Lead	7439-92-1	2.5	SW846 6020	1		
Magnesium	7439-95-4	--	SW846 6010B	5000		
Manganese	7439-96-5	50	SW846 6010B	10		
Mercury	7439-97-6	0.63	SW846 7470A	0.2		
Nickel	7440-02-0	52	SW846 6020	10		
Potassium	7440-09-7	--	SW846 6010B	5000		
Selenium	7782-49-2	5	SW846 6020	5		
Silver	7440-22-4	*0.36*	SW846 6020	1		
Sodium	7440-23-5	--	SW846 6010B	5000		
Thallium	7440-28-0	0.24	SW846 6020	2		
Vanadium	7440-62-2	20	SW846 6020	5		
Zinc	7440-66-6	120	SW846 6020	20		

ATTACHMENT B
DATA PACKAGE DELIVERABLES REQUIREMENTS

DATA PACKAGE DELIVERABLE REQUIREMENTS

The laboratory is to provide a hard copy plus two compact disks (CDs) each containing a PDF file in the following format:

1. Table of Contents
2. Case Narrative
3. Chain-of-Custody
4. Data Summary Package (contains summary of all CLP or CLP-like Forms 1 through 15 per analytical fraction)
5. Analytical Fractions (PAH, pesticide, herbicides, chloride and bromide, metals, and TOC and grain size as applicable)
    a. Results and QC Summary (summary of all CLP or CLP like Forms 1 through 15 for a particular analytical fraction; for dioxins, Forms 1DFA through 7 DFB for a particular analytical fraction)
    b. Raw Sample Data (includes all sample dilutions, sample re-analyses, QC samples, etc.)
    c. Calibration Data (includes all initial and continuing calibrations)
    d. Miscellaneous (includes extraction/preparation forms, percent solids determination, IDLs, MDLs, etc.)

Each of the above sections should be bookmarked in the PDF for easy access.

The following summary forms are required as part of the data package deliverable for SW-846 6020/6010B/7000A series for metals:

Results Report - must present all information presented on CLP FORM I.
Initial and Continuing Calibration Summary -must present all information presented on CLP FORM 2A.
Blanks - must present all information presented on CLP FORM 3.
ICP Interference Check Sample Summary - must present all information presented on CLP FORM 4.
Matrix Spike Summary - must present all information presented on CLP FORM 5A.
Post Digestion Spike - must present all information presented on CLP FORM 5B.
Lab Duplicate Results - must present all information presented on CLP FORM 6.
LCS Summary - must present all information presented on CLP FORM 7.
MSA Summary (Method of Standard Addition) - must present all information presented on CLP FORM 8.
ICP Serial Dilution Summary - must present all information presented on CLP FORM 8.
Detection Limits - must present all information presented on CLP FORM 9.
Linear Range – must present all information presented on CLP FORM 11.
Prep Log - must present all information presented on CLP FORM 12.
Analysis Run Log - must present all information presented on CLP FORM 13.
ICP-MS Tune – must present all information presented on CLP FORM 14.
ICP/MS Internal Standard Relative Intensity Summary - must present all information presented on CLP FORM 15.

<u>Also must include</u>: Instrument Calibration Records, Chain-of-Custody Forms, and Case Narrative.

Summary Forms for SW-846, 8260B and 8270C (i.e., Any SW-846 GC/MS analysis of Volatile and Semivolatile Organic Compounds) should be presented in a CLP-Like format. The following Summary Forms must be included:

Result Summary	One Sample per summary page. Presentation of analytical results for both method blanks and environmental samples, date of collection, preparation, and analysis. Environmental samples should be identified with the field identification numbers on the COCs.

Surrogate Recovery Form	Present all information contained on CLP Form II.
Summary of Matrix Spike/Matrix Spike Duplicate Recovery	Present all information contained on CLP Form III.
Instrument Performance Check Summary Form - Mass Spec Tuning Form	Present all information Contained on CLP Form V.
Initial Calibration Summary	Present all information contained CLP Form VI.
Continuing Calibration Summary	Present All Information contained on CLP Form VII.
Internal Standard Area and Retention Time Summary	Present all information contained CLP Form VIII.

Summary Forms for SW-846 8081A and 8082 Pesticide and Polychlorinated Biphenyl (PCB) Organic Compounds (and 8151A, 8141A, and all other SW-846 GC methods) should be presented in a CLP-Like format. The following Summary Forms must be included:

Result Summary	One Sample per summary page. Presentation of analytical results for both method blanks and environmental samples, date of collection, preparation, and analysis. Environmental samples should be identified with the field identification numbers on the COCs.
Surrogate Recovery Form	Present all information contained on CLP Form II for both Analytical Columns.
Summary of Matrix Spike/Matrix Spike Duplicate Recovery	Present all information contained on CLP Form III.
Summary of Pesticide Initial Calibration of Single Component Analytes	Present all information contained on CLP Form VI-PEST-2.
Summary of Pesticide Calibration Verification	Present all information contained on CLP Form VII-PEST-1 and Form VII-PEST-2.
Pesticide Analytical Sequence	Present all information contained on CLP Form VIII-PEST.
Pesticide Identification Summary For Single Component Analytes and for Multiple Component Analytes	Present all information contained on CLP Form X PEST 1 and 2.

MODIFIED ATTACHMENT C
ELECTRONIC DATA DELIVERABLE REQUIREMENTS
(See accompanying Word file)

ELECTRONIC DATA FORMAT REQUIREMENTS

1.0 INTRODUCTION

The laboratory is to submit text-based tab delimited EDD files for each SDG using Tetra Tech's laboratory data checker explained below. The files must be in the format specified in this Attachment. Additional information such as laboratory name, project name, fractions included, project number, site name/number, laboratory contact person and any specific comments related to the EDD should be included in the comments section of the EDD Submittal page.

The RESULT for nondetects should be populated with the project-specific sample quantitation reporting limits (i.e., either the sample quantitation limit or method detection limit, as specified in Section 3.0 of this scope of work. Any corrections made to the hardcopy data must also be made to the electronic file. Appropriate qualifiers as identified by the analytical protocol must also be designated; laboratory QC non-compliance codes are not to be depicted.

Tetra Tech's electronic EDD format follows the ADAPT structure and requires the A1 and A3 files. The A2 file is only required if the project is using ADAPT; and, for non-ADAPT EDD submittals the A2 file may be omitted. The EDD consists of separate, tab-delimited ASCII text files. Each file corresponds to a database table. The tables are identified as the Analytical Results Table (A1) and Sample Analysis Table (A3). A separate set of text files must be created and submitted for each sample delivery group (SDG). The files must be identified to correspond to the (A1) table and the (A3) table. The file naming convention is: the Sample Delivery Group (SDG) followed by the table identifier (A1 or A3), followed by the ".txt" extension. The file names must not contain spaces or special characters. For example, the EDD file names for a laboratory-reporting batch identified as SDG001 would be as follows:

SDG001A1.txt
SDG001A3.txt

On certain projects Tetra Tech will utilize the ADAPT Electronic Data Validation software, which will require the laboratory to use the ADAPT electronic data deliverable checker software prior to submitting the files through Tetra Tech's laboratory data checker (this will be clearly specified in the Tetra Tech laboratory statement of work). The ADAPT checker software can be downloaded from Laboratory Data Consultants' web site: http://www.lab-data.com. For projects which Tetra Tech is using the ADAPT software, Tetra Tech will provide the laboratory with the project library. The laboratory is not permitted to modify the project library. ADAPT projects will require the laboratory to export all three checked files (A1, A2, and A3) from the ADAPT software and submit them through Tetra Tech's laboratory data checker. **ADAPT error logs generated must be included with the electronic PDF data validation package and cannot be submitted through the laboratory data checker.**

The values reported in the EDD text files must agree exactly with the final values reported on the PDF data package sample result summaries. The details of file naming conventions, data structure and data checker use are discussed below.

Analytical Results Table (A1 File)

The Analytical Results table contains analytical results and related information for target analytes in field samples and associated laboratory quality control samples (excluding calibrations and tunes). Field samples and laboratory method blanks must report a result record for each analyte reported within a method. Laboratory control samples (LCS and LCSD) and matrix spike samples (MS and MSD) must report a result record for every analyte specified as a spiked analyte in the laboratory statement of work. Table A1 in this document lists the field names and data type descriptions for the Analytical Results Table (A1).

Lab Instrument Table (A2 File)

A2 file is only required if the project is using ADAPT. In all other EDD submittals, the A2 file may be omitted. Laboratories should refer to the ADAPT User Guide for populating the A2 Table.

Sample Analysis Table (A3 File)

The Sample Analysis table contains information specific to field environmental samples and laboratory quality control analyses (excluding calibrations and tunes). A sample record must exist for each sample/method/matrix/analysis type combination. Table A3 in this document lists the field names and data type descriptions for the Sample Analysis Table (A3).

All electronic data deliverables are due within the same time established for the associated hardcopy data packages.

In addition, the laboratory QC officer must read and sign a copy of the Quality Assurance Review Form displayed on the next page of this Attachment. Electronic deliverables are not considered to be complete without the accompanying Quality Assurance Review Form.

I _____, as the designated Quality Assurance Officer, hereby attest that all electronic deliverables have been thoroughly reviewed and are in agreement with the associated hardcopy data. The enclosed electronic files have been reviewed for accuracy (including significant figures), completeness and format. The laboratory will be responsible for any labor time necessary to correct enclosed electronic deliverables that have been found to be in error. I can be reached at
(____)_____if there are any questions or problems with the enclosed electronic deliverables.

Signature:_____ Title: _____Date: _____

2.0 EDD Field Properties

Tables A1 and A3 in this document specify the EDD field properties. Laboratories should refer to the ADAPT User Guide for populating the A2 Table. These include the field name, sequence order, field description, data type/length and reporting requirement for each field. Fields in the EDD **must** be sequenced according to the order that they appear below in Tables A1 and A3. For example, in the Analytical Results table (A1), the field "ClientSampleID" will always be the first piece of information to start every new line of data (or database record), followed by the field "LabAnalysisRefMethodID", "AnalysisType", etc.

When creating an EDD as a text file, use the ASCII character set in a file of lines terminated by a carriage return and line feed. No extra characters are allowed at the end of a line, after the carriage return and line feed. Enclose each data value with double quotes (text qualifier) and separate each field value with a **tab** character (tab delimiter). Data fields with no information (null) may be represented by two consecutive tabs. For example, in the Sample Analysis table, since the "Collected", "ShippingBatchID", and "Temperature" fields do not apply to laboratory generated QA/QC samples, the record for a Laboratory Control Sample by Method 8270C would be entered as follows. Note that the first two fields ("ProjectNumber" and "ProjectName") are omitted in this example.

…"LCSW100598" "AQ" "LCSW100598" "LCS" "8270C",…etc.

If a field is populated with less than the maximum allowed number of characters, do not pad the values with leading or trailing spaces. In the above example, although the "MatrixID" field can accommodate up to 10 characters, only 2 characters were entered in this field. **Do not include the delimiter (tab character) within any of the field values.** Example EDD files may be downloaded from the LEDD Checker application.

An example database shall be sent for review prior to the first electronic deliverable in the required .txt format. The example file will be examined for completeness and comments will be sent to the laboratory. Any questions regarding the electronic deliverable should be directed to LabSupport@tetratech.com.

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ClientSampleID	Client or contractor's identifier for a field sample If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Method Blanks, LCS, and LCSD enter the unique LaboratorySampleID into this field. Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the AnalysisType field is used for this distinction). For example, MW01DL and MW01RE are not allowed. Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Analytical Results table for both EDDs.	Text	25	X
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or a Lab Identifier for a method such as a SOP Number, however; method ID is specified by the project. The method ID must be entered into the standard list.	Text	25	X
AnalysisType	Defines the analysis type (i.e., Dilution, Reanalysis, etc.). This field provides distinction for sample result records when multiple analyses are submitted for the same sample, method, and matrix; for example dilutions, re-analyses, and re-extracts.	Text	10	X
LabSampleID	Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except for field length and that the LabSampleID must be distinct for a given field sample or lab QC sample and method. Suffixes may be applied to the LabSampleID to designate dilutions, reanalysis, etc.	Text	25	X
LabID	Identification of the laboratory performing the analyses.	Text	7	X
ClientAnalyteID	CAS Number or unique client identifier for an analyte	Text	12	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	or isotope. If a CAS Number is not available, use a unique identifier provided by the client or contractor. The ClientAnalyteID for a particular target analyte or isotope should be specified by the project and must exist in the standard value tables for Analytes. For the LCS, LCSD, MS, and MSD, it is only necessary to report the compounds designated as spikes in the library (and surrogates for organic methods.) For TICs from GC/MS analyses, enter the retention time in decimal minutes as the Client Analyte ID.			
AnalyteName	Chemical name for the analyte or isotope. The project specifies how an analyte or isotope is named. The analyte name must be associated to a ClientAnalyteID in the standard values table for Analytes (excluding compounds designated as TIC's).	Text	60	X
Result	Result value for the analyte or isotope. Entries must be numeric. For non-detects of target analytes or isotopes and spikes, **do not enter "ND" or "0". Do not leave this field blank**. If an analyte or spike was not detected, enter the reporting limit value corrected for dilution and percent moisture as applicable. Do not enter "0"	Numeric (1)	20(6)	X
ResultUnits	The units defining how the values in the Result, DetectionLimit, and ReportingLimit fields are expressed. For radiochemistry this also includes how the value in the Error field is expressed.	Text	10	X
LabQualifiers	A string of single letter result qualifiers assigned by the lab based on client-defined rules and values. <u>The "U" Lab Qualifier must be entered for all non-detects.</u> Other pertinent lab qualifiers may be entered with the "U" qualifier. Order is insignificant. Lab qualifiers other than those listed in the standard	Text	7	Q

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	values table may be used. If so, these must be added to the standard value table in the application.			
DetectionLimit	For radiochemistry methods, the minimum detectable activity for the isotope being measured. For all other methods: The minimum detection limit value for the analyte being measured.	Numeric (1)	10(6)	X
DetectionLimitType	Specifies the type of detection limit (i.e., MDA, MDL, IDL, etc.).	Text	10	X
RetentionTime or Error	For radiochemistry methods only, enter the 2 Sigma Counting Errors. The units for error are entered in the ResultUnits field. For GC/MS methods only, enter the time expressed in decimal minutes between injection and detection for GC/MS TICs only For target analytes in all other methods, leave this field blank. Note: GC retention times are not evaluated at this time.	Text	5	T
AnalyteType	Defines the type of result, such as tracer, surrogate, spike, or target compound.	Text	7	X
PercentRecovery	For radiochemistry methods: The tracer yield, if applicable. For all other analytical methods: The percent recovery value of a spiked compound or surrogate. If the spike or surrogate was not recovered because of dilution, enter “DIL”. If a spike or surrogate was not recovered because of matrix interference, enter “INT”. If a spike or surrogate was not recovered because it was not added to the sample, enter “NS”.	Numeric (1)	5(3)	R
RelativePercentDifference	The relative percent difference (RPD) of two QC results, such as MS/MSD, LCS/LCSD, and Laboratory Duplicates. Report RPD in Laboratory Duplicate, LCSD, and MSD records only.	Numeric (1)	5(3)	D
ReportingLimit	Reporting limit value for the measured analyte or isotope Factor in the dilution factor and percent moisture correction, if applicable. The Reporting Limit for each analyte and matrix in a given method is specified in	Numeric (1)	10(6)	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	the project library or QAPP.			
ReportingLimitType	Specifies the type of reporting limit (i.e., CRQL, PQL, SQL, RDL, etc). The Reporting Limit Type for each method and matrix is specified in the project library or QAPP.	Text	10	X
ReportableResult	This field indicates whether or not the laboratory chooses an individual analyte or isotope result as reportable. Enter "YES" if the result is reportable. Enter "NO" if the result is not reportable. If only one analysis is submitted for a particular sample and method, enter "YES" for all target compounds (where Analyte Type = TRG). For GC/MS methods enter yes for tentatively identified compounds (where Analyte Type = TIC). If two or more analyses are submitted for a particular sample and method (i.e. initial analysis, reanalysis and/or dilutions), enter "YES" from only <u>one</u> of the analyses for each target compound. For example: a sample was run a second time at dilution because benzene exceeded the calibration range in the initial, undiluted analysis. All target analytes are reported in each analysis. For the initial analysis, (Analysis Type = RES), enter "NO" for benzene and enter "YES" for all other compounds. For the diluted analysis (Analysis Type = DL), enter "YES" for benzene and enter "NO" for all other compounds. For TICs (Analyte Type = TIC), if more than one analysis is submitted for a particular sample and method, choose only one of the analyses where Reportable Result = YES for <u>all</u> TICs. For example, a sample was run a second time because one or more target compounds exceeded the calibration range in the undiluted analysis. Choose a particular analysis and enter "YES" for all TICS. In the other analysis enter "NO" for all TICs. Note that it is not necessary to report the full target analyte list for the initial result, dilution, re-analysis, or re-extraction. However, each target analyte must be reported YES once and once only in the case of multiple analyses for a given sample, method, and matrix. In the case of organics, all surrogates must be reported for all analyses submitted for a given sample, method, and, matrix.	Text	3	X

Table A1
Field Descriptions for the Analytical Results Table (Table A1)
Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
SpkConcnAdded	The spike added. This value must be reported in the same units as the result. Where (SA) in the following equation: % Recovery = (SSA-SC)/SA x 100% where : SSA is the spiked sample concentration (amount) after spiking. SC is the sample concentration (amount) before spiking. SA is the the expected increase in sample concentration (amount) as a result of spiking. This value must incorporate all correction factors such as dilution factor and moisture content that are applied to the spiked sample when computing the spiked sample concentration or amount.	Numeric (1)	10(6)	R
SpkParentSampleID	The sampleID of a sample (often called the original sample) that receives a spike aliquot to form a spiked sample such as a matrix spike. This is not the same as the ID of the spiked sample (such as a matrix spike) after spiking. The result for SpkParentSampleID and the result (i.e., SpkConcnAdded) for the spiked sample are used to compute percent recovery of the analyte.	Text	25	R
SamplePrepInitial	The initial sample preparation volume in liters (L) for aqueous samples or grams (g) for solid samples.	Numeric (1)	20(6)	
SamplePrepFinal	The final sample preparation volume in liters (L) for aqueous samples or grams (g) for solid samples.	Numeric (1)	20(6)	
Comment	Add any comments or additional information specific to the analyte test result data record.	Text	200	

X Required field.
Q Only required if laboratory has qualified the result.
T Only required for tentatively identified compounds by GC/MS.
R Only required if Analyte_Type =”SPK” or “SURR”.
D Only required for spiked compounds in the LCSD and MSD.
(1) Field Length indicates decimal precision in parenthesis. For example, 5(2) = a total width of 5 numbers including a maximum of 2 decimal places.

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
ProjectNumber	Project number assigned by the client.	Text	30	X
ProjectName	Project name assigned by the client.	Text	90	X
ClientSampleID	Client or contractor's identifier for a field sample If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Method Blanks, LCS, and LCSD enter the unique LaboratorySampleID into this field Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the Analysis_Type field is used for this distinction). For example, MW01DL and MW01RE are not allowed Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Sample Analysis table for both EDDs.	Text	25	X
Collected	Date and Time of sample collection. Refer to the date/time format at the end of this table. Leave this field blank for Method Blank, LCS, and LCSD	Date/ Time	16*	C
MatrixID	Sample matrix (i.e., AQ, SO, etc.)	Text	10	X
LabSampleID	Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except field length and that the LabSampleID must be unique for a given field sample or lab QC sample and method.	Text	25	X
QCType	This record identifies the type of quality control sample QC (i.e., Duplicate, LCS, Method Blank, MS, or MSD). For regular environmental samples, populate this field with "NM".	Text	10	X
ShippingBatchID	Unique identifier assigned to a cooler or shipping container used to transport client or field samples. Links all samples to a cooler or shipping container. No	Text	25	C

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	value is required for method blanks, LCS, and LCSD.			
Temperature	Temperature (in centigrade degrees) of the sample as received. This field is not required for radiochemistry methods.	Numeric (1)	10(6)	C
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or laboratory identifier for a method such as a SOP number, however; values used for Laboratory Method IDs are specified by the project and must in the in standard value list for method IDs.	Text	25	X
PreparationType	Preparation Method Number (i.e., 3010A, 3510C, 3550C, 5030B, etc.) For analytical procedures that do not have a specific preparation method number, use “Gen Prep”.	Text	25	X
AnalysisType	Defines the type of analysis such as initial analysis, dilution, re-analysis, etc. This field provides distinction for sample records when multiple analyses are submitted for the same sample, method, and matrix, for example: dilutions, re-analyses, and re-extracts.	Text	10	X
Prepared	For radiochemistry leave this field blank. For all other methods enter the date and time of sample preparation or extraction. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
Analyzed	Date and time of sample analysis. Refer to the date and time format at the end of this table.	Date/ Time	16*	X
LabID	Identification of the laboratory performing the analysis.	Text	7	X
QCLevel	The level of laboratory QC associated with the analysis reported in the EDD. If only the Analytical Results Table (A1) and the Sample Analysis Table (A3) information are submitted for the sample, enter “COA”. If the Laboratory Instrument Table (A2) information is also submitted for the sample, enter “COCAL”	Text	6	X
ResultBasis	Indicates whether results associated with this sample records are reported as wet or percent moisture corrected. This field is only required for soils and sediments. Enter “WET” if results are not corrected for	Text	3	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	percent moisture. Enter "DRY" if percent moisture correction is applied to results.			
TotalOrDissolved	This field indicates if the results related to this sample record are reported as a total or dissolved fraction. This field is only required for metal methods. For all other methods leave this field blank.	Text	3	X
Dilution	Dilution of the sample aliquot. Enter "1" for method blanks, LCS, and LCSD, or if the field samples was analyzed without dilution.	Numeric (1)	10(6)	X
HandlingType	Indicates the type of leaching procedure, if applicable (i.e., SPLP, TCLP, WET). Leave this field blank if the sample analysis was not performed on a leachate.	Text	10	L
HandlingBatch	Unique laboratory identifier for a batch of samples prepared together in a leaching procedure (i.e., SPLP, TCLP, or WET preparation). The HandlingBatch links samples with leaching blanks. Leave this field blank if the sample analysis was not performed on a leachate	Text	12	L
LeachateDate	Date and time of leaching procedure (i.e., date for SPLP, TCLP, or WET preparation). Refer to the date and time format at the end of this table. Leave this field blank if the sample analysis was not performed on a leachate	Date /Time	16*	L
Percent_Moisture	Percent of sample composed of water. Enter for soil and sediment samples only.	Numeric (1)	10(6)	X
MethodBatch	Unique laboratory identifier for a batch of samples of similar matrices analyzed by one method and treated as a group for matrix spike, matrix spike duplicate, or laboratory duplicate association The method batch links the matrix spike and/or matrix spike duplicate or laboratory duplicates to associated samples. Note the MethodBatch association may coincide with the PreparationBatch association. The MethodBatch is specifically used to link the MS/MSD and/or DUP to associated samples.	Text	12	X
PreparationBatch	Unique laboratory identifier for a batch of samples	Text	12	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	prepared together for analysis by one method and treated as a group for method blank, LCS and LCSD association. The PreparationBatch links method blanks and laboratory control samples (blank spikes) to associated samples. Note, the PreparationBatch association may coincide with the MethodBatch association but the PreparationBatch specifically links the Method Blank and LCS to associated samples.			
RunBatch	For radiochemistry methods leave this field blank. For all other methods the RunBatch is the unique identifier for a batch of analyses performed on one instrument under the control of one initial calibration and initial calibration verification. The RunBatch links both the initial calibration and initial calibration verification to subsequently analyzed and associated continuing calibrations, field samples, and QC analyses. For GC/MS methods, the RunBatch also links a BFB or DFTPP tune. A distinct RunBatch must used with every new initial calibration within a method The value entered in this field links a particular sample/method/analysis type record to a set of associated initial calibration and initial calibration verification records from Table A2. This field is only required if the A2 table is included with the EDD.	Text	12	X
AnalysisBatch	For radiochemistry methods leave this field blank. For all other methods the AnalysisBatch is the unique identifier for a batch of analyses performed on one instrument and under the control of a continuing calibration or continuing calibration verification. The AnalysisBatch links the continuing calibration or calibration verification to subsequently analyzed and associated field sample and QC analyses. For GC/MS methods, the AnalysisBatch also links the BFB or DFTPP tune. A distinct AnalysisBatch must be used with every new continuing calibration or continuing calibration verification within a method The value entered in this field links a particular sample/method/analysis type record to a set of associated continuing calibration records in the Laboratory Instrument table.	Text	12	X

Table A3
Field Description for the Sample Analysis (Table A3)
This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Required Value
	This field is only required if the A2 table is included with the EDD.			
LabReportingBatch	Unique laboratory identifier for the EDD. This is equivalent to the sample delivery group, lab work number, login ID, etc. The LabReportingBatch links all records in the EDD reported as one group. The value entered in this field must be the same in all records.	Text	12	X
LabReceipt	Date and time the sample was received in the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
LabReported	Date and time hard copy reported delivered by the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/ Time	16*	X
Comment	Add any comments or additional information specific to the sample analysis data record.	Text	200	

C Only required for regular samples, duplicates and MS/MSDs.
L Only required if analysis performed is SPLP, TCLP or WET extracts.
X Required field.
(1) Field Length indicates decimal precision in parenthesis. For example, 5(2) = a total width of 5 numbers including a maximum of 2 decimal places.
* Format Date and Time as MM/DD/YYYY hh:mm; where MM = two digit month, DD = two digit day, and YYYY = four digit year, hh = hour in 24 hour format, and mm = minutes.

3.0 Laboratory Data Checker

The Laboratory Data Checker is a web-based application that will review Laboratory Electronic Data Deliverables (LEDDs) for adherence to Tetra Tech's EDD format requirements. EDDs will be reviewed for elements such as missing data and/or columns of data, and compliance of the data within each column to the required data types/lengths. Once an EDD passes through the checker with no errors, it must be submitted to Tetra Tech through the LEDD Checker application.

Access to the LEDD Checker application will be provided by an initial registration/approval process. An Information Systems Group (ISG) Administrator will approve requests for access**.** To access the site or begin the registration process, visit the ISG web site at http://isg.ttnus.com and select the "Laboratory Checker" link on the left of the home page. Registered users may access the checker immediately by logging in to the system using theircredentials. New users must select the "Register" button and provide all of the requested information**.**

After completing all fields on the registration form, select the "Submit" button to complete the request process. Upon verification by an ISG Administrator, an email notification will be sent verifying the user ID, password and account status. Forgotten passwords may be retrieved by using the "Forgot password?" link on the login page. Note that the email address that was provided for registration or password retrieval is the user ID and must be a valid e-mail address.

The general process for submitting EDD files through the LEDD Checker involves a 3-stage process that includes an upload stage, an error checking stage and a submittal stage.

Log into the LEDD Checker by typing your login credentials and select the "Login" button. The LEDD Checker home page provides a general overview of the checker functionality and EDD file format requirements. At the bottom of the home page, example EDDs are provided that may be viewed or downloaded. To download the files, right click on the link and select "Save target as" from the menu. Each LEDD Checker page includes a navigation bar with links to return to the home page or continue the checking and submittal process. Users should **NOT** use the back or forward buttons on the browser, instead use the links provided in the application to navigate through the site.

Detailed information regarding EDD preparation, formatting requirements and text file naming conventions are provided in the Electronic Data Format Requirements Section of the Laboratory SOW.

Begin the upload stage by selecting the "Upload/Check Files" link on the home page. Follow the steps on the upload page starting with the selection of the laboratory name that corresponds to your organization. If your organization is not listed, contact LabSupport@tetratech.com, and provide a full description of your organization name, contact information and include "Laboratory Contractor ID Request" in the subject line. An ISG Administrator will respond to the request via e-mail.

Load the appropriate A1, A2, or A3 target EDD files by clicking the "Browse" button next to each data table input box. A file browser dialog will appear allowing files to be selected from a local or network drive. After the EDD files are loaded, click the "Upload" button to complete the upload stage. Note that each table may be uploaded and checked separately; however, a minimum of the A1 and A3 files are required in order to submit the EDDs.

If the file upload was successful, the checking page will immediately load. Begin the checking stage by selecting the "Check Files" button. The LEDD Checker will begin validating the EDD files for compliance. Depending on file size and network activity the validation process may take several minutes. The progress should be displayed in the information bar at the bottom of the browser window. **Do not** select the "Check Files" button again or otherwise use the browser during this process. Other applications may be used; however, note that the LEDD Checker may not sit idle for more than 30 minutes. If the time is exceeded a new session must be started in a new browser window.

Any errors will be processed and returned on the error page. The following general errors may be returned.

- Column count / table structure errors – due to column header names being included, improper delimiter, extra tabs, extra or missing columns of data, spaces or other characters at the end of a row.
- Row and column value specific errors – may occur for one or more reasons including: data truncation, invalid date / time format, invalid decimal precision or field width exceedance, or if a value is not in a list of valid values or expected range.

If column count / table structure errors are encountered, the LEDD Checker will return an error and stop the checking process.

The EDDs will not be processed any further until the column errors are resolved. Text fields are validated for truncation. Date / Time fields are validated for truncation and format compliance. Numeric decimal fields are validated for truncation, character type compliance and decimal precision. All required fields are validated for null values or empty text strings (i.e. spaces). The LEDD Checker will return a list of all errors in and include a reference to the row number on which the error occurred. Note that consecutive EDD files may be loaded and checked, and submitted while logged in. However, no data may be submitted until all EDD files have passed through the LEDD Checker without errors. The list of errors may be printed by selecting the "Print this Page" button from the checker error page.

If the EDD files pass with no errors, the submittal page will immediately load. To complete the submittal stage, include the following information in the comment and additional information area of the form: laboratory name, laboratory contact person, project name, project number, site name/number, fractions included and any specific comments related to the EDD. Select the “Submit Files” button to continue the submittal process.

The submittal stage is not considered complete until a unique ticket key reference is returned in the browser window. The ticket key reference must be printed for record of submission and future reference. In addition, a copy of the ticket key reference must be included in the PDF data package.

APPENDIX D

LABORATORY SOPs

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-101
Revision History
Cover Page
Page 1

TITLE: **EQUIPMENT MAINTENANCE**

Prepared By: Julie Ricardi Date: 8-96

Approved By:

Group Supervisor: _____ Date: _____

Operations Manager: Joel 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 Lachat maintenance. Attached equipment list.	DN	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.	DN	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.	DN	03-06	03-06

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: **EQUIPMENT MAINTENANCE**

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated ICP maintenance to include maintenance for new ICP (7500). Updated equipment list.	DR	04·07	04·07

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-101-06**, titled **EQUIPMENT MAINTENANCE**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **CA-101-06**, 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 maintenance procedures employed for analytical equipment utilized by Katahdin Analytical personnel. It is applicable to all equipment utilized by laboratory personnel for analytical 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 shall be consulted for 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

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., 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 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.

7.3.1 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.

7.3.2 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. If maintenance was conducted as the result of a suspected or known instrument problem, a summary of the problem and its final resolution must be included. The maintenance record must clearly indicate whether the problem was resolved and the date when return to control was noted.

7.3.3 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).

7.3.4 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

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Not applicable.

9.0 METHOD PERFORMANCE

Not applicable.

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

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 O_2/moisture traps as needed
- Clean/replace GC detector as needed.

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.

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.

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.
- Sonicate and replace solvent with every use.
- Replace the UV lamp as needed.
- Check and replace seal-pak as needed

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.

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.

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.

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.

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 Instrumentation

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 biannually.

TABLE 3

ROUTINE MAINTENANCE SCHEDULE FOR GENERAL LABORATORY EQUIPMENT

General Laboratory Areas

- Clean and calibrate balances biannually (minimum).
- Check balance calibration each day of use.
- Clean balance pan prior to each use.
- Calibrate automatic pipettes with each use.
- Calibrate thermometers yearly against an NIST traceable thermometer; calibrate heavily used digital thermometers quarterly.
- Record refrigerator, freezer, and oven temperatures each weekday.
- 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 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 5972 GC/MS with EPC; Tekmar LSC-3000 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-2000 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 5970 GC/MS with EPC and Model 7673A autosampler.	1989/ 1996	8270, 625 & Current versions of CLP SOWs (OLM, OLC, & SOM) (SVOA)	X
Hewlett Packard 5972 GC/MS with EPC; Tekmar LSC 3100 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 5973 GC/MS with EPC; Agilent 7683 ALS. The instrument is capable of being flipped from volatile to semivolatile analysis.	1999/ 2001	8270, 625 & Current versions of CLP SOWs (OLM, OLC, & SOM) (SVOA)	U

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; Agilent 7673 ALS.	**2006**	8270, 625 & Current versions of CLP SOWs (OLM, OLC, & SOM) (SVOA)	R
Hewlett Packard 5972 GC/MS with EPC, Centurion Water Purge and Trap Autosampler with foam sensor option and EST EnCon Purge and Trap Concentrator	**2006**	5030/8260 5035/8260, 624, Current versions of CLP SOWs (OLM, OLC, & SOM) (VOA), & 524.2	T
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	GC08
Hewlett Packard Model 5890 gas chromatograph with EPC and dual electron capture detectors(ECD); Hewlett Packard Model 7673 autosampler.	**1993**	8081, 8082, 608	GC06
Hewlett Packard Model 5890 gas chromatograph with dual electron capture detectors (ECD); Hewlett Packard Model 7673A autosampler.	**1988**	504, 8011, 8151	GC03
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

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 dual flame ionization detectors; Tekmar ALS 2016 autosampler; Tekmar LSC 3000 purge and trap.	**1991**	8015 MOD., GRO	GC04
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
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 dual EPC injection ports and micro electron capture detectors; Agilent Model 7683 autosampler	**2000**	8081, 8082 (INCLUDING CONGENERS), 608 & Current versions of CLP SOWs (OLM, OLC, & SOM) (P/P)	GC07
Waters HPLC system with 600 multisolvent delivery system; 717 Plus autosampler; Spectra System UV2000 detector.	**1997/ 2004**	8330, 8332	HPLC01
Waters HPLC system with 600E multisolvent delivery system; 717 Plus autosampler; Waters 846 UV Tunable Absorbance Detector.	**1997/ 2003**	FOOD TESTING	HPLC02

TITLE: EQUIPMENT MAINTENANCE

ATTACHMENT A

ANALYTICAL EQUIPMENT LIST, cont'd

ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION		DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
ABC Autoprep 1000 GPC with UVD-1 ultraviolet detector and chart recorder.		1994	Current versions of CLP SOWs (OLM, OLC, & SOM) (GPC-3640)	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 #1
Thermo ICAP 6500 ICP Emission Spectrometer with autosampler		2006	6010, 200.7, ILM05.3	I
Agilent 7500a ICP-MS with autosampler and autodiluter.		2001	6020, 200.8, ILM05.3	F
CETAC M-6100 Automated Mercury Analyzer with Autosampler		2004	7470/7471, 245.1, 245.5, ILM05.3	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
Waters 717 Autosampler, Waters 431 conductivity detector, Spectra System P400 pump.	Dionex IonPak AS4A column; Dionex AMMS III chemical suppression system	1991/ 2004	Ion chromatography – Various Anions	See "Key Instrumentation" column for identification
	Dionex AS16 column; Dionex ultra ASRS electronic suppression system.		Ion Chromatography - Perchlorate	See "Key Instrumentation" column for identification

TITLE: EQUIPMENT MAINTENANCE

ATTACHMENT A

ANALYTICAL EQUIPMENT LIST, cont'd

ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Scanning Fluorescence Detector	**1997**	Out of Service	NA
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 Analyzer w/ 40 mL vials; Model SSM-5000A Solid Sample Module.	**2002**	TOC	WC2
10 position Lab Crest Cyanide Midi-Distillation system.	**1998**	Cyanide	WC3
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
Thermo Spectronic Genesys 10uv spectrophotometer	**2005**	Food Testing	Various
DryVap Concentrator System 5000	**2005**	GC and GC/MS Extractables	DryVap #1

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270

Prepared By: Casarin/Millett/Galasyn Date: 2/97

Approved By:

Group Supervisor: [signature] Date: 011201

Operations Manager: [signature] Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1·23·01

General Manager: [signature] Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
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	LAD	02/05	02/05
07 8270C	Sect. 5.3.2.1 changed 200 ug/ml to 1500 ug/ml Sect. 7.2 changed Instrument "K" to Instr. "R" Sect. 7.5.1 updated table ~~Sect.~~ Table 5: minor edits	LAD	04/06	04/06

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
08 8270C	Added ICV Criteria to Definitions, 7.5.22 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	LAD	~~05/07~~ 07/07	05/07 07/07
09 8270C	Reworded correlation coefficient criteria Added new instrument (G). Updated Section 10. Fixed grammatical errors.	LAD	02/08	02/08

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

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-204-09, 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-204-09, 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 GC/MS: SW 846 METHOD 8270.

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 percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

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

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of semivolatile organic compounds by EPA Method 8270. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of semivolatiles by Method 8270 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to

initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory.

Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous 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 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

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 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 two standard stock 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. Both stocks should be prepared at 200 ug/mL.

5.3.2.2 Independent Calibration Verification (ICV) Standard – From a source other than that used to make the calibration standards, prepare a standard in methylene chloride containing the compounds listed in Table 3. The final concentration of each compound is 150 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.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

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.

- Look at the batch to be analyzed and check the following:

  - Make sure that the data files are in numerical order with no duplication and that the method file is the same as that used for ICAL or Continuing Calibration analysis.

  - Bottle numbers match with the numbers on the autosampler tray.

After the batch has been deemed free of errors, start the batch by using the "Position and run" command under the SEQUENCE menu in MSTop.

7.4 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks or samples, the GC/MS system must be shown to meet the mass spectral key ion and ion abundance criteria for decafluorotriphenylphosphine (DFTPP) tabulated below. Pentachlorophenol, benzidine and DDT are also present in this standard.

Mass	Criteria
51	30.0 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	140-260°C at 10 degrees/minute
Final column temperature hold	260°C
Injection port temperature	250°C
Transfer line/source temperature	285°C
Injector - splitless, valve time	0.18 minutes
EPC	inlet B
Constant flow	ON
Constant flow pressure	10psi
Constant flow temperature	30°C
Vacuum comp.	ON
Run time	10-12 minutes
Scan start time	5.0 minutes
Sample volume	2.0 uL of 25 ng/uL DFTPP solution
Carrier gas	helium at 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.

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; 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 two six point curves, one using mix A (8270 compounds) and one using mix B (Appendix IX compounds). The calibration levels are 10, 25, 50, 100, 150 and 200 ng/uL Calibration is done to determine instrument sensitivity and the linearity of GC/MS response for the semivolatile target and surrogate compounds.

Final conc. (ng/uL)	200 ng/uL SVOA Stock Soln Added (uL) A or B	$MeCl_2$ Added (uL)	Final Vol (uL)	IS Added (uL)
10	10	190	200	2
25	25	175	200	2
50	50	150	200	2
100	50	50	100	1
150	75	25	100	1
200	100	0	100	1

If additional compound mixtures are added, the volume of $MeCl_2$ is adjusted to maintain a final volume of 200 or 100 uL. A 100 uL aliquot of each of the standards above is spiked as above with 4000 ng/uL Internal Standard stock and analyzed.

Internal Standards
1,4-Dichlorobenzene-d4
Naphthalene-d8
Acenaphthene-d10
Phenanthrene-d10
Chrysene-d12
Perylene-d12

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

Column Temperature Program	40°C for 3 minutes to 320°C at 10°/minute
Final Column Temperature hold	320°C
Run Time	34-36 minutes
Scan Start Time	1.8 minutes (time may vary dependent upon column length)
Injection volume	1 uL

The conditions are set up in the method files L8270CXX.M.

After analysis of the six calibration points, they must be processed and evaluated for adherence to QC criteria. Minimum requirements of ID files are the use of specific quantitation ions and quantitating a specific set of targets and surrogates with a set internal standard. These requirements are found in Tables 3 and 5.

7.5.2 Initial Calibration Criteria

Relative response factors (RRFs) must be calculated and evaluated for each target compound and surrogate. The RRF is defined as follows:

$$RRF = \frac{Ax}{A_{IS}} \times \frac{C_{IS}}{Cx}$$

where: Ax = area of the primary ion for the target compound
A_{IS} = area of the primary ion for the corresponding istd
C_{IS} = concentration of the istd (ng/uL)
C_x = concentration of the target compound

After the calibration points have been quantitated, update the calibration curve points using the Target data processing software to generate the 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.

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.

Achieving CCC and SPCC criteria is not a substitute for performing a calibration for all target analytes.

7.5.2.1 Linearity of Target Analytes

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 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 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.

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.

The area for the internal standards in the calibration verification must be within a factor of two (-50% to 100%) from the mid-point standard of the most recent initial calibration. This is listed in the ISTD monitor report.
If the calibration verification does not meet criteria, corrective action must be taken. Depending on the situation, corrective action may be as follows:

- Re-analyze the 50 ng/uL continuing calibration check.
- Change the septum; clean the injection port; install a clean, silanized quartz liner; cut off a small portion (1" to 3") of the front end of the capillary column (this is usually performed when acid RFs are low and/or chromatography is poor).
- Analyze a new initial calibration curve.

The last option, the generation of a new initial calibration curve, is usually chosen when percent difference are >30%. In these instances, there is little or no chance of a continuing calibration reanalysis meeting criteria. If there is any doubt concerning which corrective action to undertake, consult the Department Manager or a senior chemist within the group.

If the calibration verification does meet the criteria specified above then analysis may proceed using initial calibration response factors.

7.6 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
- 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 necessarily be valid hits. It is the responsibility of the GC/MS analyst to use his/her technical judgment to determine if the identification of a target compound is correct or not.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the concentration of the largest target compound hit in the upper half of the initial calibration range. LCS and MS/MSD samples need not be diluted to get spiked analytes within the calibration range.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity > 10% must be present in the sample spectrum.
- The relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the

identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial review should be accomplished at the beginning of a work shift for the previous set of analyses.

7.7.3.1 Tentatively Identified Compounds (TIC)

TIC's may be requested by certain clients for samples. Refer current Katahdin to SOP CA-207 "GC/MS Library Search and Quantitation.

7.7.4 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into 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

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 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.

If the method blank exceeds these contamination levels, the analytical system is considered out of control and corrective action must be taken before sample analysis.

Reanalysis of the blank is the first step of the corrective action; if that does not solve the problem, a Katahdin Corrective Action Report (CAR) will be initiated.

Corrective action will be specified after consultation including the Department Manager, Operations Manager, and QA Officer.

8.2 Surrogate Recoveries

There are six surrogates, which can be divided as follows:

- B/N - Nitrobenzene-d5, 2-Fluorobiphenyl and Terphenyl-d14
- Acid - Phenol-d5, 2-Fluorophenol and 2,4,6-Tribromophenol

The surrogates have laboratory derived statistical limits that are updated on an annual basis and are available in the QA office.

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.

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).

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 judgements while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

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 over 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.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

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 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.

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TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

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	± 20 % D	1) Reanalyze standard 2) Reprep standard 3) Reprep standard from fresh stock.
Continuing calibration verification	Once per each 12 hours, prior to sample analysis	CCCs ≤ 20%D; 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 there after.	All recoveries within method QC acceptance limits.	Recalculate results; locate and fix problem; reextract/reanalyze P&A study for those analytes that did not meet criteria
Method blank	One per prep batch	No analytes detected > PQL	(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.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

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	Once per year	Ideally, PQL = at least 3 X MDL.	Repeat MDL study

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-204-09	Method 8270, current revision
Apparatus/Materials	none	
Reagents	none	
Sample preservation/ handling	none	
Procedures	none	
QC - Spikes	none	
QC - LCS	none	
QC - Accuracy/Precision	none	
QC - MDL	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

TABLE 3

Analyte Quantitation and Internal Standards

Internal Standard: 1,4-dichlorobenzene-d4

Target and Surrogates:

Pyridine
N-Nitrosodimethylamine (not on TCL list)
Aniline (not on TCL list)
Phenol
Bis (2-chloroethyl) ether
2-Chlorophenol
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
Benzyl alcohol (not on PP list)
2-Methylphenol (not on PP list)
2,2'-oxybis(1-chloropropane) (also known as Bis (2-Chloroisopropyl) ether)
4-Methylphenol (not on PP list)
N-Nitroso-di-n-propylamine
Hexachloroethane
Ethyl methanesulfonate (8270 C)
Methyl methanesulfonate (8270 C)
2-Picoline (8270 C)
N-Nitrosomethylethylamine (Appendix IX)
N-Nitrosodiethylamine (Appendix IX)
N-Nitrosopyrrolidine (Appendix IX)
N-Nitrosomorpholine (Appendix IX)
2-Fluorophenol (surrogate)
Phenol-d6 (surrogate)

Internal Standard: Naphthalene-d8

Target and Surrogates:

Nitrobenzene
Isophorone
2-Nitrophenol
2,4-Dimethylphenol
Acetophenone (8270 C)
Benzoic acid (not on PP list)
Bis (2-chloroethoxy) methane
2,4-Dichlorophenol
2,6-Dichlorophenol (8270 C)
1,2,4-Trichlorobenzene
a, a-Dimethyl-phenethylamine (8270 C)
Naphthalene
4-Chloroaniline (not on PP list)
Hexachlorobutadiene
4-Chloro-3-methylphenol
2-Methylnaphthalene
N-Nitrosodi-n-butylamine (8270 C)
N-Nitrosopiperidine (8270 C)
o-toluidine (Appendix IX)
o, o, o-Triethylphosphorothioate (Appendix IX)
Hexachloropropene (Appendix IX)
Isosafrole (Appendix IX)
Nitrobenzene-d5 (surrogate)

Internal Standard: Acenaphthene-d10

Target and Surrogates:

Hexachlorocyclopentadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol (not on PP list)
1-Chloronaphthalene (8270 C)
2-Chloronaphthalene
2-Nitroaniline (not on PP list)
Dimethyl phthalate
Acenaphthylene
3-Nitroaniline (not on PP list)
Acenaphthene
2,4-Dinitrophenol
4-Nitrophenol
Dibenzofuran (not on PP list)
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Diethyl phthalate
4-Chlorophenylphenyl ether
Fluorene
4-Nitroaniline (not on PP list)
1-Naphthylamine (8270 C)
2-Naphthylamine (8270 C)
Pentachlorobenzene (8270 C)

TABLE 3 (cont.)

Analyte Quantitation and Internal Standards

1, 2, 4, 5-Tetrachlorobenzene (8270 C)
2, 3, 4, 6-Tetrachlorophenol (8270 C)
p-Phenylenediamene (Appendix IX)
Safrole (Appendix IX)
1,4-Naphthoquinone (Appendix IX)
Thionazine (Appendix IX)
5-Nitro-o-toluidine (Appendix IX)
1,2-Diphenylhydrazine (not on TCL list)
2-Fluorobiphenyl (surrogate)
2,4,6-Tribromophenol (surrogate)

Internal Standard: Phenanthrene-d10

Target and Surrogates:

4,6-Dinitro-2-methylphenol
N-Nitrosodiphenylamine
Diphenylamine (8270 C)
4-Bromophenylphenyl ether
Phenacetin (8270 C)
Hexachlorobenzene
4-Aminobiphenyl (8270 C)
Pentachlorophenol
Pentachloronitrobenzene (8270 C)
Pronamide (8270 C)
Phenanthrene
Anthracene
Di-n-butylphthalate
Carbazole (8270 B)
Fluoranthene
Sym-Trinitrobenzene (Appendix IX)
Diallate (Appendix IX)
4-Nitroquinoline-1-oxide (Appendix IX)
Methapyrilene (Appendix IX)
Isodrin (Appendix IX)

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)

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

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 8270.

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	107,108,77,79,90
Bis(2-Chloroisopropyl)ether	45	77,121
4-Methylphenol	107	107,108,77,79,90
N-Nitroso-di-n-propylamine	70	42,101,130
Hexachloroethane	117	201,199
Nitrobenzene	77	123,65
Isophorone	82	95,138
2-Nitrophenol	139	65,109
2,4-Dimethylphenol	122	121,107
Benzoic acid	122	105,77
Bis(2-chloroethoxy)methane	93	95,123
2,4-Dichlorophenol	162	164,98
1,2,4-Trichlorobenzene	180	182,145
Naphthalene	128	129,127
4-Chloroaniline	127	129,65,92
Hexachlorobutadiene	225	223,227
4-Chloro-3-methylphenol	107	144,142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235,272
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,97,132,99
2-Chloronaphthalene	162	164,127
2-Nitroaniline	65	92,138
Dimethyl phthalate	163	194,164
Acenaphthylene	152	151,153
3-Nitroaniline	138	108,92
Acenaphthene	153	152,154
2,4-Dinitrophenol	184	63,154
4-Nitrophenol	109	139,65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63,89
2,6-Dinitrotoluene	165	89,63
Diethyl phthalate	149	177,150
4-Chlorophenylphenylether	204	206,141
Fluorene	166	165,167
4-Nitroaniline	138	92,108,65,80,39
4,6-Dinitro-2-methylphenol	198	105,51
N-Nitrosodiphenylamine	169	168,167
4-Bromophenylphenylether	248	250,141

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY CAPILLARY COLUMN GC/MS: SW 846 METHOD 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
Benzidine	184	92,185
Pyrene	202	200,203
Butylbenzylphthalate	149	91,206
3,3-Dichlorobenzidine	252	254,126
Benzo(a)anthracene	228	229,226
Bis(2-ethylhexyl)phthalate	149	167,279
Chrysene	228	229,226
Di-n-octyl phthalate	149	167,43
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,125
Indeno(1,2,3-cd)pyrene	276	138,277
Dibenz(ah)anthracene	278	139,279
Benzo(ghi)perylene	276	138,277
N-Nitrosodiethylamine	102	42,57,44,56
N-Nitrosopyrrolidine	100	41,42,68,69
N-Nitrosomorpholine	56	116,86
Acetophenone	105	71,51,120
2,6-Dichlorophenol	162	63,98
α,α-Dimethylphenethylamine	58	91,65,134,42
N-Nitrosodi-n-butylamine	84	57,41,116,158
N-Nitrosopiperidine	114	42,55,56,41
O-toluidine	106	107,77,51,79
O,O,O-Triethylphosphorothioate	198	121,97,65
Hexachloropropene	213	211,215,117,106,141
Isosafrole	162	131,104,77,51
1-Chloronaphthalene	162	127,164
1-Naphthylamine	143	115,89,63
2-Naphthylamine	143	115,116
Pentachlorobenzene	250	252,108,248,215,254
1,2,4,5-Tetrachlorobenzene	216	214,179,108,143,218
2,3,4,6-Tetrachlorophenol	232	131,230,166,234,168
p-Phenylenediamene	108	80,53,54,52
Safrole	162	104,77,103,135
1,4-Naphthquinone	158	104,102,76,50,130
Thionazine	107	96,97,143,79,68
5-Nitro-o-toluidine	152	77,79,106,94
4-Aminobiphenyl	169	168,170,115
Diphenylamine	169	168,167
Pentachloronitrobenzene	237	142,214,249,295,265
Phenacetin	108	180,179,109,137,80
Pronamide	173	175,145,109,147
sym-Trinitrobenzene	75	213,120

TABLE 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: 060708

JOB	SAMPLE	DATAFILE	DF	ALS #	METHOD	UL INJ	CHEMIST	COMMENTS
	50 ng DFTPP	U0626	1	1	DFTPP390	2.0	JW	OK
B	SSTD050U0107	U0563		2	U8270C02	1.0		✓
	010	64		3				✓
	025	65		4				✓ Curve OK
	100	66		5				✓
	150	67		6				✓
	200	68		7				✓
A	SSTD050U0107	69 (a)		8				✓ (a) = UTCLP02
	010	70 (a)		9				✓ files
	025	71 (a)		10				✓
	100	72 (a)		11				✓
	150	73 (a)		12				✓
	200	74 (a)		13				✓
	8270 IND CHK	75 (a)		14				
				JW 060708				

STANDARD	CODE
DFTPP	S1176
CAL. STD.	S1181 S1182
IS MIX	STC 3505

REVIEWED AND APPROVED BY: _____
DATE: _____

QAMS364

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:

AMP0946

AccuStandard 125 Market St. • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com

APP-9-176-D-20X
Pentachlorophenol
2.0 mg/mL in CH2Cl2
Lot: B3010100
Exp. Jan 10, 2013

1 mL

FOR LABORATORY USE ONLY
WARNING: This product contains a chemical(s) known to the State of California to cause cancer.

STORAGE Ambient

POISON [redacted]

Dual 3/16/06 JZG

AMP0947

AccuStandard 125 Market St. • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com

APP-9-090-50X
4,6-Dinitro-o-cresol
5.0 mg/mL in MeOH
Lot: B1100296
Exp. Aug 16, 2012

1 mL

FOR LABORATORY USE ONLY

STORAGE Ambient

FLAMMABLE [redacted]

AMP0948

AccuStandard 125 Market St. • New Haven, CT 06513 • USA Tel. 203-786-5290 • www.accustandard.com

APP-9-145-50X
p-Nitrophenol
5.0 mg/mL in MeOH
Lot: B5050205
Exp. May 18, 2015

1 mL

FOR LABORATORY USE ONLY

STORAGE Ambient

POISON [redacted]

QAMS294

0000045

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 MeOH)	3-15-06	7-7-06	JLW	AMP0844	8270 Mixes	300	2-22-07	4.0ml	150 ng/ul
					AMP0837	↓	300	3-15-07		
					AMP0911	App IX #2	600	3-2-07		
					AMP0910	↓ 1	100	3-9-07		
					AMP0570	↓ 1	200	7-7-06		
					AMP0699	Organophosphorus	300	8-19-06		
					AMP0878	Benzoic Acid		3-9-07		
					AMP0807	Hexachlorophene		2-22-07		
					AMP0804	Benzidine		3-9-07		
					AMP0836	3,3'-Dichlorobenzidine	↓	3-14-07		
					AMP0932	8270 Surr	150	3-9-07		
					50861	DEA	300	3-13-07		
					B435290	MeCl2	550	~		
50864	8270 level 1	3-15-06	7-7-06	JLW	50863	8270 Stock	70	7-7-06	1.05ml	10 ng/ul
					B435290	MeCl2	980			
50865	8270 level 2	3-15-06	7-7-06	JLW	50863	8270 Stock	150	7-7-06	0.90ml	25 ng/ul
					B435290	MeCl2	750			
50866	8270 level 3	3-15-06	7-7-06	JLW	50863	8270 Stock	600	7-7-06	1.8ml	50 ng/ul
					B435290	MeCl2	1200			
50867	8270 level 4	3-15-06	7-7-06	JLW	50864	8270 Stock	700	7-7-06	1.05ml	100 ng/ul
					B435290	MeCl2	350			

Reviewed by/Date:

CAMS284

00000057

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

Prepared By: GC/MS Department Date: 6/98

Approved By:

Group Supervisor: [signature] Date: 020101

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

QA Officer: Deborah J. Nadeau Date: 1.31.01

General Manager: [signature] Date: 2/01/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8270C Mod.	Format changes, added pollution prevention, added instrument and other calibration options. Other minor changes to sections 7, 8 & QA Table.	DN	1.31.01	1.31.01
02 8270C	Many changes in formatting. Some additions to section 8 + Table 1 to comply with Navy.	DN	09.30.04	09.30.04
03 8270C	Sect. 7.2: Removed "K" Instrument & added "R" instrument. Added Pentafluorophenol surr. to Tables 3, 5 and Sect. 8.2. Removed all References to TIC's.	LAD	04/06	04/06
04 8270C	Sect. 8.2 - changed 5 to 4 and removed pentachlorophenol. Table 3 and 5 - removed pentachlorophenol. Changed linear regression correlation coefficient criteria. Added M1 SOP reference. Added LCS exceedance criteria. Added ICV requirement and criteria. Added RT window procedure.	LAD	06/07	06/07
05 8270C	Added "G" instrument, Removed "X" instrument Edited section 7.5.1 - initial cal. table	LAD	02/08	02/08

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

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-213-05, 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-213-05, titled "ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)".

Recipient: _____ Date: _____

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

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel to prepare and analyze water and soil sample extracts for semivolatile organics by EPA SW-846 Method 8270, current revision, modified for selected ion monitoring.

In order to maintain consistency in data quality, this SOP consolidates all aspects of the analyses in one working document, to be revised as necessary.

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

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

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of semivolatile organic compounds by EPA Method 8270. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of semivolatiles by Method 8270 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Management Plan and follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves, and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, autosampler vials containing sample extracts in methylene chloride are returned to the SVOA hood, and the contents transferred to a labeled waste container. The contents of this container are disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

The process involves the extraction of semivolatiles from a sample using an appropriate solvent followed by clean up steps (where applicable) and concentration of the extract (refer to Katahdin SOP CA-502, "Preparation of Aqueous Samples for Extractable Semivolatile Analyses", SOP CA-512, "Preparation of Sediment/Soil Samples by Sonication Using Method 3550 for Subsequent Extractable Semi-Volatiles Analysis" and SOP CA-526, "Preparation of Sediment/Soil Samples by Soxhlet Extraction Using Method 3540 for Subsequent Extractable Semivolatile Analysis"). An aliquot of the final extract is injected into the gas chromatograph

for compound separation by capillary column, followed by the electron impact mass spectrometer for identification and quantitation.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of SVOCs is analyzed immediately after a sample containing high concentrations of SVOCs. Any samples that have suspected carryover must be reanalyzed.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 5890 and/or 6890

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

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be onstructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Autosamplers: HP 7673As

4.5 Hamilton syringes: 2.00 uL to 10 mL

4.6 Volumetric glassware: Grade A or equivalent

4.7 Columns: DB-5MS 30m, 0.25mm I.D., 25um film thickness, columns (J&W Scientific) or equivalent.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

5.0 REAGENTS

5.1 J.T. Baker Ultra Resi-Analyzed methylene chloride (or equivalent)

5.2 Purge and trap grade methanol

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 "Standard Preparation and Documentation".

5.3.1 The expiration date for all standards is one year from date of opening the ampule. If the manufacturer's expiration date is before this one year date, the manufacturer's expiration must be followed. New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

5.3.2.1 The standards are prepared 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 – 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 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.

5.3.2.4 DFTPP Solution – Prepare standard in methylene chloride containing DFTPP at a final concentration of 25 ug/mL.

5.3.2.5 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 10 ug/mL.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All semivolatile sample extracts must be analyzed within forty days following the date of extraction.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 "Standard Preparation and Documentation".

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition and storage: C:\HPCHEM\1\DATA
Tune file: DFTPP.U

Method files: LSIMXX.M (all samples and standards)
Where:
XXX = 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 LSIMXXX.M and DFTPP2. M.

Data Files: L_ _ _ _.D, where _ _ _ _ is a number in chronological order from 0001 to 9999 and L is the instrument ID (R, U, or G). This file also contains the Quantitation output file.

Data Files for DFTPP: LD_ _ _.D, where _ _ _ is a number in chronological order from 001 to 999 and L is the instrument ID (R, U, or G).

7.3 INSTRUMENT SPECIFIC PROCEDURES

It is the policy of the GC/MS group that all data be acquired in the batch mode. The following items must be checked prior to data acquisition in the batch mode:

- Ensure that the proper sequence and tune files are being used.
- Check the autosampler syringe (Is it clean? Does the plunger move freely? etc.), its alignment and make sure the solvent rinse vial is full. Ensure that the knurled nut holding the top of the syringe plunger is tight.
- Look at the batch to be analyzed and check the following:

  -Make sure that the data files are in numerical order with no duplication and that the method file is the same as that used for ICAL or Continuing Calibration analysis.

  -Bottle numbers match with the numbers on the autosampler tray.

After the batch has been deemed free of errors, start the batch by using the “Position and run” command under the SEQUENCE menu in MSTop.

7.4 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks or samples, the GC/MS system must be shown to meet the mass spectral key ion and ion abundance criteria for decafluorotriphenylphosphine (DFTPP) tabulated below. Pentachlorophenol, benzidine and DDT are also present in this standard.

DFTPP Key Ions and Ion Abundance Criteria	
Mass	**Criteria**
51	30.0-80.0 percent of mass 198
68	less than 2.0 percent of mass 69
69	present
70	less than 2.0 percent of mass 69
127	40.0 – 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent of mass 198
199	5.0-9.0 percent of mass 198
275	10.0-30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present, but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0-23.0 percent of mass 442

All ion abundances must be normalized to m/z 198, the nominal base peak.

The following are the GC/MS operating conditions for injection of DFTPP.

GC/MS OPERATING CONDITIONS - DFTPP	
Initial column temperature hold	140°C for 3 minutes
Column temperature program	140-260°C at 10 degrees/minute
Final column temperature hold	275°C
Injection port temperature	250°C
Transfer line/source temperature	285°C
Injector - splitless, valve time	0.18 minutes
EPC	inlet B
Constant flow	ON
Constant flow pressure	10psi
Constant flow temperature	30°C
Vacuum comp.	ON
Run time	10-12 minutes
Scan start time	5.0 minutes
Sample volume	2.0 uL of 25 ng/uL DFTPP solution

GC/MS OPERATING CONDITIONS – DFTPP (CONT.)	
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: 0.20, 0.50, 1.0, 3.0, 6.0 and 10.0 ng/uL. This is done to determine instrument sensitivity and the linearity of GC/MS response for the semivolatile target and surrogate compounds.

Final conc. (ng/uL)	20 ng/uL SI M PAH Stock Added (uL)	20 ng/uL HCB/BEHP/ PCP Stock Added (uL)	$MeCl_2$ Added (uL)	Final Volume (uL)
0.20	10	50	940	1000
0.50	25	100	875	1000
1.00	100	200	700	1000
3.00	150	300	550	1000
6.00	300	400	300	1000
10.0	500	500	0	1000

If additional compound mixtures are added, the volume of $MeCl_2$ is adjusted to maintain a final volume of 1000 uL. A 100 uL aliquot of each of the standards above is spiked with 1 uL of internal standards and analyzed.

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 320°C at 10°/min.
Final column temperature hold	320°C
Run time	35 minutes (time may vary dependent upon column length)
Scan start time	6.0 minutes (time may vary dependent upon column length)
Sample volume	1 uL

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

After analysis of the five calibration points, they must be quantitated and evaluated for adherence to QC criteria. Minimum requirements of ID files are the use of specific quantitation ions and quantitating a specific set of targets and surrogates with a set internal standard. Of particular importance when performing SIM analysis are the ion ratios. These requirements are found in Tables 3 and 5.

7.5.2 Initial Calibration Criteria

Relative response factors (RRFs) must be calculated and evaluated for each target compound and surrogate. The RRF is defined as follows:

$$RRF = \frac{Ax}{A_{IS}} \times \frac{C_{IS}}{Cx}$$

where:
- Ax = area of the primary ion for the target compound
- A_{IS} = area of the primary ion for the corresponding istd
- C_{IS} = concentration of the istd (ng/uL)
- C_x = concentration of the target compound

After the calibration points have been quantitated, update the calibration curve points using the Target data processing software to generate the RRF's and %RSD's for all analytes. If information is needed concerning the use of these programs, consult the Organic Department Manager or a senior chemist within the group.

Response factor criteria have been established for the calibration of the semivolatile target and surrogate compounds. These criteria must be met in order for the calibration curve to be considered valid. The percent RSD for each calibration check compound (CCC) must be less than or equal to 30 percent. There are three CCC's: Acenaphthene, Fluoranthene, and Benzo(a)pyrene. There are no criteria for the SPCC compounds.

7.5.2.1 Linearity of Target Analytes

If the RSD of any target analyte is 15% or less, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15%, then a calibration option outlined in section 7.0 of method 8000 will need to be employed.
Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration.

The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. Target software calculates the correlation coefficient squared (r^2). This must be equal to or greater than 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.990.

If time remains in the clock after meeting the initial calibration acceptance criteria, samples may be analyzed. The calibration must be verified each twelve hour time period (time period starts from the moment of the DFTPP injection) for Method 8270-SIM. The SSTD1.0 in the curve may be used as the continuing calibration standard as long as it meets the continuing calibration acceptance criteria. All sample results must be quantitated using the initial calibration response factors.

7.5.3 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.

If the continuing calibration check does not meet criteria, corrective action must be taken. Depending on the situation, corrective action may be as follows:

- Re-analyze the 1.0 ng/uL continuing calibration check.
- Change the septum; clean the injection port; install a clean, silanized quartz liner; cut off a small portion (1" to 3") of the front end of the capillary column. This is usually performed when chromatography is poor.
- Analyze a new initial calibration curve.

The last option, the generation of a new initial calibration curve, is usually chosen when percent difference are >30%. In these instances, there is little or no chance of a continuing calibration reanalysis meeting criteria. If there is any doubt concerning which corrective action to undertake, consult the Organic Department Manager or a senior chemist within the group.

If the continuing calibration does meet the criteria specified above then analysis may proceed using initial calibration response factors.

7.5.4. Retention Time Windows

Retention time windows are set at the midpoint standard of the calibration curve, following every ICAL. When a CV is analyzed (and not an ICAL), the retention time windows of the daily CV must be within 30 seconds of the midpoint calibration standard of the most recent ICAL. The samples analyzed following the daily CV must have retention times within 30 seconds of those for the daily CV. Each successive daily CV must be compared to the most recent ICAL midpoint standard.

7.6 SAMPLE ANALYSIS

Sample extracts may be analyzed only after the GC/MS system has met tuning criteria, initial calibration and continuing calibration requirements. Ensure that the same instrument conditions are being used for tuning, calibration and sample analysis by reviewing the GC parameters using the "Edit entire method" option under the Method menu in MSTOP. Note that you can not edit a method if the instrument is running.

Extracts are stored in the refrigerator in the organics extraction laboratory at 4°C ±2°C. Remove them from the refrigerator and place them in the GC/MS laboratory semivolatile hood when ready for analysis.

Prepare a 1.8 mL clear glass vial (crimp top) with a disposable insert (350 uL). Add 100 uL of sample extract and 1.0 uL of the 80 ng/uL IS stock to the vial and then cap. This gives a 0.8 ng/uL final concentration for the internal standard compounds. The samples are topped with Teflon lined crimp top caps.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

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

- Surrogate Recoveries
- Internal Standard Area Stability
- Method Blank Acceptance

- Chromatography
- Target Compound Detection/Quantitation/Review for false positives
- Laboratory Control Sample Recoveries
- Matrix Spike/Matrix Spike Duplicate Recoveries

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed. During this review, the analyst checks the integration of each individual peak. The hardcopy has false positives crossed out so they can be reviewed for appropriateness by the Organic Department Manager.

7.7.2 Chromatography

The chromatography should be examined for the presence or absence of any ghost peaks and can also be used as an indication of whether or not matrix interferences might be affecting surrogate recoveries and/or istd area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g. surrogate recoveries) to determine the necessity of reanalyzing.

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

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

This manual integration package must then be submitted to the Department Manager or his/her designee, who will review each manual integration.

For specific manual integration procedures, refer to Katahdin SOP QA-812, “Manual Integration”, current revision.

7.7.3 Target Compound Detection/Quantitation

The semivolatile ID files have been set up to err on the side of false positives; that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits. It is the responsibility of the GC/MS analyst to use his/her technical judgment to determine if the identification of a target compound is correct or not.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the concentration of the largest target compound hit in the upper half of the initial calibration range. LCS and MS/MSD samples need not be diluted to get spiked analytes within the calibration range.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity > 10% must be present in the sample spectrum.
- The relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial review should be accomplished at the beginning of a work shift for the previous set of analyses.

7.7.4 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC label requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the

necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A complete review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

7.8 INJECTION PORT LINER CLEANING AND SILANIZING PROCEDURE

- Remove the rubber o-ring from the liner and place the liner in a large Erlenmeyer flask.
- In the hood, pour nitric acid into the flask until the liner is covered. Place the flask on a hotplate and boil for 2-3 hours.
- Let cool; drain nitric acid and thoroughly flush the liner with water.
- Bake briefly in the muffle oven until liner is dry and cool to room temperature.
- Place the liner in a beaker, fill with Sylon and let it soak for at least two hours.
- Take out the liner and rinse it thoroughly with toluene.
- Rinse the liner thoroughly with purge and trap grade methanol.
- Bake the liner in the muffle oven for a minimum of three hours.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

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

8.1 Method Blank Criteria

A method blank is defined as a volume of a clean reference material (deionized distilled water for water samples, baked organic-free sand for soil/sediment matrices) that is carried through the entire analytical procedure. One method blank must be extracted with each group of samples of a similar matrix and must be analyzed on the GC/MS system that was used to analyze the samples.
An acceptable method blank must contain less than or equal to the PQL of any target compound.

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-Dibromo-phenol, 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%.

If specifications are not met, the sample (or blank) should be reanalyzed. If specifications are met in the reanalysis, this reanalysis should only be submitted. If surrogate specifications are not met in the sample or method blank reanalysis, a Corrective Action Report (CAR) should be initiated. Corrective action will be specified after consultation including the Department Manager and Operations Manager.

For further information regarding the acceptance of surrogate recoveries, consult the Organic Department Manager.

8.3 Internal Standard Responses

Internal standard responses and retention times (RT) in all samples and blanks must be evaluated as part of the technical data review. The method files have been set up to only detect compounds that fall within a set RT window. For Method 8270-SIM analysis, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the daily continuing calibration standard, reanalysis must occur. If the reanalysis meets criteria, only the in-criteria run should be reported. If the reanalysis is still out of criteria, both analyses should be included in the sample package set.
MS/MSD samples that do not meet the EICP area criteria above do not have to be reanalyzed.

8.4 Laboratory Control Sample (LCS)

An LCS must be performed for each group of samples of a similar matrix, for the following, whichever is more frequent:

- Every 20 samples of a similar matrix or similar concentration, or
- Every batch of samples extracted.

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the section supervisor, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts", current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 30-150% may be used for some projects or states (i.e. South Carolina).

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits with the following sporadic exceedance allowances.

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time

Please note that for compounds with only nominal limits (i.e. insufficient data points were available to generate statistical limits), no corrective action is required for out-of-criteria recoveries until enough data points are established to generate statistical limits.

8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria

Matrix Spike and Matrix Spike Duplicates must be extracted and analyzed for each group of up to 20 samples of a similar matrix or similar concentration. In the event insufficient sample volume is available an LCS/LCS Duplicate is extracted and analyzed in place of the MS/MSD.

Statistical limits are compiled annually for MS/MSD recoveries for a short list of the spiked compounds (Acenaphthene, Pentachlorophenol and Pyrene). Nominal limits of 30-130% are used for all other compounds. Generally, corrective action is only taken for the short list of the spiked compounds. The specific corrective actions will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

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.

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

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 current revision of Method 8270 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 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.

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

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.4	Retune instrument, and verify
Six-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD ≤30 for RFs of the CCCs; Average %RSD < 15% for all compounds. Refer to section 7.5.2.1 for more details.	Repeat calibration if criterion is not met
Independent calibration verification	Once after Initial calibration	± 20 % D	1) Reanalyze standard 2) Reprep standard 3) Reprep standard from fresh stock.
Continuing calibration verification	Once per each 12 hours, prior to sample analysis	CCCs ≤ 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
ISs	Immediately after or during data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Demonstration of ability to generate acceptable accuracy and precision	Once per analyst initially and annually thereafter	All recoveries within method QC acceptance limits.	Recalculate results; locate and fix problem; reextract/reanalyze P&A study for those analytes that did not meet criteria
Method blank	One per prep batch	No analytes detected > PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e.If the blank results are above the PQL, report samples that are <PQL or > 10X the blank result. Reprep a blank and the remaining samples.
LCS for all analytes	One LCS per prep batch	Statistically derived from lab data or nominal limits depending on the project. . See also section 8.4 of this SOP for more information on allowable exceedances.	(1) Evaluate the samples and associated QC: i.e.If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS rec. is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.

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	Once per year	Ideally, PQL = at least 3xMDL	Repeat MDL study

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

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-213-05	METHOD 8270, current revision
Apparatus/Materials	none	
Reagents	none	
Sample preservation/ handling	none	
Procedures	none	
QC - Spikes	none	
QC - LCS	none	
QC - Accuracy/Precision	none	
QC - MDL	none	

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

TABLE 3

ANALYTE QUANITIATION AND INTERNAL STANDARDS

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

`TABLE 4

PROCEDURE CONDENSATION

Clock

12 hours from injection of 50ng DFTPP.

Calibration Curve Criteria

<30% RSD for CCCS
<15% RSD average for all analytes in calibration standard

Continuing Calibration Check Criteria

<20% D for CCC compounds

Additional QC

LCS every extraction batch
MS/MSD every 20 samples

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

TABLE 5

SVOA COMPOUNDS AND CHARACERISTIC IONS

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

Primary ions must not be changed except in unusual instances where interference occurs with a co-eluting non-target analyte. In this case, a secondary ion may be used for quantitation with the following rules:

(1) The corresponding standard(s) (initial calibration curve and continuing calibration standard) must be re-quantitated with the secondary ion.

(2) Approval must be obtained from the Organic Department Manager or the laboratory Operations Manager.

The quantitation ion must then be changed back to the one specified in the table above after quantitation of the samples(s).

Secondary ions are recommended only and may be changed depending upon instrument conditions (sensitivity, etc.). However, it is Katahdin policy that a minimum of 2 ions (primary and one secondary) be used for all GC/MS analyses.

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

FIGURE 1

EXAMPLE OF RUNLOG LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES
GC/MS SVOA INJ LOG INSTRUMENT: 5970-X

DATE OF DFTPP INJECTION: 031009

JOB	SAMPLE	DATAFILE	DF	ALS #	METHOD	UL INJ	CHEMIST	COMMENTS
	50 ng DFTPP	X01600	1	1	DFTP0390	2.0	JK	OK
	SSTD060X0310	X9437		2	X625A022	1.0		✓
	150	38		3				✓
	100	39		4				✓ curve JK
	30	40		5				✓
	05	41		6				✓
	3510 MIX 1	42		7				OK
	2	43		8				
	3	44		9				
	4	45		10				
	5	46		11				
	6	47		12				
	7	48		13				
	8	49		14				
	9	50		15				
	BLANK 1	51		16				
	2	52		17				

STANDARD	CODE
DFTPP	50856
CAL. STD.	5089-30 5084-49
IS MIX	AHP0934

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:

AMP0946

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

Dent 8/16/06 JLG

AMP0947

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

AMP0948

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

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

FIGURE 3

EXAMPLE OF SVOA STANDARDS PREPARATION LOGBOOK ENTRY

GC/MS SVOA STANDARD PREP LOGBOOK

50863	8270 Stock (w/o MeOH)	3-15-06	7-7-06	JLK	AMP0844	8270 Mixes/Surr	300	2-22-07	4.2ml	150 ng/ml
					AMP0837	↓	350	3-15-07		
					AMP0911	APP IX #2	600	3-2-07		
					AMP0910	↓ 1	160	3-9-07		
					AMP0570	↓ 1	200	7-7-06		
					AMP0699	Organophosphorus pest	300	8-19-06		
					AMP0871	Benzoic Acid	↓	3-9-07		
					AMP0867	Hexachlorophene		2-22-07		
					AMP0806	Benzidine		3-9-07		
					AMP0936	3,3'-Dichlorobenzidine	↓	3-14-07		
					AMP0932	8270 Surr	150	3-9-07		
					50861	DEA	300	3-13-07		
					B43590	MeCl2	550	—		
50864	8270 level 1	3-15-06	7-7-06	JLK	50863	8270 Stock	70	7-7-06	1.05ml	10 ng/ml
					B43590	MeCl2	980			
50865	8270 level 2	3-15-06	7-7-06	JLK	50863	8270 Stock	150	7-7-06	0.90ml	25 ng/ml
					B43590	MeCl2	750			
50866	8270 level 3	3-15-06	7-7-06	JLK	50863	8270 Stock	600	7-7-06	1.8ml	50 ng/ml
					B43590	MeCl2	1200			
50867	8270 level 4	3-15-06	7-7-06	JLK	50864	8270 Stock	700	7-7-06	1.05ml	100 ng/ml
					B43590	MeCl2	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. Burton Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1.22.01

General Manager: Dennis P. Kufrak 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	03/05 LAD	03/05 6-13-06

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

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
07	Updated R.T. window criteria Sect. 7.4.2 - Added to shake samples before vialing	LAD	03/06	03/06
08	Sect. 1.4 - updated to include waste streams sect. 4.5 - removed balance changed makeup gas from N_2 to Ar Me. Changed T.C. calibration from 5 to 2 peaks. Updated 2nd column conf. Changed corr. coef. to coeff. of determination.	LAD	06/07	06/07

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-302-08**, 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-302-08**, 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 8081, current revision, as performed by Katahdin Analytical Services, Inc. including sample analysis, data review, standard preparation and instrument calibration.

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

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; however a universal blank matrix does not exist for solid samples, and therefore, no matrix is used. The blank is taken through the appropriate steps of the process.

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

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution, which is used to calibrate the instrument response with respect to analyte concentration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS) : A complete multiuser system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

PE NELSON TURBOCHROM: A data acquisition system that is used to collect chromatographic data. The system can also be used to archive raw data files.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

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

1.2 Responsibilities

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

It is the responsibility of all Katahdin technical personnel involved in analysis by method 8081, current revision, to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Health and Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical 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 Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

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 connected to the Turbochrom or Enviroquant data system,or equivalent.

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

4.1.3 Detectors: Electron capture detectors (ECD).

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

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

4.4 Vials: various sizes and types including crimp tops.

4.6 Refrigerator for storage of extracts and standards.

5.0 REAGENTS

5.1 Solvents

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

5.2 Standards

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

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

5.2.3 Pesticide Working standards: Prepared by diluting the stock mix of 2000 ug/ml that contains all single component pesticides into hexane to give final concentrations of: 0.005, 0.01, 0.025, 0.05, 0.10, and 0.25 ug/ml. The mix,

referred to as INDAB, also contains two surrogates: Tetrachloro-m-xylene and Decachlorobiphenyl, which are at the same concentrations as the pesticides.

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

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

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

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

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

7.0 PROCEDURES

EXTRACTION - Refer to the appropriate SOP for the correct extraction procedure. In general, water samples are extracted using methods 3510 or 3520 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 c alibration 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 heights of the 5 to 10 peaks are averaged. A calibration curve is prepared in Target using the average of the peak heights of the 5 to 10 peaks against the concentration of the standard.

Technical Chlordane is calibrated using the 2 major peaks of the standard. The heights of the 2 peaks are averaged. A calibration curve is prepared in Target using the average of the peak heights of the 2 peaks against the concentration of the standard.

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

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

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

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

$$y = bx + c$$

where: y = Instrument response
b = Slope of the line

x = Concentration of the calibration standard
c = The intercept

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

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

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

7.3 RETENTION TIME WINDOWS

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

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

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

7.3.4 Retention time windows are calculated for each standard on each GC column and whenever a new GC column is installed. The data is kept on file in the laboratory.

7.3.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being too narrow. The windows are: ± 0.05 for Heptachlor, Aldrin and all BHC compounds, ± 0.07 for all other target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive by carefully evaluating the chromatograms. Please note that the use of nominal

retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.

7.4 GAS CHROMATOGRAPHIC ANALYSIS

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

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

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

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

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

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

7.4.2 Gently shake sample extracts before vialing for analysis.

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

7.4.4 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration as listed in section 7.2 followed by sample extracts interspersed with mid-concentration calibration standards. Before any samples are analyzed the instrument must be calibrated by

analyzing a six-point calibration or a 0.05ppm concentration standard (calibration verification standard). If a CV is run, the calculated concentration must not exceed a difference of ± 15%. 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 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.

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

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

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

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

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

7.5 Data Review

7.5.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed samples. This

data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

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

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

7.5.2 Surrogate recovery

All recoveries must meet the most recently laboratory established acceptance limits, which are listed on the Laboratory Surrogate Acceptance Limit sheet.

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

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

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

7.5.3 Chromatography

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

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

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an "m" qualifier will automatically be printed on the quantitation report summary. The analyst will date and initial the "m" on the quanitation report summary and assign a code that indicates the reason for the manual integration. Refer to Katahdin SOP QA-812 "Manual Integration on GC/MS, GC, HPLC and IC Datasystems" for more information.

7.5.4 Target Compound Detection

GC analysis relies heavily on the experience of the analyst. Sample chromatograms must be evaluated focusing on scientific judgment, knowledge of the column behavior and matrix effects. The chromatogram from channel A is evaluated with that from channel B. If a target analyte is present on both channels and the concentration is within the calibration range, and the quantitation from both chromatograms agrees within ±40%, the analyte is considered to be present in the sample. In cases where the RPD is greater than 40% and the analyte is reported, the analyte must be J-flagged indicating that the result is an estimated value. The higher of the two concentrations is reported unless matrix interference is causing erroneously high results. In this case report the lower result and narrate. Sometimes interference on one column (i.e. sulfur) will prevent a target analyte from detection and it is present on the conformational column. In this scenario, the

result would be reported from one column and need to be "Q" flagged to indicate that it was not confirmed on a second column.

All flagged data must be discussed in the narrative

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

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

7.6 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A completed review checklist (Figure 2) is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time

considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

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

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

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

	WATER ug/L	SOILS ug/Kg
Pesticides	0.50	16.7

The surrogate spike concentrations in the final extract are:

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

8.3 LCS and MS/MSD acceptance criteria and Corrective Action: All QC samples are calculated for percent recovery of the spiked analyte(s). The recoveries are compared to laboratory established acceptance limits. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision.

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 mayvary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

If a spike compound is outside of the acceptance limits in the matrix spike sample but is acceptable in the LCS, the data is considered acceptable. The cause of the failure is possibly attributable to matrix interference. However, if the compound fails in both the LCS and the MS/MSD, the result for that analyte is suspect and may not be reported for regulatory compliance purposes.

Please note that established acceptance limits that are wider than 70-130% may not be allowable for certain states, federal programs, or clients. For South Carolina, the acceptance limits for the spiked analytes will be 70-130% or narrower.

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.

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.

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

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 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. Promulgated Update III dated December, 1996, Method 8081A.

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.

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

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch of twenty or fewer samples	No analyte detected >PQL	(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	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are < PQL, narrate. Otherwise, reprep a blank and the remaining samples.
CCV	If calibration curve previously analyzed, analyze daily before samples and after every 10 samples.	± 15% D	(1) Evaluate the samples: If the %D>+15% and sample results are <PQL, narrate. If %D>±15% only on one channel, narrate. If %D>±15% for the closing CV, and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples back to last acceptable CV.
Matrix Spike\ Matrix Spike Duplicate	One for every set of 20 samples	Same as for LCS	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.
6 pt of INDAB mix with mid-pt cal of Toxaphene and Chlordane	Initial cal prior to sample analysis	6pt calibration coefficient of determination ≥ 0.990	(1) Repeat Initial calibration (2) If single pt cal Toxaphene, or Chlordane is identified in analysis of sample, 6 pt calibration run of identified compound with reanalysis of sample.
Independent calibration verification	Once after Initial calibration	± 20 % D	Reanalyze standard Reprep standard Reprep standard from fresh stock.
Demonstrate ability to generate acceptable P & A using 4 replicate analyses of a QC check standard	One time per analyst initially and annually thereafter.	All recoveries within method QC acceptance limits	Recalculate results; locate and fix problem; rerun P & A study for those analytes that did not meet criteria prior to sample analysis
MDL study	Once per year	Ideally, PQL = at least 3 * MDL..	Repeat MDL study

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

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-302-08	METHOD 8081, current revision
Apparatus/ Materials	None	
Reagents	None	
Sample preservation/ handling	None	
Procedures	7.3.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.05 for Heptachlor, Aldrin and all BHC compounds, ± 0.07 for all other target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.	7.6.3 If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. (Recording retention times to three decimal places rather than only two should minimize the instances in which the standard deviation is calculated as 0.000).
QC - Continuing Calibration	None	
QC - LCS	None	
QC - Accuracy/Precision	None	
QC - MDL	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL

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

FIGURE 1

EXAMPLE OF INSTRUMENT RUN LOG

Katahdin Analytical Services, Inc.
Instrument: GC08
Amount Injected 2ul

Reviewed by/ Date:

GC Laboratory Instrument Runlog

Method: 608 / 8081 / 8082
(circle)

Date	Init.	Result File	Sample ID	Y/N	Method	Column	Comments
6.13.07	SK	8AF1/2 154	WG39913-1 3510	Y	PSTA/B274A	293/294	
		155	-2				
		156	-3				
		157	-4				TCLP Blank
		158	SA2894-1 3550				
		159	-2				
		160	SA2750-1 3510				
		161	Hexane	N			
		162	INDAB 0.025ppm	Y			P4228
		163	SA2799-7 3510	Y			
		164	SA2883-2 3550				
		165	SA2753-1 3510				
		166	-2				1DCB on B
		167	SA2799-2 3550	N			needs SC
		168	-3				
		169	-4				
		170	-5				
		171	-6	Y			
		172	-1				
		173	Hexane	N			
		174	INDAB 0.05ppm	Y			most low P4229
6.18.07		175	Hexane	N			
		176	Prime				
		177	EWL	Y			P4188
		178	INDAB 0.05ppm	N			most low P4229
		178A	INDAB 0.05ppm				
		179	0.005ppm	Y	PSTA/B275A		P4226
		180	0.01				P4227
		181	0.025				P4228
		182	0.05				P4229

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

FIGURE 2

DATA REVIEW CHECKLIST

Verbal Due Date _____ Due Date_____

Client:		Primary	Secondary
Method:		Date:	Date:
SDG No:	Level:	Initials:	Initials:
KAS No:			Approved : ☐ Yes

PRIMARY REVIEW CHECKLIST

Highlight Method / project specific information. _____

All needed forms are present . _____

Sample Data Summary Included (Level III & IV). _____

Correct Work Order Number or SDG name (all forms). _____

Correct project name and spelling (all forms). _____

Correct file numbers (all forms). _____

Analysis Date Correct. _____

Extraction Method & Analysis Method Correct. _____

Product list compared to ROAs (compounds & PQLs). _____

Chromatogram reviewed for unlabeled peaks (check product list). _____

Flagging of all ROAs correct (Florida Flagging ☐). _____

All runes included (level IV) . _____

All log book pages included (Soil weights,TCLP & SPLP). _____

Verify quant results for CLP. _____

Update sample history files. _____

Sign & Date Manual integration (Narrate as needed). _____

Sample I.D's Truncated (NARRATE). YES ☐ Please list KAS # below :

_____

First correction ☐ → Review and replace appropriate SDS Forms ☐.

Second correction ☐ → Review and replace appropriate SDS Forms ☐

Last saved by kasgc04/13/07

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

FIGURE 3

PQLS FOR METHOD 8081

Parameter/Method	Analyte	Practical Quantitation Level (PQL)	
		Waters (ug/L)	Soils (ug/kg)
Organochlorine	Aldrin	0.05	1.7
Pesticides	Alpha BHC	0.05	1.7
	Beta BHC	0.05	1.7
SW3510/SW8081A (W)	Delta BHC	0.05	1.7
SW3520/SW8081A (W)	Gamma BHC (Lindane)	0.05	1.7
SW3550/SW8081A(S)	Chlordane	0.50	17
	alpha-Chlordane	0.05	1.7
	gamma-Chlordane	0.05	1.7
	4,4'-DDD	0.10	3.3
	4,4'-DDE	0.10	3.3
	4,4'-DDT	0.10	3.3
	Dieldrin	0.10	3.3
	Endosulfan I	0.05	1.7
	Endosulfan II	0.10	3.3
	Endosulfan Sulfate	0.10	3.3
	Endrin	0.10	3.3
	Endrin Aldehyde	0.10	3.3
	Endrin Ketone	0.10	3.3
	Heptachlor	0.05	1.7
	Heptachlor Epoxide	0.05	1.7
	Methoxychlor	0.50	17
	Toxaphene	1.00	33

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. Burton Date: 1/24/01

QA Officer: Deborah J. Nadeau Date: 1.24.01

General Manager: Dennis F. Hufnagle 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.	HRC	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 dnt. 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

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added waste stream to Sect. 1.0. Added N_2 gas as detector gas. Updated 2nd Column criteria & added flagging criteria. Changed correlation coefficient to Coefficient of determination in Sect. 7.2.1 and Table 1.	LAD	08/07	08/07

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-305-06**, titled **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-305-06**, titled **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

Recipient: _____ Date: _____

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 be analyzed for and quantitated using this method as part of a client's special request.

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; 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 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.

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 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 Katahdin Analytical safety procedures and 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.

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

4.1.1 GC Hewlett Packard 5890 series I or II connected to the Turbochrom 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, 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.

TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

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 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 ± 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 ± 15%, a new calibration curve must be prepared for that analyte.

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.

7.3.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.07 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 ul 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 ± 15%. 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 reinjected 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 <u>after</u> a group of samples exhibits a response for an analyte that is <u>above</u> the acceptance limit, i.e. >15%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed, as the CV standard has demonstrated that the analyte would have been detected were it present. In contrast, if an analyte above the QC limits <u>was</u> detected in a sample extract, then re-injection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 15% below the initial calibration response, then re-injection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present.

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

7.4.3 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.

7.4.4 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.

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)

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.

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.

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

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 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 Herdicides 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-Dichlrophenylacetic acid (DCAA)	0.5	0.5

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 nominal acceptance limits (see Table 4). When enough data points are collected, laboratory derived limits will be established.

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.

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.

8.4 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 Organics 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. 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.

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Table 2 Summary of Method Modifications
Table 3 Laboratory PQLs
Table 4 QC Acceptance criteria
Figure 1 Instrument Run Log
Figure 2 Review Checklist

TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: 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	Nominal limits. Refer to table 4.	(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	(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	(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 .
Demonstra-tion of analyst proficiency – 4 replicates	Once per analyst initially and annually thereafter	P&A meet method criteria	Repeat P&A study
MDL study	Once per year	Ideally, PQL = at least 3xMDL	Repeat MDL

TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-305-06	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 to narrow. The windows are: ± 0.07 for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms.	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**

TABLE 4

QC ACCEPTANCE CRITERIA METHOD 8151

Analyte	Accuracy Water * (%R)	Precision Water (RPD)	Accuracy Soil * (%R)	Precision Soil (RPD)
2,4-D	30-120	<30	30-120	<50
2,4,5-TP	30-120	<30	30-120	<50
2,4-DB	30-120	<30	30-120	<50
2,4,5-T	30-120	<30	30-120	<50
Dalapon	30-120	<30	30-120	<50
Dicamba	30-120	<30	30-120	<50
Dichloroprop	30-120	<30	30-120	<50
Dinoseb	30-120	<30	30-120	<50
MCPA	30-120	<30	30-120	<50
MCPP	30-120	<30	30-120	<50

* Nominal limits

TITLE: **ANALYSIS OF CHLORINATED HERBICIDES BY GC USING METHYLATION DERIVATIZATION: SW-846 METHOD 8151**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

Katahdin Analytical Services, Inc.
Instrument: GC07
Amount Injected 2ul
Reviewed by/ Date: JLP 7/1/07

GC Laboratory Instrument Runlog

Method: SW846 8082 / SW846 8081 (circle) 8151

OLM04.3 / OLM03.2 / SOM01.1

Date	Init.	Result File	Sample ID	Y/N	Method	Column	Comments
5-21-07	JLP	7AE031	Hexane	N	HERB01	301/302	
5-22-07	JLP	32	↓				
		41	Test				
		42					
		43					
		44					
		45	↓	↓			
↓	↓	↓ 46	2,4,5-T	Y	↓	↓	
5-22-07	JLP	7AE047	ICAL 0.5ug/mL	Y	HERB01		P3957
		48	0.1				P3958
		49	0.25				P3959
		50	0.75				P3960
		51	1.0				P3961
		52	↓ 2.0 ↓				P3962
		53	IND 0.5ug/mL	↓			P3994
		54	WG39111-1U 3510	Y			
		55	-2				
		56	-3				
		57	↓ -4				
		58	WG39139-1				
		59	↓ -2				
		60	SA2406-7RE				
		61	↓ -13 ↓	↓			
		62	Hexane	N			
		63	CV 0.5ug/mL	Y			P3957
		64	WG38975-1 3510	Y			
		65	↓ -2				
		66	↓ -3				
		67	SA2250-1 ↓	↓			
↓	↓	↓ 68	Hexane	N	↓	↓	

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 : ☐ 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 SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS

Prepared By: Mike Thomas Date: 7/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: [illegible] Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10·23·00

General Manager: Duncan F. Hughes Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10·23·00	10/23/00
02	removed references to medium level extraction. New logbook figures minor changes through out	LAD	020305 ~~DT~~ LAD 020305	020305
03	updated compound list changes in wording to clarify updated logbook	LAD	04/06	04/06
04	Added definitions, added waste information, added LCSD, updated solvent exchange, updated Table 1, replaced fig. 2, added PCB cleanup to Sect. 2	LAD	09/07	09/07

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

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

I acknowledge receipt of copy ___ of document **SOP CA-500-04**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-500-04**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for pesticides/PCBs analysis.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the extraction of sediment/soil samples for pesticides/PCBs analysis. Each analyst must demonstrate the ability to generate acceptable results with this method.

It is the responsibility of all Katahdin personnel involved in the preparation of solid samples for pesticides/PCBs analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should

also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for the data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials, and appropriate segregation of hazardous wastes. Everyone involved with the procedure must be familiar with the material safety data sheets for all the materials used in this procedure. Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical 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 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. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the "N-Lo" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

Pesticides/PCBs are extracted from solid samples by sonication with a methylene chloride/acetone solution (1:1 by volume) following EPA Method 3550, current revision. The resulting extract is dried, concentrated, and solvent exchanged to hexane for analysis by GC. Sulfuric acid cleanup is performed on extracts for 8082 PCB analysis.

This SOP applies to low level extraction of pesticide/PCB pollutants from solid sample matrices.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Whenever possible, plastic items in this lab, must be replaced with metal, teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with methylene chloride.

4.1 Beakers - 400 mL

4.2 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL
Evaporative flask - 500 mL
Snyder column - 3-ball macro

4.3 Powder funnels, 100 mm diameter, 35 mm stem

4.4 Vacuum filtration flask - 500 mL Erlenmeyer

4.5 Buchner funnel, porcelain, Coors® with 85 mm plate diameter (or equivalent)

4.6 Sonic disruptor – Misonix XL2015 (or equivalent), equipped with dual titanium horn extenders for extracting two samples at a time.

4.7 Spatula - stainless steel

4.8 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.9 Boiling chips - 12 mesh, silicon carbide (or equivalent)

4.10 Water bath - eight position concentric ring bath or equivalent, equipped with a calibrated thermometer

4.11 Filter paper - 7.0 cm, Whatman, #4, or equivalent

4.12 Syringe - gas tight, 1.0 mL, solvent rinsed between each use

4.13 Balance – top-loading, capable of weighing to 0.1 g

4.14 Nitrogen evaporation apparatus

5.0 REAGENTS

5.1 Sodium sulfate - (ACS reagent grade) powdered, anhydrous, certified by the manufacturer/vendor as purified by heating to 400 °C prior to receipt by the laboratory. Solvent rinse immediately before use by rinsing three times with pesticide grade methylene chloride.

5.2 Sodium sulfate - (ACS reagent grade) granular, anhydrous, purified as described in section 5.1.

5.3 Methylene chloride - (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300mLs to 1.0 mL followed by GC/MS analysis.

5.4 Acetone and hexane - pesticide grade or equivalent

5.5 Organic-free sand, purified by baking at 400 °C for four hours. Method blanks serve as checks on the baked sand.

5.6 Surrogate spiking solution - Prepare a solution of decachlorbiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL each in acetone. Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.7 Pesticide Matrix spike/Lab control sample spiking solution - Prepare a spiking solution in pesticide grade methanol that contains all target analytes listed below:

Analyte	ug/mL
4,4'-DDD	0.5
4,4'-DDE	0.5
4,4'-DDT	0.5
Aldrin	0.5
alpha-BHC	0.5
beta-BHC	0.5
delta-BHC	0.5
Dieldrin	0.5
Endosulfan I	0.5
Endosulfan II	0.5
Endosulfan Sulfate	0.5
Endrin	0.5
Endrin Aldehyde	0.5
Endrin Ketone	0.5
gamma-BHC (Lindane)	0.5
Heptachlor	0.5
Heptachlor Epoxide	0.5
Methoxychlor	0.5
alpha-Chlordane	0.5
gamma-Chlordane	0.5

Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.8 PCB Matrix Spike/Lab Control Sample Spiking Solution – Prepare spiking solution in pesticide grade acetone that contains PCB's Arochlor 1016 and Arochlor 1260 (1660), both at 5.0 ug/mL.

Store the solution at –10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Soil samples are collected in glass soil jars and stored at 4°C (±2°C) until time of extraction.

Holding time for extraction of sediment/soil samples for Method 3550 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

LOW LEVEL EXTRACTION OF SOIL/SEDIMENT FOR PESTICIDES/PCBs

The low level extraction procedure is designed for the preparation of soil/sediment samples that may contain analytes at levels lower than 20,000 ug/kg. The procedure involves extraction of pesticides and PCBs from an initial sample weight of 30.0 ± 0.1 g using an ultrasonic cell disruptor.

Many solid samples may need to be cleaned up to reduce matrix interferences. The cleanup procedure employed will be dependent upon the nature of the interferences and the target compounds to be analyzed, and options may include acid wash, sulfur cleanup, florisil cleanup, or gel permeation chromatography (GPC). The Department Manager should be consulted to determine if a particular sample should be subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs. All extracts or extract splits for subsequent 8082 PCB analysis will, at a minimum, undergo acid cleanup. (Refer to SOP CA-525, current revision)

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

7.1 Discard any excess water on the sediment sample. Mix with a stainless steel spatula to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, or rocks, and note actions taken in the appropriate extraction logbook.

7.2 Weigh out a 30.0 ± 0.1 g portion of sample into a labeled 400 mL beaker. Record sample weight to nearest 0.1 g in appropriate extraction logbook. Add between 30 g and 60 g of powdered sodium sulfate, as required, to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.3 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one 30.0 ± 0.1 g portion of purified sand in a labeled 400 mL beaker. Add 60 g sodium sulfate and mix well. (Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)

7.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30.0 ± 0.1 g portion of purified sand in a labeled 400 mL beaker. Add 30 g sodium sulfate and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB LCS's must be prepared (refer to sections 5.7 and 5.8). If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30.0 ± 0.1 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.1 g in appropriate extraction logbook. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB MS/MSD pairs must be prepared (refer to sections 5.7 and 5.8).

7.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL pesticide/PCBs surrogate spiking solution using a 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to each use.

7.7 To LCS/LCSD and MS/MSD pairs add 1.0 mL of pesticide or PCB matrix spike/LCS spiking solution using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to spiking a different solution and when spiking is completed.

7.8 To assure optimum operation and maximum energy output, the sonicators must be tuned daily prior to extracting samples. The following tuning procedure must be performed with the sonicator probes vibrating in air.

7.8.1 Turn OUTPUT CONTROL knob counter-clockwise to zero, and turn Pulsar Duty Cycle to off (or continuous mode).

7.8.2 Press POWER SWITCH to ON (up) position. Engage the Timer Switch (W-375)

7.8.3 Press and hold down the TUNE switch.

7.8.4 Turn the Output Control Knob towards setting 10. Note the position of the needle on the % output power meter. **Do NOT exceed 70%. If you reach 70% - STOP!!** Rotate the Tuning Knob clockwise or counter-clockwise until a minimum (not maximum) reading (usually less than 20%) is obtained.

7.8.5 Turn the Output Control Knob towards setting 10. Again, note the position of the needle and do not exceed 70%. Rotate the Tuning Knob until you obtain a minimum reading of 20% or below.

7.8.6 Release the TUNE switch. **CAUTION: Do NOT touch probe. The probe is still active.**

7.8.7 Turn OUTPUT CONTROL KNOB counter-clockwise to zero (or disengage timer).

7.8.8 Confirm that the sonicators were tuned by recording the date and/or percent in the extractions logbook.

Note: If the unit will not be used immediately, please turn the power switch to off.

7.9 Prior to extracting any samples, ensure that the sonicator probes are decontaminated by rinsing with a methylene chloride wash bottle. Collect the waste in a waste beaker. It may sometimes be necessary to wipe the upper part of each probe with a methylene chloride dampened KimWipe. Repeat this decontamination step between each sample on each probe.

7.10 To the mixed and spiked blank and LCS, add 100 mL of the 1:1 methylene chloride/acetone solution and proceed with steps 7.11 through 7.14.

7.11 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula to loosen up the mixture prior to extracting. Position beaker in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4 inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.

7.12 Sonicate for 3 minutes with the output control knob set at 10, and mode switch on "pulsed" and % duty cycle knob set at 50%. While the mixture is sonicating, one should be able to see all, or most of the material, moving in the beaker under the influence of the energized probes. If not, stir the mixture again.

7.13 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter; prerinse flask, funnel and filter with methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask through Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered.

7.14 Repeat the extraction two additional times using 100 mL portions of 1:1 methylene chloride:acetone. Before each extraction, make certain that the sodium sulfate is free-flowing and not a consolidated mass. As required, break up large lumps with the clean spatula. Decant the extraction solvent into the Buchner funnel after each sonication. On the final sonication, pour the entire sample contents into the Buchner funnel and rinse thoroughly with 1:1 methylene chloride:acetone to complete the quantitative transfer of the extract.

CONCENTRATION OF LOW LEVEL EXTRACTS

7.15 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels.

7.16 For a solvent exchange, add approximately 50 mL Hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only.

7.17 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to

pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow to drain.

7.18 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.19 If samples are not to be GPC'd follow Steps 7.19 through 7.23 to concentrate extracts to final volume of 10.0 mLs. Otherwise proceed to GPC cleanup procedure as described in the current revision of Katahdin SOP CA-513.

7.20 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.21 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.22 Reduce the extract in the concentrator tube to approximately 10 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.

7.23 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane extract to 10 mL in either a 12 or 16 mL vial using the appropriate "reference vial" for volume comparison.

7.24 Transfer the sample label from the concentrator tubes to the vials. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and "tray location" of the individual extract vials.

7.25 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. All sample extracts for 8081 pesticide analysis do not undergo further cleanup unless requested by the client. All sample extracts for combined 8081/8082 analyses must be split. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. Prior to splitting, contents of vial must be shaken well. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for

the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 A method blank must be extracted for each and every item listed below:

- Each day of extraction (24 hours midnight - midnight)
- Each extraction method
- Every 20 samples extracted in a 24-hour period

8.2 A laboratory control sample (LCS) is required for each and every item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticides and/or PCBs)

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Method 3550B, USEPA SW-846, Third Edition, Final Update III, December 1996.

Table 1 Summary of Method Modifications
Figure 1 Example of Pest./PCB Soil Sample Prep Logbook Page

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-500-04	METHOD 3550, current revision
Apparatus/Materials	1. short stem funnels	1. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. extract dried using Na_2SO_4 in short stem funnels 2. place sonicator horns ½ way between the surface of the solvent and the sediment layer 3. no apparatus height specification for concentration on water bath 4. water bath at 75-85 deg C 5. sample removed from water bath when volume reaches ~6 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane at the start of concentration process	1. extract dried using Na_2SO_4 in drying columns 2. place sonicator horns ½ inch below the solvent surface but above sediment layer 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. water bath at 80-90 deg C 5. sample removed from water bath when volume reaches 1-2 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes	Refer to analytical SOP	
QC - LCS		
QC - Accuracy/Precision		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

Pest Son

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: SW846 3550 (sonication) X SW846 3540 (soxhlet)____ SW846 3545 (ase)____

Analytical Method: SW846 8081 X SW846 8082____ CLP OLM03.1____ CLP OLM04.2____

Date QC Started: 9-21-07 QC Expiration Date: 10-5-07 QC Batch ID____

Surrogate ID: GCO 434 Spike ID: GCO 418 Spike ID:____

Solvent Lot # $MeCl_2$: E30E66, Acetone: C20E10 Solvent Lot # Hexane: E30E44 Sonicator Horns Tuned: S.Z.B.

Nitrogen Bath Temp ~34°C

Date Extracted	Ext. Init.	Sample ID	Initial Vol.	Surr. Vol.	Spike Vol.	Fraction: Pest	Fraction: PCB	Final Vol.	Date Conc.	Tray Location	Initials	Clean-Up: GPC	Clean-Up: Flor.	Clean-Up: Acid Wash	Clean-Up: Other	Comments
9-21-07	Km	WL693633-1	30.03	1ml	NR	✓		10ml	9-25-07	PA736 A5	GN					R 70262
		-2	30.01		1ml					A6						
		-3	30.02							A7						MS SA5208-7
		-4	30.00							A8						MSD
		-5	30.01							A9						MS SA5210-1
		-6	30.03							A10						MSD
									GN 9-25-07							

Date Extracted	Ext. Init.	Sample ID	Initial Vol.	Surr. Vol.	Spike Vol.	Fraction: Pest	Fraction: PCB	Final Vol.	Date Conc.	Tray Location	Initials	Clean-Up: GPC	Clean-Up: Flor.	Clean-Up: Acid Wash	Clean-Up: Other	Comments
9-21-07	Km	SA5207-5	30.01	1ml	NR	✓		10ml	9-25-07	A11	GN					
		SA5208-2	30.00							A12						
		-3	29.97							B1						
		-4	30.02							B2						
		-5	30.00							B3						
		-6	29.99							B4						
		-7	29.99							B5						MS/MSD
		-8	30.04							B6						
		-9	30.00							B7						
		-10	29.98							B8						
		-11	29.98							B9						
		-12	30.03							B10						
		-13	30.02							B11						
		-14	30.00							B12						
		-15	29.98							C1						
		SA5210-1	30.04							C2						MS/MSD
		SA5268-1	30.02							C3						
		-2	30.01							C4						
		-3	30.01							C5						
		-4	30.02							C6						

QAEX133

QAEX133

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-502
Revision History
Cover Page
Page 1

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Prepared By: Micheal Thomas Date: 07-24-00

Approved By:

Department Manager: [signature] Date: 6-23-06

Operations Manager: Deborah J. Radeau Date: 6·23·06

QA Officer: Susie Dimond Date: 6-23-06

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03	Changes to sect. 5.5 & Figures 3 & 4 to reflect current spike solutions and concentrations Replaced cover page. Original cover page filed with SOP CA502-02	LAD	04/06	04/06
04	Added definitions, added waste information added LCSD, added SIM LCS/D, MS/D, updated Table 1, added use of narrow range pH paper. Minor changes throughout to reflect current practice.	LAD	09/07	09/07

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-502-04**, 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-502-04**, 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 the extraction portion of EPA Method 625, as well as EPA Methods 3510 (modified separatory funnel extraction) and 3520 (continuous liquid-liquid extraction), current revisions.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to

Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their department follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDS's for all the materials used in this procedure.
Each qualified analyst or technician must be familiar with Katahdin Analytical 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.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards

are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware, disassembly of CLLEs after extraction, etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction aqueous samples are considered either N-Hi or N-Low waste and should be disposed of in the corresponding satellite waste accumulation area nearest the point of generation. Sodium sulfate used for sample drying should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

For aqueous samples extracted by CLLE, a one liter aliquot of sample is adjusted to pH $\leq$ 2 and extracted with methylene chloride using a continuous liquid-liquid extractor. The pH is then adjusted to pH $\geq$ 11 and the sample is extracted again with methylene chloride. A modified separatory funnel extraction may also be used. If this procedure is used, the sample aliquot is first adjusted to pH $\geq$ 11 and then to pH $\leq$ 2. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL. Specifically, for Method 625, separate base/neutral and acid fractions are produced, with the base/neutral fraction extracted first and the acid fraction second; each is dried and concentrated to 1.0 mL.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC/MS analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in the total ion current profiles (TICPs). Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or teflon or other non-phthalate plastic substitute.

TITLE: **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

Special care should be taken to ensure that clean glassware and apparatus are used and pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Brand names and catalog numbers are included for illustration purposes only.

4.1 Continuous liquid-liquid extractors - including body, 500 mL round bottom flask and Alhin condensers and equipped with Teflon or glass connecting joints requiring no lubrication (Hershberg-Wolf Extractor, Ace Glass Company, Vineland, NJ, P/N 6841-10 or equivalent).

4.2 Glass powder funnels.

4.3 Fluted filter paper, 18.5cm diameter.

4.4 Concentrator tube - Kuderna-Danish, 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test.

4.5 Evaporation flask - Kuderna-Danish, 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with neck clips.

4.6 Snyder column - Kuderna-Danish, three- or four-ball macro (Kontes K-503000-0121 or equivalent).

4.7 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.

4.8 Vials - Glass, 1.8 mL capacity, with polytetrafluoroethylene (PTFE)-lined screw top and 12 mL with Teflon-lined caps.

4.9 2 L separatory funnel, equipped with Teflon stopper and stopcock; Nalgene Teflon FEP separatory funnels may also be used.

4.10 Organic Free Boiling Chips - approximately 10/40 mesh, Teflon or silicon carbide (or equivalent). Rinsed with methylene chloride.

4.11 Water bath - heated, with concentric ring cover, capable of temperature control (± 20°C). The bath should be used in a hood.

4.12 Nitrogen evaporation apparatus.

4.13 Wide range pH test strips, pH 0-14, Whatman CF Type.

4.14 Glass rods for stirring samples.

4.15 Amber bottles or other appropriate containers for collection of extracts from separatory funnel extraction.

4.16 5 ¾" Pasteur pipets.

4.17 Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.

4.18 Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.

5.0 REAGENTS

All reagent and solvent lots must be checked for possible contamination. Refer to the current version of Katahdin SOP CA-105, Reagent and Solvent Handling, for further details. The extraction staff is responsible for submitting samples to the GC or GC/MS sections for appropriate analysis. All information concerning preparation of the reagent/solvent lot sample will be recorded in the Organic Extraction Log (Figure 1) and acceptance or rejection of these lots must be recorded in the solvent/reagent lot check logbook (Figure 2). All reagents and solvents must be free (<PQL) of any target compounds.

5.1 Laboratory Reagent Grade Water - defined as water in which an interferent is not observed at or above the PQL of each parameter of interest. Deionized water filtered through activated charcoal.

5.2 Sodium sulfate - granular. Bake at 400°C for 4 hours (may be done by vendor). Purify by rinsing three times with pesticide grade methylene chloride. Allow residual methylene chloride to evaporate before each use. Cool in a desiccator and store in a glass bottle with a Teflon-lined cap.

5.3 Sulfuric acid solution (1:1 H_2SO_4 : H_2O) - slowly add 500 mL of H_2SO_4 (sp gr 1.84) to 500 mL reagent water.

5.4 Acetone, methanol, methylene chloride - pesticide residue analysis grade or equivalent.

5.5 Standard Preparation - For all standard preparations, see current revision of the following Katahdin Analytical SOPs:

- "Standards Preparation, Documentation and Traceability", (CA-106, current revision)
- "Balance Calibration," (CA-102, current revision)

5.5.1. Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound	Conc.
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5.2 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5.3 SVOA Matrix Spike/Lab Control Samples Spiking Solution - the matrix spike/LCS solution consists of the compounds listed in Figure 3.

Prepare a spiking solution that contains each of the base/neutral compounds listed in Figure 3 at 50 ug/mL in methanol and the acid compounds at 100 ug/mL in methanol. Matrix spike/LCS standards are stored in the freezer (-10°C to -20°C) located in the storage area.

5.5.4 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 2 ug/mL for base/neutral. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL of methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.5.5 Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution – Prepare a spiking solution in methanol that contains the compounds listed in Figure 4 at concentrations of 100 ug/ml. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Continuous liquid-liquid (Method 3520) and/or separatory funnel (Method 3510) extractions for semivolatiles must be started within seven days of date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific. If sampling date is unknown, the hold time is counted from one day prior to date received.

7.0 PROCEDURES

The internal chain-of-custody must be signed when removing and replacing samples in storage locations. The sample preparation/extraction log must be filled out with the necessary information as extraction of each sample is completed.

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), matrix (Aq), 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. An MS/MSD is required if requested by the client or per 20 samples or every 14 days, whichever occurs first. (Refer to the logbook page, "date QC expires"). If extra MS/MSD aliquots of sample are unavailable a laboratory control sample duplicate (LCSD) may be substituted.

7.1.6 Check the pH of each sample with wide range pH paper by removing a couple of sample drops with a clean disposable pipet or on the tip of a stirring rod. Adjust the pH of the samples (including method blank, LCS/LCSD, and MS/MSD) to $\leq$ pH 2 with 1:1 H_2SO_4 after addition of surrogates and spikes and prior to attaching Allihn condensers (Step 7.1.11). Stir with a glass stirring rod and check pH by tapping the glassrod onto wide range pH paper. The pH must be ≤ 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. Measure the initial volume of the samples by filling the sample bottles with water up to the level marked earlier. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.1.9 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL base/neutral and acid (SVOA) surrogate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record surrogate spike volume and identification

code in extraction logbook. Thoroughly rinse syringe with acetone before and after each use.

7.1.9.1 If the request is for SVOA, use the SVOA Surrogate Solution (sect. 5.5.1).

7.1.9.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5.2).

7.1.9.3 If the request is for SIM-SVOA, use the SIM surrogate solution as well as SVOA surrogate solution. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.1.10 To LCS/LCSD and MS/MSD add 1.0 mL base/neutral and acid (SVOA) matrix spike/LCS spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with methanol before and after each use.

7.1.10.1 If the request is for SVOA -
add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3).

7.1.10.2 If the request is for SIM -
add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3) and
add 1.0 mL of Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution (sect 5.5.4).

7.1.10.3 If the request is for SVOA Appendix IX, use the SVOA Appendix IX Spiking solution as well as the SVOA spiking solution -
add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3) and
add 1.0 mL of Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution (sect 5.5.5).

7.1.11 Attach cooling water Allihn condensers, after first rinsing each 45/50 joint with methylene chloride. Turn on the heating mantles and allow the samples to extract for 22 $\pm$ 2 hours. Turn off the mantles and let samples cool.

7.1.12 Detach condensers and verify that the pH is still $\leq$ 2 in the same manner mentioned in 7.1.6. If the pH has changed, more acid should be added to make the pH $\leq$ 2 and the sample extracted for several more hours.

7.1.13 Upon completion of acid extraction, allow the sample to cool. Detach condensers and add enough 10N NaOH to adjust the pH to $\geq$ 11 with stirring. Use glass stirring rods to stir and check the pH of each sample in the same manner mentioned in 7.1.6.

7.1.14 Re-attach Allihn condensers, turn on heating mantles, and allow samples to extract for 22 $\pm$2 hours. Turn off mantles and allow samples to cool.

Proceed to Step 7.4 for sample extract concentration procedures.

7.2 SEPARATORY FUNNEL EXTRACTION (Modified Method 3510)

If an emulsion prevents acceptable recovery or client history indicates samples may demonstrate matrix interference, then samples should be extracted by continuous liquid-liquid extraction (CLLE).

7.2.1 Rinse <u>all</u> 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 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 separatory funnel.

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. An MS/MSD is required if requested by the client or per 20 samples or every 14 days, whichever occurs first. (Refer to the logbook page, "date QC expires"). If extra MS/MSD aliquots of sample are unavailable, a laboratory control sample duplicate (LCSD) may be substituted.

7.2.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL base/neutral and acid (SVOA) surrogate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with acetone before and after each use.

7.2.6.1 If the request is for SVOA, use the SVOA Surrogate Solution.

7.2.6.2 If the request is for SIM, use the SIM Surrogate Solution.

7.2.6.3 If the request is for SIM-SVOA, use the SIM surrogate solution as well as SVOA surrogate solution. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.2.7 To LCS/LCSD and MS/MSD add 1.0 mL base/neutral and acid (SVOA) matrix spike/LCS spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in the extraction logbook. Thoroughly rinse syringe with methanol before and after each use.

7.2.7.1 If the request is for SVOA, use the SVOA Spiking Solution.

7.2.7.2 If the request is for SIM, use the SIM Spiking solution.

7.2.7.3 If the request is for SVOA Appendix IX, use the SVOA Appendix IX Spiking solution as well as the SVOA spiking solution

7.2.8 For each sample, rinse the original sample container with 60 mL of methylene chloride. Add this rinse to the separatory funnel.

7.2.8 Determine the initial volume of each sample. Measure the initial volume of the sample by filling the sample bottle with water up to the level marked earlier. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.2.10 Adjust the pH of the samples (including method blank, LCS/LCSD, and MS/MSD) to pH $\geq$ 11 with 10N NaOH after addition of surrogates and spikes. Stir with a glass stirring rod and check pH by tapping the glass stirring rod onto wide range pH paper. The pH must be $\geq$ 11. If the pH test strip does not clearly indicate the pH is greater than 11, narrow range pH paper must be used.

7.2.11 Add 60 mL of methylene chloride directly to the method blank and LCS/LCSD separatory funnels.

7.2.12 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.

7.2.13 If an emulsion forms, mechanical techniques must be employed to achieve maximum separation. Such means include swirling, centrifugation, and draining through a small separatory funnel. In certain instances, transferring the entire sample into a continuous liquid-liquid extractor (CLLE) may be the only alternative. If any such techniques are used, they must be noted in the extractions logbook, and the batch transferred to a CLLE batch with its own batch ID.

7.2.14 Add a second 60 mL aliquot of methylene chloride to the separatory funnel and extract for the second time (see 7.2.12 – 7.2.13). Collect the methylene chloride layer in the same amber collection bottle.

7.2.15 Repeat the extraction for a third time as described in 7.2.14.

7.2.16 Following the third shake, using a glass stirring rod, check the pH to ensure that it has remained at $\geq$ 11. If the pH has changed back to neutral range, it must be readjusted to $\geq$ 11 and the sample must be extracted at least one more time, adding the methylene chloride to the same amber bottle, that was previously used. If the pH has remained at a value $\geq$ 11, the pH is then adjusted to $\leq$ 2 with 1:1 H_2SO_4. Add enough 1:1 H_2SO_4 to adjust the pH to $\leq$ 2 with stirring. Use glass stirring rods to stir.

7.2.17 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.

Proceed to Section 7.4 for extract concentration procedures.

7.3 625 BN/A EXTRACTION (Either Method 3510 or 3520 may be used.)

This extraction procedure is used, where extraction will be followed by 625 GC/MS analysis rather than 8270 GC/MS. Either a separatory funnel extraction technique or a continuous liquid-liquid extraction technique (described above in 7.1 and 7.2) can be used for this procedure. There are two main differences in this procedure from those described in sections 7.1 and 7.2: (1) the pH adjustments articulated in section 7.1

will be reversed when 3520 CLLE is used; and (2) the collection of base and acid fractions are collected in separate containers and concentrated separately.

7.3.1 For extraction using a separatory funnel technique (Method 3510), follow the general procedure as described in 7.2. **NOTE:** The pH adjustments are made in the same manner as described in section 7.2 with the first adjustment to $\geq$ 11, followed by extraction of the samples three times in methylene chloride. The methylene chloride layer is collected in an amber jar. Label this as the base/neutral fraction. After the third shake, the pH is adjusted to $\leq$ 2, followed by extraction of the samples three times in methylene chloride. The methylene chloride layer is collected in a separate amber jar. Label this the acid fraction.

7.3.2 For extraction using a continuous liquid-liquid technique (Method 3520), follow the general procedure as described in 7.1. **However; the pH adjustments are reversed from those described in section 7.1.** Following the first 22 $\pm$ 2 hours extraction at pH $\geq$ 11, the round bottom flask with the base/neutral fraction is removed, covered, and stored. A new round bottom flask with 200 mL methylene chloride is attached to the CLLE body and the pH is adjusted to $\leq$ 2. Extraction continues at this pH for an additional 22 $\pm$ 2 hours.

7.4 CONCENTRATING THE EXTRACTS

For Methods 3510 and 3520, the combined fractions are concentrated to a final volume of 1.0 mL. For Method 625, the base/neutral fraction and acid fraction extracts are routinely concentrated separately to 1.0 mL. Figure 4 shows an EPA Region I memo which discusses GC/MS analysis of the separate base/neutral and acid extracts versus GC/MS analysis of combined extracts from Method 625.

7.4.1 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.

7.4.2 Transfer the methylene chloride extract to a K-D concentrator setup through a short stem funnel filled with 1-2 inches of sodium sulfate in fluted filter paper. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mls of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mls of methylene chloride and allow to drain

7.4.3 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.

7.4.4 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.

7.4.5 Reduce the methylene chloride extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.

7.4.6 Reduce each extract to slightly less than 1 mL and then, using a 5 ¾" pasteur pipet, transfer the final extract and label to a 1.8 mL vial with PTFE-lined cap.

7.4.7 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.

7.4.8 Store in refrigerator until GC/MS analysis.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)

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- 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.

Code of Federal Regulations (40 CFR), Part 136, Appendix A, Rev. July 1, 1995, Method 625.

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TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-502-03	METHOD 3510, current revision
Apparatus/Materials	1) 250 mL amber bottle or flask 2) 1.0 mL syringe 3) short stem funnels	1) 250 mL Erlenmeyer flask 2) 5.0 mL syringe 3) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) extract collection in amber bottle or Erlenmeyer flask 2) Add surrogate/spike to sample in CLLE 3) Extract for 3 minutes on mechanical shaker 4) extract three times at pH $\geq$ 11, then extract three times at pH $\leq$ 2. 5) extract dried using Na_2SO_4 in short stem funnels 6) Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 7) water bath temp 75-85 deg C 8) no apparatus height specification for concentration on water bath 9) sample removed from water bath when volume reaches ~6 mL 10) N bath temp no higher than 39 deg C	1) extract collection in Erlenmeyer flask 2) Add surrogate/spike directly to sample bottle 3) Extract by shaking vigorously for 1 - 2 minutes with periodic venting 4) extract three times at pH $\leq$ 2, then extract three times at pH $\geq$ 11. 5) extract dried using Na_2SO_4 in drying columns 6) Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer 7) water bath temp 15-20 deg C above solvent boiling temp 8) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min 9) sample removed from water bath when volume reaches 1 mL 10) N bath temp 35 deg C
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL

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TABLE 1, continued

SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)

TOPIC	KATAHDIN SOP CA-502-03	METHOD 3520, current revision
Apparatus/Materials	1) short stem funnels	1) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) Add surrogate/spike to sample in CLLE 2) Add approximately 500 - 600 mL of methylene chloride to the CLLE body 3) CLLE for 22 ± 2 hours 4) Extract dried using Na_2SO_4 in short stem funnels 5) Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 6) water bath temp 75-85 deg C 7) no apparatus height specification for concentration on water bath 8) sample removed from water bath when volume reaches ~6 mL 9) N bath temp no higher than 39 deg C	1) Add surrogate/spike directly to sample bottle 2) Add 300 - 500 mL of methylene chloride to the distilling flask of the extractor 3) CLLE for 18 - 24 hours 4) Extract dried using Na_2SO_4 in drying columns 5) Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer 6) water bath temp 15-20 deg C above solvent boiling temp 7) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min 8) sample removed from water bath when volume reaches 1 mL 9) N bath temp 35 deg C
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL

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TABLE 1, continued

SUMMARY OF METHOD MODIFICATIONS (METHOD 625)

TOPIC	KATAHDIN SOP CA-502-03	METHOD 625
Apparatus/Materials	See above tables for 3510 and 3520	See above tables for 3510 and 3520
Reagents		
Sample preservation/ handling		
Procedures	1) See above tables for 3510 and 3520 2) For 625 analysis, a new round bottom flask with 200 mL methylene chloride is attached to the CLLE body for second pH extraction	1) See above tables for 3510 and 3520 2) For 625 analysis, attach a clean distilling flask containing 500 mL of methylene chloride to the continuous extractor for second pH extraction
QC - Spikes	See above tables for 3510 and 3520	See above tables for 3510 and 3520
QC - LCS	See above tables for 3510 and 3520	See above tables for 3510 and 3520

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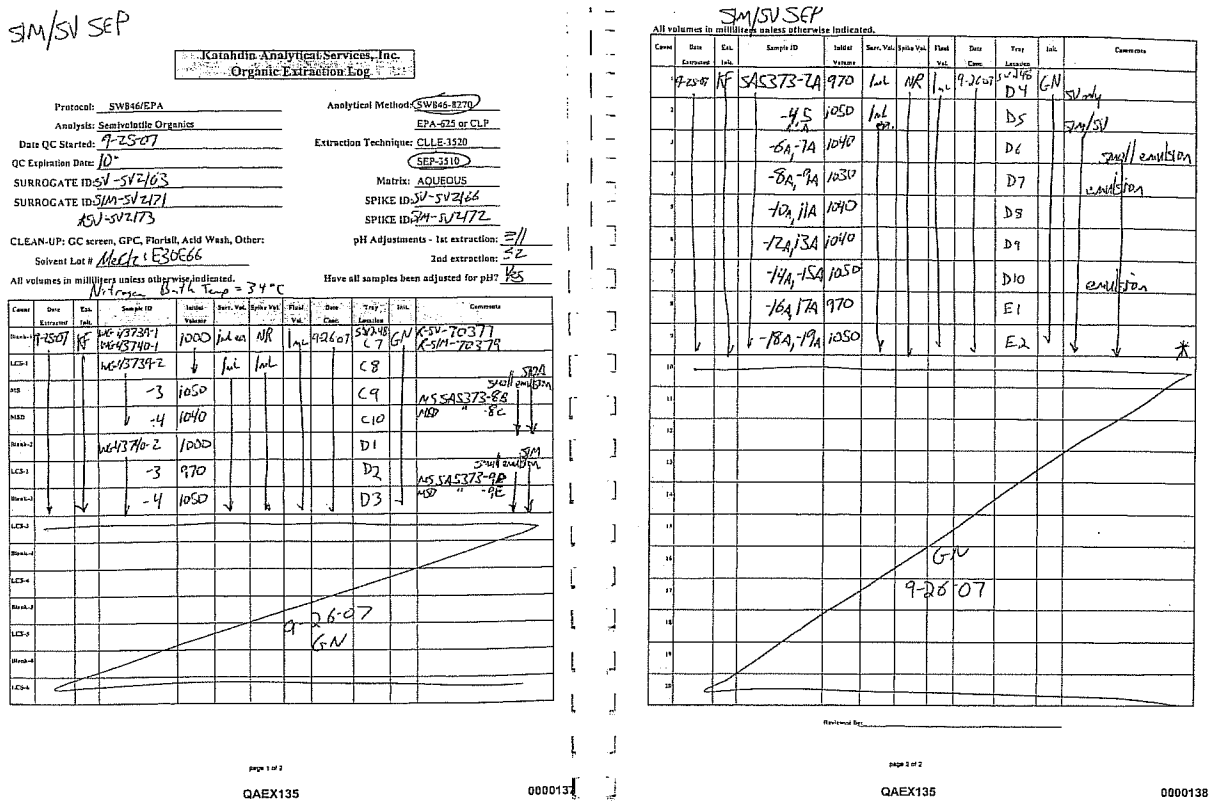
FIGURE 1

EXAMPLE OF SEMIVOLATILES LOGBOOK PAGE



SM/SV SEP

Katahdin Analytical Services, Inc.
Organic Extraction Log

Protocol: SW846/EPA
Analysis: Semivolatile Organics
Date QC Started: 9-25-07
QC Expiration Date: 10-
SURROGATE ID: SV-SV2163
SURROGATE ID: SM-SV2171
SV-SV2173
CLEAN-UP: GC screen, GPC, Florisil, Acid Wash, Other:
Solvent Lot # Mock/1 : E30E66
All volumes in milliliters unless otherwise indicated.
Nitrogen Bath Temp = 34°C

Analytical Method: SW846-8270
EPA-625 or CLP
Extraction Technique: CLLE-3520
SEP-3510
Matrix: AQUEOUS
SPIKE ID: SV-SV2166
SPIKE ID: SM-SV2172
pH Adjustments - 1st extraction: 11
2nd extraction: 2
Have all samples been adjusted for pH? Yes

Cover	Date Extracted	Ext. Init.	Sample ID	Initial Volume	Surr. Vol.	Spike Vol.	Final Vol.	Date Conc.	Tray Location	Init.	Comments
Blank-1	9-25-07	KF	WG-437397-1 WG-437340-1	1000	1mL	NR	1mL	9-26-07	SV2148 C7	GN	K-SV-70377 K-SM-70379
LCS-1			WG-437397-2		1mL	1mL			C8		
MS			-3	1050					C9		MS SA5373-8A SV01 emulsion
MSD			-4	1040					C10		MSD " -8C
Blank-2			WG-437340-2	1000					D1		
LCS-2			-3	970					D2		SM emulsion
Blank-3			-4	1050					D3		MS SA5373-9A MSD " -9C
LCS-3											
Blank-4											
LCS-4											
Blank-5											
LCS-5											
Blank-6											
LCS-6											

9-26-07 GN

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SM/SV SEP

All volumes in milliliters unless otherwise indicated.

Cover	Date Extracted	Ext. Init.	Sample ID	Initial Volume	Surr. Vol.	Spike Vol.	Final Vol.	Date Conc.	Tray Location	Init.	Comments
1	9-25-07	KF	SA5373-7A	970	1mL	NR	1mL	9-26-07	SV2148 D4	GN	SV only
2			-4S 2A	1050	1mL				D5		SM/SV
3			-6A, -7A	1040					D6		SM/ emulsion
4			-8A, -9A	1030					D7		emulsion
5			-10A, 11A	1040					D8		
6			-12A, 13A	1040					D9		
7			-14A, -15A	1050					D10		emulsion
8			-16A, 17A	970					E1		
9			-18A, -19A	1050					E2		*
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											

GN 9-26-07

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FIGURE 2

SOLVENT/REAGENT LOT CHECK LOGBOOK

SOLVENT:

LOT#:

DATE RECEIVED:

DATE CONCENTRATED:

CONCENTRATED BY:

PREP METHOD:

TRAY LOCATION:

ANALYZED BY:

PASS/FAIL:

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FIGURE 3

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Benzyl alcohol
1,1-Biphenyl	Bis (2-chloroethoxy) methane
1,2,4-Trichlorobenzene	Bis (2-chloroethyl) ether
1,2-Dichlorobenzene	Bis (2-Chloroisopropyl) ether)
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	Dimethyl phthalate
3-Nitroaniline	Di-n-butylphthalate
4-Bromophenylphenyl ether	Di-n-octyl phthalate
4-Chloroaniline	Fluoranthene
4-Chlorophenylphenyl ether	Fluorene
4-Nitroaniline	Hexachlorobenzene
Acenaphthene	Hexachlorobutadiene
Acenaphthylene	Hexachlorocyclopentadiene
Acetophenone	Hexachloroethane
Aniline	Indeno (1,2,3-cd) pyrene
Anthracene	Isophorone
Atrazine	Naphthalene
Azobenzene	Nitrobenzene
Benzaldehyde	N-Nitrosodimethylamine
Benzidine	N-Nitroso-di-n-propylamine
Benzo (a) Anthracene	N-Nitrosodiphenylamine
Benzo (a) pyrene	Phenanthrene
Benzo (b) fluoranthene	p-toluidine
Benzo (ghi) perylene	Pyrene
Benzo (k) fluoranthene	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 4

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

TITLE: **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 5

COPY OF EPA REGION I MEMO REGARDING COMBINATION OF EXTRACTS FOR METHOD 625

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION I
Environmental Services Division
60 Westview Street, Lexington, MA 02173-3185

MEMORANDUM

DATE: March 6, 1995

SUBJ: Analysis of Combined Extracts from Method 625

FROM: Arthur E. Clark, Chemist, Quality Assurance Office (EQA)

TO: James O'Dell, ATP Coordinator, QA Research Division, EMSL-CI

I am writing to request that you support the position within the Region I ESD that the extracts from EPA method 625 samples may be combined and analyzed together as long as the analysts can document their ability to do so successfully. You and I have discussed this previously. We have now been asked by a treatment authority to state our position formally.

As you know, method 625 for semivolatile organics requires that separate extractions be performed for the acid and base/neutral compounds. However, the method does not state that the extracts must be analyzed separately. Indeed, it is common practice among commercial laboratories to combine the two extracts for analysis. The RCRA and CERCLA Contract Lab Program protocols combine the extracts for analysis. If a program believes that combined analysis will compromise their objectives, the fractions should be analyzed separately. But, when neither fraction is compromised by the other, combined analysis is acceptable; the lab must prove that it can perform the analysis successfully.

One of our guidance documents[1] states that method 625 requires that the extracts be analyzed separately, citing two sections and two tables in the method. However, although they give instructions for analyzing the two fractions, they do <u>not</u> make any statement requiring separate analysis. The intent of the guidance document is to provide <u>guidance</u> for analyzing semivolatile samples when interferences are present.

While analyzing the acid and base/neutral extracts separately is one way to decrease interferences, we do not think that separate analysis should be imposed as an across-the-board requirement. There are many samples which can be analyzed successfully with the extracts combined.

I have spoken with William Telliard, Chief, Analytical Methods Staff, Engineering and Analysis Division, OST. While he does not support blanket approval for combined extract analysis, he believes that it is acceptable as long as the analysts document their ability to do so successfully.

[1] Guidance on Evaluation, Resolution, and Documentation of Analytical Problems Associated with Compliance Monitoring, EPA 821-B-93-001, p. 35 (June 1993).

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-515
Revision History
Cover Page
Page 1

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

Prepared By: Mike Thomas Date: 8/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: JCBurton Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10·23·00

General Manager: Dennis F. Hulgan 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 through out Added wording to Sections 6 and 8	LAD	013105	013105
03	Added separate QC for Pest. and PCB. updated concentration procedure to reflect current practices. Changes in wording for clarification. Update Logbook page	LAD	04/06	04/06
04	Added waste generated and disposal info. Added missing definitions. Updated SPE extraction procedure. Updated Table 1 and 2. Added Table 3.	LAD	09/07	09/07

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

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

I acknowledge receipt of copy _____ of document **SOP CA-515-04,** titled **PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS**.

Recipient: _____Date:_____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **SOP CA-515-04,** titled **PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS**.

Recipient: _____Date:_____

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, and 3535A, current revisions).

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the extraction of aqueous samples for pesticides/PCBs analysis. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training”.

It is the responsibility of all Katahdin personnel involved in the preparation of aqueous samples for pesticides/PCBs analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the

appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for the data.

It is the responsibility of the Supervisor to oversee that members of their group follow this SOP, that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical 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 supervisor, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. This includes the methylene chloride waste layer generated during CLLE extraction. Special care should be taken to pour this layer off into the appropriate waste stream, leaving the sample waste to be disposed of as follows. Since Pesticide/PCB samples are at a neutral pH, SEP funnel or CLLE sample waste may be dumped into either the "N-Hi" or "N-low" satellite accumulation area. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in

the "N-Lo" 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 and 3535A. The methylene chloride is exchanged with hexane for the final extract. Method detection limit studies must be performed annually for pesticides/PCBs using all extraction methods, if the extraction lab wishes to use either or all techniques. Method 3510 (separatory funnel) is generally preferred for pesticides/PCBs since organochlorine pesticides may dechlorinate if under elevated pH conditions for an extended period of time. (Section 3.2, Method 3510B, Rev. 2, 9/94)

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates which are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves which have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with the solvent to be used for extraction.

4.1 Separatory Funnel - 2000 mL capacity, Nalgene Teflon FEP separatory funnels with Nalgene Tefzel® screw-cap closures (or equivalent)

4.2 Concentrator tube - 10 mL, graduated

4.3 Evaporative flask - Kuderna-Danish, 500 mL capacity attached to concentrator with neck clips

4.4 Snyder column - Kuderna-Danish, three ball macro

4.5 Graduated cylinders - 100 mL, 1000 mL, or 2000 mL

4.6 Short Stem Funnels

4.7 250 mL amber collection bottles with Teflon-lined caps

4.8 12 mL and/or 16 mL glass vials with Teflon-lined caps

4.9 Continuous liquid-liquid extractors (CLLE) including body, 500 mL flat bottom boiling flask and Alhin condensers

4.10 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.11 Nitrogen evaporation apparatus.

4.12 Boiling chips - approximately 10/40 mesh, Teflon or selenized carborundum, 12 mesh (or equivalent).

4.13 Water bath - eight position concentric ring bath or equivalent, equipped with a calibrated thermometer.

4.14 Vials, 60 mL with PTFE – lined screw caps.

4.15 Horizon SPE-DEX 4790 Automated Extractor System.

4.16 Atlantic DVB disks, or equivalent.

4.17 1-L amber bottles

5.0 REAGENTS

5.1 Laboratory reagent grade water - water in which an interferent is not observed at or above the PQL for any parameter of interest (carbon filtered ASTM Type II water or equivalent)

5.2 Sodium Hydroxide (10N) – Purchased from vendor, "Baker-analyzed", or equivalent

5.3 Sodium Sulfate (ACS) - Granular, anhydrous. Bake at 400°C for 4 hours (may be done by vendor). Purify by rinsing three times with pesticide grade methylene chloride. Allow residual methylene chloride to evaporate before use. Stored in a Teflon capped glass bottle.

5.4 Sulfuric Acid Solution (1:1) - Add 500 mL concentrated sulfuric acid (certified ASC grade or better) slowly to 500 mL laboratory reagent grade water. Prepare as needed and store in a ground glass stoppered bottle.

5.5 Methylene Chloride ($MeCL_2$) - Pesticide grade or better. Lot must be verified by concentrating 300-400 mL to 1.0 mL and evaluating by GC/MS.

5.6 Acetone and Hexane - Pesticide grade or better. Lot must be verified by concentrating approximately 20-30 mL to 1.0 mL and evaluating by GC/ECD.

5.7 Pesticide/PCB Surrogate spiking solution - Prepare a solution of decachlorbiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1.0 ug/mL ea in acetone. Store the solution at –10 to -20 °C in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.8 Pesticide Matrix Spike/Lab Control Sample spiking solution - Prepare a matrix spiking solution in pesticide grade methanol that contains all target analytes listed below:

ANALYTE	ug/mL
4,4'-DDT	0.5
4,4'-DDD	0.5
4,4'-DDE	0.5
Aldrin	0.5
Dieldrin	0.5
Endrin	0.5
Endrin Aldehyde	0.5
Endrin Ketone	0.5
Endosulfan I	0.5
Endosulfan II	0.5
Endosulfan Sulfate	0.5
alpha-BHC	0.5

ANALYTE (cont.)	ug/mL
beta-BHC	0.5
delta-BHC	0.5
gamma-BHC (Lindane)	0.5
Heptachlor	0.5
Heptachlor epoxide	0.5
Methoxychlor	0.5
alpha-Chlordane	0.5
gamma-Chlordane	0.5

5.9 PCB Matrix Spike/Lab Control Sample spiking solution - Prepare a matrix spiking solution in pesticide grade acetone that contains 5.0ug/ml ea of Aroclor® 1016/1260 mix (Restek catalog# 32039).

5.10 Store the spiking solutions at –10 to -20 °C in a Teflon sealed container. The solutions must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

6.0 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.

7.0 PROCEDURES

SEPARATORY FUNNEL SAMPLE EXTRACTION

If an emulsion prevents acceptable recovery or client history indicates samples may demonstrate matrix interence, then samples should be extracted by continuous liquid-liquid extraction (CLLE).

7.1 Rinse all glassware three times with methylene chloride prior to use.

7.2 Label a 2 L Teflon separatory funnel and a 250 mL amber collection bottle clearly. Label should include laboratory sample number, matrix, analyte, and extraction date. Be sure that the detachable stopcocks are secured to the separatory funnels before adding samples.

7.3 Mark the sample level on the side of the sample bottle for later measurement of initial volume. Transfer the contents of the sample bottle to a 2 L separatory funnel.

7.4 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.

7.5 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). 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.

7.6 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. Note: Sufficient sample volume should be available without depleting all remaining sample aliquots.

7.7 Check the pH of the samples. If it is not between pH 5 and 9, adjust the pH with 10N sodium hydroxide or 1:1 sulfuric acid solution. Note the addition of NaOH or H_2SO_4 in the extraction logbook.

7.8 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.

7.9 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.

7.10 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).

7.11 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.

7.12 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.

7.13 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.

7.14 Repeat the extraction for a third time as described in 7.13.

7.15 Measure the initial volume of the sample by filling the sample bottle with water up to the level marked earlier. Record the volume and any notable characteristics (e.g. color, sediment present, odor) in the extraction logbook.

7.16 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.

7.18 Add 2-3 boiling stones to the round bottom flask and approximately 300 - 400 mL of methylene chloride to the CLLE body.

7.19 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.

7.20 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.

7.21 Mark the sample levels on the sample bottles. Transfer the samples to the CLLE bodies.

7.22 Check the pH of the samples. If it is not between pH 5 and 9, adjust the pH with 10N sodium hydroxide or 1:1 sulfuric acid solution. Note the addition of NaOH or H_2SO_4 in the extraction logbook.

7.23 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 or every 14 days, whichever occurs first, and each analysis. (Refer to the logbook page, "date QC expires"). Note: Sufficient sample volume should be available without depleting all remaining sample aliquots.

7.24 For each sample, rinse the original sample container with approximately 30 mL of methylene chloride. Add this rinse to the CLLE body.

7.25 Determine the initial volume of the sample as described in 7.15 and record it and any notable characteristics in the extraction logbook.

7.26 Add 1.0 mL of the Pesticide/PCB Surrogate Spike to each sample including the blank, LCS/LCSD and MS/MSD, if performed.

7.27 Add 1.0 mL of Pesticide or PCB Matrix Spike to the appropriate LCS/LCSD and MS/MSD pair, if performed, and stir.

7.28 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 ± 2 hours. Turn off the mantles and let samples cool.

7.29 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

7.30 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.

7.31 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.

7.32 Turn on the Horizon controller (switch in the back).

7.33 Check the lubrication oil on the air pump. Fill as needed. Turn the air pump on.

7.34 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.

7.35 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.

7.36 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.

7.37 Press *select* on the control panel to designate an extractor (1, 2, 3, 4 or "." for all), then press *enter*.

7.38 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.

7.39 Repeat this process 2-3 times before using the Horizon autoextractors.

ANALYSIS OF SAMPLES WITH AUTOEXTRACTOR

7.39 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.

7.40 Place an Atlantic DVB disk (or equivalent) into a Horizon disk holder assembly and assemble per owner's manual. Place the disk holder assemble on top of the disk holder platform. There should be roughly 1 cm separating the speedisk from the extractor downtube

7.41 Mark the volume level of liquid in each sample on the outside using a grease pencil.

7.42 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.

7.43 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.

7.44 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.

7.45 Add 1.0 mL of pesticide or PCB matrix spike to the appropriate LCS/LCSD and MS/MSD samples. Recap and shake well.

7.46 Remove cap and add 5.0 mL of 1:1 H_2SO_4 to each sample including the blank, LCS/LCSD and MS/MSD set immediately prior to extracting the sample.

7.47 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.

7.48 Load the sample bottle on the holder and twist ¾ of a rotation. Stop twisting when air bubbles rise to the top of the sample bottle. Do not twist completely around. The foil may loosen and jam the valve.

7.49 Press *select* on the control panel to designate an extractor (1, 2 or "." for both), then press *enter*.

7.50 Type in 8081.3 for the method and press enter. Once the method is loaded, start the extractors by pressing the *start* buttons on the individual extractors. The red LED will blink when the method is complete. The extract will be collected in the 60 ml vial.

7.51 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

7.53 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.

7.54 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.

7.55 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.

7.55 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).

7.56 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 ≈ 2 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of hexane. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL hexane.

7.57 Reduce the extracts to ≈ 10 mL using Nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (45 °C for hexane). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging needle closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook. Transfer extract to a 12 or 16 mL vial. Using a reference vial for volume comparison, adjust the final extract volume to 10 mL by rinsing sides of tube with hexane and transferring rinsings to vial.

7.59 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.

7.60 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. All sample extracts for 8081 pesticide analysis do not undergo further cleanup unless requested by the client. All sample extracts for combined 8081/8082 analyses must be split. 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.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

A method blank must be extracted for <u>each</u> and <u>every</u> item listed below:

- Each day of extraction (24 hours midnight - midnight)
- Each extraction method
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for <u>each</u> and <u>every</u> item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticide and/or PCB) to be performed

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Methods 3510C and 3520C, USEPA SW-846, Third Edition, Final Update III, December 1996.

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TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-515-04	METHOD 3510, current revision
Apparatus/Materials	1. 12 or 16 mL vials used for final extract 2. 250 mL amber bottle or flask used 3. 1.0 mL syringe 4. short stem funnels	1. 2 mL vials used for final extract 2. 250 mL Erlenmeyer flask 3. 5.0 mL syringe 4. drying column
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to separatory funnel	1. one liter graduated cylinders used to transfer initial sample volume to separatory funnel
Procedures	1. extract collection in amber bottle or Erlenmeyer flask 2. extract dried using Na_2SO_4 in short stem funnels 3. no apparatus height specification for concentration on water bath 4. sample removed from water bath when volume reaches ~10 mL 5. hexane added directly to K-D body at start of concentration process	1. extract collection in Erlenmeyer flask 2. extract dried using Na_2SO_4 in drying columns 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. sample removed from water bath when volume reaches 1-2 mL 5. solvent exchange via large K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TABLE 2

SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)

TOPIC	KATAHDIN SOP CA-515-04	METHOD 3520, current revision
Apparatus/Materials	1. short-stem funnels 2. 12 or 16 mL vials used for final extract	1. drying columns 2. 2 mL vials used for final extract
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to CLLE	1. one liter graduated cylinders used to tranfer initial sample volume to CLLE
Procedures	1. CLLE for 18 $\pm$ 2 hours 2. extract dried using Na_2SO_4 in short stem funnels 3. no apparatus height specification for concentration on water bath 4. sample removed from water bath when volume reaches ~10 mL 5. hexane added directly to K-D body at start of concentration process	1. CLLE for 18-24 hours 2. extract dried using Na_2SO_4 in drying columns 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. sample removed from water bath when volume reaches 1-2 mL 5. solvent exchange via macro K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

TABLE 3

SUMMARY OF METHOD MODIFICATIONS (METHOD 3535, current revision)

TOPIC	KATAHDIN SOP CA-515-04	METHOD 3520, current revision
Apparatus/Materials	1. Horizon SPE-DEX 4790 Automated Extractor System.	1. Empore solid-phase extraction system
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to separatory funnel	1. one liter graduated cylinders used to transfer initial sample volume to separatory funnel
Procedures	1. no methanol addition 2. extraction using Horizon SPE-DEX 4790 Automated Extractor System. 3. extract dried using Na_2SO_4 in short stem funnels 4. no apparatus height specification for concentration on water bath 5. sample removed from water bath when volume reaches ~10 mL 6. hexane added directly to K-D body at start of concentration process	1. 5mL methanol added to all samples and blanks 2. extraction using Empore solid-phase extraction system 3. extract dried using Na_2SO_4 in drying columns 4. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 5. sample removed from water bath when volume reaches 1mL 6. solvent exchange via macro K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

PCB+ TCLP Pest
SEP

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - AQUEOUS PESTICIDE/PCB

Extraction Method: SW846 3520 (CLLE)____ SW846 3510 (SEP) ✓ SW846 3535 (SPE)____

Analytical Method: SW846 8081 ✓ SW846 8082____ EPA 608____ CLP OLM03.1____ CLP OLM04.2____ CLP OLC2.1____

Date QC Started: 3 31-06 QC Expiration Date: 4-14-06

Surrogate ID: 6C0315 Spike ID: 6C0303 (Pest) Spike ID: 6C0293 (PCB)

Solvent Lot # MeCl, C03H16 Solvent Lot #____

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction: Pest	Fraction: PCB	Final Vol. mL	Date Conc.	Tray Location	Initials	Clean-Up: GPC	Clean-Up: Flor	Clean-Up: Acid Wash	Clean-Up: Other	Comments
3 31-06	GN	WG266534-1	1000	1mL	NR	✓		10mL	3-31-06	PP665 F2	KF					R47185
		-2			1mL	✓				F3						
		-3				✓				F4						
		-4	200		NR	✓				F5						PB-7706A
4-4-06	GN	WG26746-1	1000	1mL	NR		✓	10mL	4-4-06	PP666 A5	KF			✓		R47319
		-2			1mL		✓			A6						Pest. Double spike
		-3					✓			A7						
4-5-06	GN	WG267785-1	1000	1mL	NR		✓	10	4-6-06	PP666 C5	EC			✓		R47373
		-2			1mL		✓			C6						
		-3					✓			C7						
4-14-06	TR	WG27077-1 WG27078-1	1000 mL	1mL	NR	✓	✓	10mL	4-25-06	PP667 E10	GN			✓		Pest: R47749 PCB: R47750
		WG27077-2			1mL	✓				E11						
		WG27078-2					✓			E12				✓		

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00001

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction: Pest	Fraction: PCB	Final Vol. mL	Date Conc.	Tray Location	Initials	Clean-Up: GPC	Clean-Up: Flor	Clean-Up: Acid Wash	Clean-Up: Other	Comments
4-4-06	GN	WW1246-8	200	1mL	NR	✓		10mL	3-31-06	PP665 F6	KF					TCLP
4-4-06	GN	WW1396-1	990	1mL	NR		✓	10mL	4-4-06	PP665 F8	KF			✓		
4-5-06	GN	WW1522-1	1060	1mL	NR		✓	10mL	4-6-06	PP666 C8	EC			✓		
		-2	1060				✓			C9				✓		
4-14-06	TR	WW1627-10	1060 mL	1mL	NR	✓	✓	10mL	4-25-06	PP667 F1	GN			✓		

QAEX119

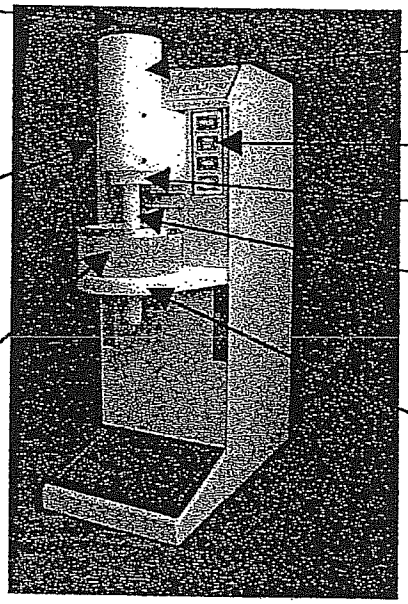
00001

Reviewed By____ Date____

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 F. Thomas Date: 1/26/01

Operations Manager: Joel C. Burton Date: 1/26/01

QA Officer: Deborah J. Nadeau Date: 1.26.01

General Manager: Duncan F. Hughes 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.	DN	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

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-516-03**, 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-516-03**, 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.

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 safety procedures and 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

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

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 Sodium Sulfate: Use sodium sulfate that has been heated at 400 □C to remove phthalates and other interfering organic substances. Combine 100 grams of sodium sulfate with enough hexane to just cover the solid, acidify by adding 0.2 mL of concentrated sulfuric acid and mix thoroughly. Remove hexane by covering and allowing to dry overnight in a hood. Mix 1 gram of the resulting solids with 5 mL of water and measure pH with pH paper. It must be below pH 4.

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 □g/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.

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: (H_2SIO_5) - 100 mesh powder.

5.16 10N Sodium Hydroxide

5.17 Sodium Chloride: (NaCl) - Pre-baked at 400°C for at least 4 hours.

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.

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 Culligan 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 ether rinse a 2 liter separatory funnel with stopcock and closure, a 500 mL glass sample bottle, and a 2 Liter Na_2SO_4 jar for each sample including blank, lab control sample and lab control sample duplicate.

7.5 Mark the meniscus on the sample bottles for later determination of initial sample volumes (See 7.16). 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.

7.6 Culligan DI 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 sample (50g for TCLP samples), seal and shake to dissolve the salt.

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 4 minutes on mechanical shaker. Let samples sit for 20 minutes, shake for 4 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 2 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 1-L samples (3.4ml to TCLP 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 4 minutes. Allow the phases to separate for 10 minutes.

7.15 Collect the aqueous (bottom) layer in a 2 liter Na_2SO_4 jar, and the ether (top) layer in a 500 mL sample bottle containing about 20g of acidified Na_2SO_4. Cap and shake the ether layer and drying agent. Return the aqueous layer to the separatory funnel.

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 Determine the initial volumes of the samples by filling the sample bottles with tap water up to the levels marked earlier. Using graduated cylinders, measure and record the sample volumes to the nearest 10 mLs.

7.18 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 Add an additional 50-60g of acidified Na2SO4 to the extract. Shake the extract (ether phase) and the dyring agent for one minute.

7.18 Allow the extract to remain in contact with the Na_2SO_4 for at least two hours, but, preferably overnight.

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 acetone, hexane, and then ether.

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 1.0 mL, remove from the steam bath and allow to drain and cool for several minutes. Use caution, the ether will evaporate repidly!

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 I. 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 <u>last</u> 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 form 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 (1-2 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. 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 <u>each</u> and <u>every</u> 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.

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 extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit 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

TABLE 1

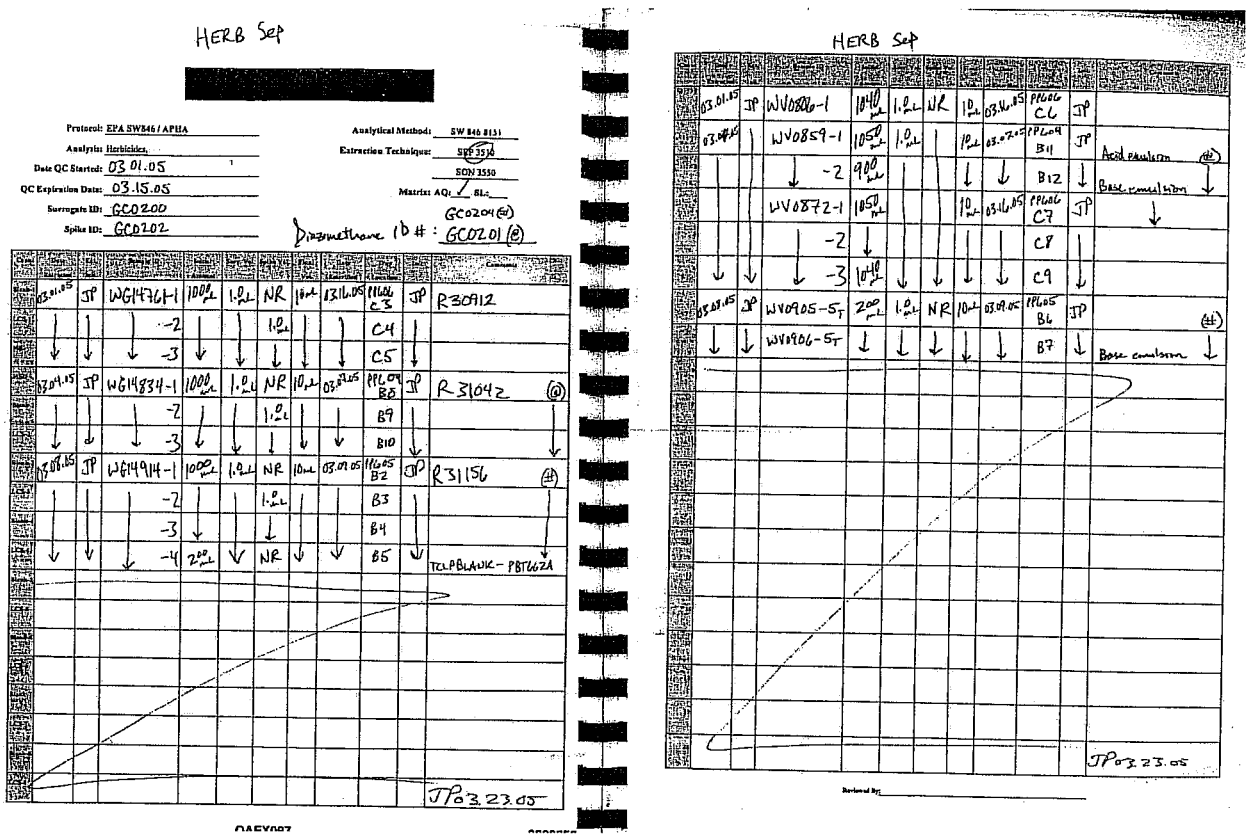
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-516-03	METHOD 8151, current revision
Apparatus/Materials	Use nitrogen blowdown technique.	Use two ball micro Snyder column.
Reagents	Unpreserved diethyl ether.	Ethyl ether preserved with BHT.
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS - METHOD 8151

FIGURE 1

EXAMPLE OF AQUEOUS HERBICIDE LOGBOOK PAGE



HERB Sep

[redacted]

Protocol: EPA SW846 / APHA
Analysis: Herbicides
Date QC Started: 03.01.05
QC Expiration Date: 03.15.05
Surrogate ID: GC0200
Spike ID: GC0202

Analytical Method: SW 846 8151
Extraction Technique: SPE 3510 / SDN 3550
Matrix: AQ ✓ SL
GC0204 (a)
Diazinon ID # : GC0201 (b)

03.01.05	JP	WG14761-1	1000 mL	1.0 mL	NR	10 mL	03.16.05	PPL06 C3	JP	R30912
		-2			1.0 mL			C4		
		-3						C5		
03.04.05	JP	WG14834-1	1000 mL	1.0 mL	NR	10 mL	03.17.05	PPL09 B8	JP	R31042 (a)
		-2			1.0 mL			B9		
		-3						B10		
03.08.05	JP	WG14914-1	1000 mL	1.0 mL	NR	10 mL	03.09.05	PPL05 B2	JP	R31156 (b)
		-2			1.0 mL			B3		
		-3						B4		
		-4	200 mL		NR			B5		TCLP BLANK - PBT662A

JP 03.23.05

HERB Sep

03.01.05	JP	WV0806-1	1040 mL	1.0 mL	NR	10 mL	03.16.05	PPL06 C6	JP	
03.04.05		WV0859-1	1050 mL	1.0 mL		10 mL	03.17.05	PPL09 B11	JP	Acid partition (b)
		-2	900 mL					B12		Base emulsion
		WV0872-1	1050 mL			10 mL	03.16.05	PPL06 C7	JP	
		-2						C8		
		-3	1040 mL					C9		
03.08.05	JP	WV0905-5T	200 mL	1.0 mL	NR	10 mL	03.09.05	PPL05 B6	JP	(b)
		WV0906-5T						B7		Base emulsion

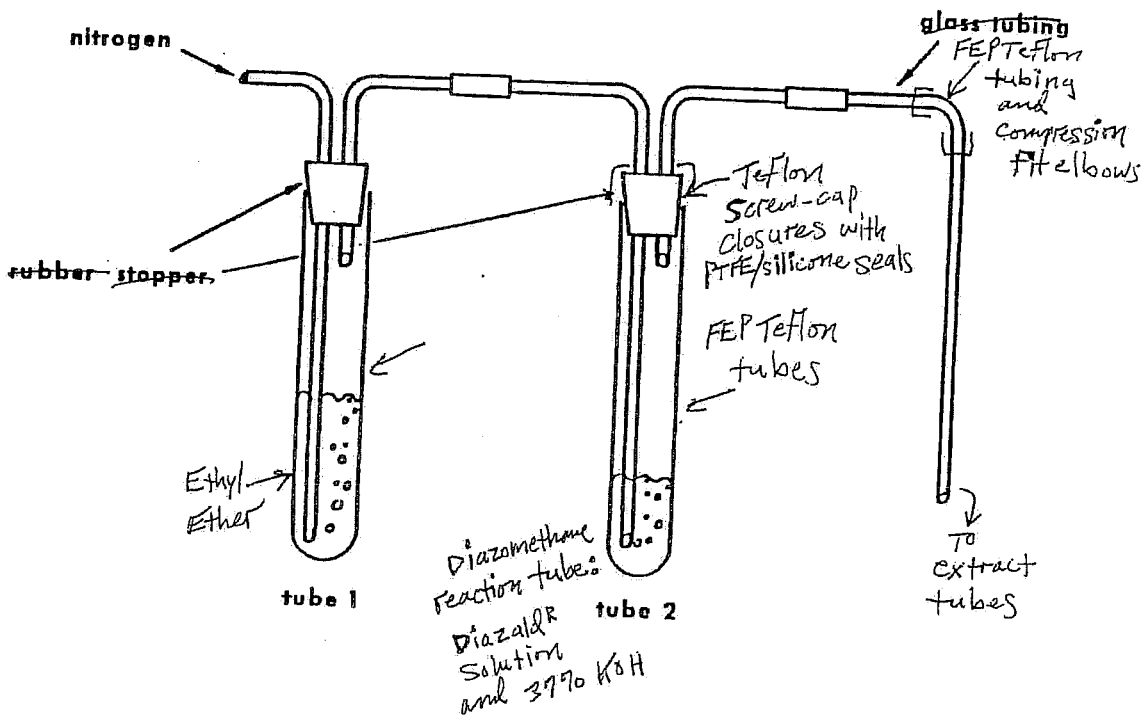
JP 03.23.05

Reviewed by: _____

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR HERBICIDE ANALYSIS - METHOD 8151

FIGURE 2

DIAZOMETHANE SOLUTION GENERATOR



TITLE: PREPARATION OF SOLID SAMPLES FOR HERBICIDE ANALYSIS BY METHOD 8151

Prepared By: _____ Date: 03.08.05

Approved By:

Department Manager: _____ Date: 3-8-05

Operations Manager: Deborah J. Nadeau Date: 3.8.05

QA Officer: Leslie Dimond Date: 030805

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Added LCSD criteria. Fixed typographical errors. Updated Logbook page	LAD	04/06	04/06

TITLE: PREPARATION OF SOLID SAMPLES FOR HERBICIDE ANALYSIS BY 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-517-01**, titled **PREPARATION OF SOLID 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-517-01**, titled **PREPARATION OF SOLID SAMPLES FOR HERBICIDE ANALYSIS BY METHOD 8151.**

Recipient: _____ Date: _____

TITLE: PREPARATION OF SOLID SAMPLES FOR HERBICIDE ANALYSIS BY METHOD 8151

1.0 SCOPE AND APPLICATION

This SOP describes the procedures used by Katahdin Analytical Services, Inc. technical personnel for the extraction and esterification of chlorinated acid herbicides in solid samples such as soils, sludges, and solid wastes. Specific analytes include those compounds on the Method 8151 list.

1.1 Definitions

EXTRACTION BATCH: Twenty or fewer samples which are extraxcted together with the same lots of reagents and with the manipulations common to each sample within the same time period.

LABORATORY REAGENT BLANK (METHOD 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 solid samples, a 1:2 (w/w) mixture of 400°C baked sand and sodium sulfate powder is used. The blank is taken through the appropriate steps of the process, just as any other sample.

LABORATORY CONTROL SAMPLE (LCS): A quality control sample that has been spiked with the analyte(s) from an independent source and is also 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 a 1:1 (w/w) mixture of 400 degree baked sand and sodium sulfate powder.

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, LCS/LCSD pairs, MS/MSD pairs and environmental 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 herbicides from solid 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".

It is the responsibility of all Katahdin technical personnel involved in the extraction of herbicides from solid samples to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and 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 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.

TITLE: PREPARATION OF SOLID SAMPLES FOR HERBICIDE ANALYSIS BY METHOD 8151

2.0 SUMMARY OF METHOD

This SOP provides extraction, hydrolysis, and derivatization conditions for chlorinated acid herbicides in soil. The samples are extracted with diethyl ether, are put through an hydrolysis process, and then esterified with diazomethane. The samples are then analyzed by gas chromatography with an electron capture detector.

3.0 INTERFERENCES

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to the disrete artifacts or elevated baseline in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis, by analyzing reagent blanks.

3.1.1 Glassware must be scrupulously cleaned. Clean each piece of glassware as soon as possible after rinsing it with the last solvent used in it. This should be followed by detergent washing with hot water and rinses with tap water, then with organic-free laboratory reagent grade water. Glassware should be solvent-rinsed with acetone, and pesticide-quality hexane. After rinsing and drying, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store glassware inverted or capped with aluminum foil. Immediately prior to use, glassware should be rinsed with the next solvent to be used.

3.2 Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis. However, hydrolysis may result in the loss of dinoseb and the formation of aldol condensation products if any residual acetone remains in the extraction.

4.0 APPARATUS AND MATERIALS

4.1 Ultrasonic cell disrupter: Heat Systems - Ultrasonics Inc., Model W385 (475 watt), affixed with Tapped Disrupter Horn, or equivalent. Settings: 10 (maximum), pulse mode, 50% duty, for three minutes.

4.2 Top loading balance: accurate to 0.01 g (Mettler PJ400).

4.3 500mL Erlenmeyer flask.

4.4 pH paper (0-14).

4.5 1.0mL gas tight volumetric syringe.

4.6 Water/Steam bath (for K-D solvent evaporation) Organomation S-Evap Model 120, or equivalent.

4.7 Kuderna-Danish apparatus:

- Concentrator tube, 10mL graduated
- Evaporator flask, 500mL
- Three ball macro Snyder column

4.8 Nitrogen blow-down apparatus (for concentrating extract in 10mL concentrator tube) Organomation N-Evap Model 111, or equivalent.

4.9 Pasteur pipette.

4.10 Whatman No. 1 filter paper.

4.11 12mL vial.

4.12 125mL Erlenmeyer flask.

4.13 250mL separatory funnel with cap and stopcock.

4.14 400mL beaker.

4.15 250mL amber jar.

4.16 Scoopula.

4.17 25mL graduated cylinder.

4.18 Buchner funnel.

5.0 REAGENTS

5.1 Potassium hydroxide (37%): Prepare by dissolving 37 g of potassium hydroxide pellets in laboratory reagent grade water and diluting to 100mL.

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. 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 250mL of sulfuric acid to 750mL of laboratory reagent grade 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 ug/mL DCAA (Dichlorophenylacetic acid) in acetone.

5.5 Underivatized Herbicide spiking solution: Contains 18 compounds at various concentrations – 100 ug/mL for all except MCPA and MCPP which are at 10,000 □g/mL. Dilute to 5.0 ug/mL and 500 □g/mL in methanol.

5.6 Diazald solution: weigh out 6.8 g of Diazald (99% N-methyl-N-nitroso-p-toluenesulfonamide) into a 50 mL volumetric flask and dissolve in a mixture of 25 mL carbitol and 25 mL of diethyl ether (50 mL of 1:1 v/v carbitol/ether). Bring to volume only after all of the Diazald is in solution. Use the sonicator bath briefly if necessary. This solution is stable for 1 month if kept at –10 to –20 °C.

5.7 Silicic Acid: 70-230 mesh

5.8 Acetone: pesticides residue grade or equivalent.

5.9 Diethyl ether

5.10 Methanol: pesticide residue grade or equivalent.

5.11 Carbitol: 2-(2-Ethoxyethoxy)ethanol

5.12 Hexane: pesticide residue grade or equivalent

5.13 Methylene chloride: pesticides residue grade or equivalent.

5.14 Iso-Octane: pesticides residue grade or equivalent.

5.15 Organic-free sand purified by baking at 400 °C overnight. Method blanks serve as checks on the baked sand.

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 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

INITIAL EXTRACTION

The extraction procedure is designed for the preparation of soil/sediment samples from an initial sample weight of 30g using an ultrasonic cell disruptor.

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 and the next solvent to be used in the extraction process.

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.

7.1 The following steps should be performed rapidly to avoid loss of the more volatile extractable. Weigh out a 30.0 ± 0.1g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.1g in appropriate extraction logbook. Add between 30g and 60g 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.

7.2 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 ± 0.1g portion of purified sand in a labeled 400 mL beaker. Add 60g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 60g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)

7.3 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30.0 ± 0.1 g portion of purified sand in a labeled 400 mL beaker. Add 30g sodium sulfate and mix well. 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.

7.4 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30g portions of the sample designated for MS/MSD into each of two labeled 400mL beakers. Record sample weights to nearest 0.1 g in appropriate extraction logbook. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. If sufficient volume of sample(s) is not available for an MS, MSD, the lab will extract an LSC, LCSD instead.

7.5 To all samples, method blank, LCS/LCSD, and MS/MSD if applicable, add 1.0 mL of the herbicide 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.

7.7 To the LCS/LCSD and the MS/MSD if applicable, add 1.0 mL of the herbicide matrix spike/LCS spiking solution using a 1.0mL gas tight syringe. 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 To assure optimum operation and maximum energy output, the sonicators must be tuned daily prior to extracting samples. The following tuning procedure must be performed with the sonicator probes vibrating in air.

7.8.1 Turn OUTPUT CONTROL knob counter-clockwise to zero. This automatically switches the duty cycle to continuous mode.

7.8.2 Press and hold down the power switch to on.

7.8.3 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.

7.8.4 Release the TUNE switch.

7.8.5 Turn OUTPUT CONTROL KNOB counter-clockwise to 50 and the power switch off.

7.8.6 Confirm that the sonicators were tuned by recording the date and/or percent in the extractions logbook.

7.9 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.

7.10 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. After step 7.14, repeat these steps with two more mixed and spiked samples.

7.11 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula to loosen up the mixture prior to extracting. Rinse the spatula with methylene chloride and collect the rinsing into a correspondent beaker.

7.12 Adjust all samples, including quality control, to pH 2 with approximately 1-3 mL of concentrated hydrochloric acid. Stir the samples well and continue to monitor the pH with pH paper for fifteen minutes with occasional stirring. If necessary, add additional hydrochloric acid until the pH remains at pH 2.

7.13 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. Sonicate for 3 minutes with the output control knob set at 10, and mode switch on "pulsed" and % duty cycle knob set at 50%. While the mixture is sonicating, one should be able to see all, or most of the material, moving in the beaker under the influence of the energized probes. If not, stir the mixture again.

7.14 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter. Prerinse the flask, funnel and filter with methylene chloride and discard rinsing into solvent waste container. Decant extract into the filter flask through the Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered.

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 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.

7.17 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mls of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mls of methylene chloride and allow to drain.

7.18 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.19 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. **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. Be sure to keep track of which column goes with which apparatus because they will be used again in the next step. At this time the steam bath should be reset to and allowed to reach a temperature of 60-65°C.

HYDROLYSIS

7.20 To each K-D flask, add 2-4 more boiling stones, 5mL of 37% KOH, and 30mL laboratory reagent grade water.

7.21 Replace the appropriate Snyder column and allow contents to reflux on the steam bath until the hydrolysis step is complete Usually 1-2 hours).

7.22 Remove all of the K-D flasks from the steam bath and allow to cool for 10-15 minutes.

CLEANUP – METHYLENE CHLORIDE WASH

7.23 Transfer contents from each K-D apparatus into a 250mL separatory funnel. Rinse the empty K-D flask/concentrator tube setup three times with methylene chloride, and add these rinses to the separatory funnel. Be sure to appropriately label all separatory funnels to correspond with its respective sample extract.

7.23 Make certain the pH of the contents in each K-D apparatus is still basic (pH > 12). If not, add small amounts of 37% KOH until the desired pH is obtained.

7.23 Add 100mL of methylene chloride to the separatory funnel. Cap tightly, shake, and vent briefly before shaking for 3 minutes. Allow phases to separate for approximately 10 minutes. Drain methylene chloride layer (bottom) out through stopcock and directly into an "O" flammable waste container.

7.23 Repeat the last step twice and discard the methylene chloride phase and keep the aqueous phase each step.

SECONDARY EXTRACTION

7.26 Adjust the pH of the solution (aqueous phase) to <2 by adding 5mL of COLD (4°C) 1:3 sulfuric acid to the separatory funnel. It is necessary to use cold acid to help overcome the high heat that is generated when sulfuric acid is diluted with laboratory reagent grade water. Add additional acid as necessary.

7.27 Add 40mL of diethyl ether to the separatory funnel. Cap tightly, shake, and vent briefly before shaking for 3 minutes. Allow the two phases to separate for approximately 10 minutes. Use a 125mL Erlenmeyer flask to collect the (bottom) aqueous phase. Collect the (top) ether phase in a 250mL wide mouth ether rinsed amber jar containing approximately 20g of acidified sodium sulfate.

7.28 Return the aqueous phase to the separatory funnel. Extract the aqueous phase two more times with 20 mL of ether each time. Combine the ether phases with the first ether phase in the amber jar. Check that the pH is <2 before transferring the aqueous phase back to the separatory funnel each time.

7.29 Neutralize the aqueous phase in a hood, then dispose of it in the appropriate aqueous waste container, marked "Acid/N-low").

7.30 Cap and shake the amber jar briefly and allow the extracts to remain in contact with the sodium sulfate for at least 4 hours. (If concentration and esterification are not to be performed immediately, store extracts overnight in a refrigerator).

EXTRACT CONCENTRATION

7.20 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with ether 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 ether. Place the assembled K-D's under the funnels.

7.21 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. Use a glass rod to crush any caked sodium sulfate in the amber jar. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with 10 mls of ether. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mls of ether and allow to drain.

7.22 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column to the K-D evaporative flask. Pre-wet the Snyder column with 1 mL of ether.

7.23 Place the K-D in a hot water bath (60-65°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 ether. 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 ether.

7.24 Reduce the extract in the concentrator tube to approximately 0.5mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent. 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 ether. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.

7.25 When the apparent volume reaches 0.5 mL, remove the concentrator tube and allow it to cool.

7.36 Add 0.5mL methanol and 1.0mL Iso-octane to the collector and rinse the walls of the collector while adjusting the volume to 4.0 mL with ether.

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.
- Use a MLA pipette with disposable tip for pipetting.
- 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.

7.37 Assemble the diazomethane bubbler (see Figure 2).

7.38 Add 5.0 mL of ether to tube 1. Add 1.5mL of 37% KOH and about 2-3mls of Diazald solution to tube 2. Add the 37% KOH solution last to begin the reaction, and quickly insert the stoppers into both tubes.

7.39 Apply a nitrogen flow of 3-5 mL/min so bubbles emerge steadily, but not rapidly. 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. Bubble directly into the sample extract's concentrator tube.

7.40 Remove the exit tube from the collector, cover, and store at room temperature for 20-30 minutes.

7.41 Destroy any unreacted diazomethane by adding 0.1 to 0.2g silicic acid to the collectors. Allow to stand until the evolution of nitrogen is complete. Pipette the sample into a hexane rinsed 12mL vial and rinse the collector twice with hexane (1-2 mL each time). Be careful to leave the silicic acid in the concentrator tube. Bring to a final volume of 10.0 mL, mix well, and let sit for a few minutes.

7.42 The extract is now ready for analysis. Make sure that initial volumes, intermediate volumes, aliquot volumes, and final volumes have been recorded in appropriate logbook. Also verify that the surrogate and spike identification numbers and amounts used have been recorded. Any deviations from the SOP or any abnormal sample observation should be noted as comments.

7.43 The data entered here are used later 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

A method blank and laboratory control sample (LCS) must be extracted for every extraction batch of twenty or fewer samples.

Refer to the current revision of the applicable Katahdin SOP for analysis of Herbicides for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the

analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The 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.

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 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 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, Method 8151.

Katahdin SOP CA-305, Analysis of Chlorinated Herbicides by GC Using Methylation Derivatization: SW-846 Method 8151, current revision.

LIST OF TABLES AND FIGURES

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Figure 1 Example of Extractions Logbook
Figure 2 Diazomethane Solution Generator

TITLE: PREPARATION OF SOLID SAMPLES FOR HERBICIDE ANALYSIS BY METHOD 8151

TABLE 1

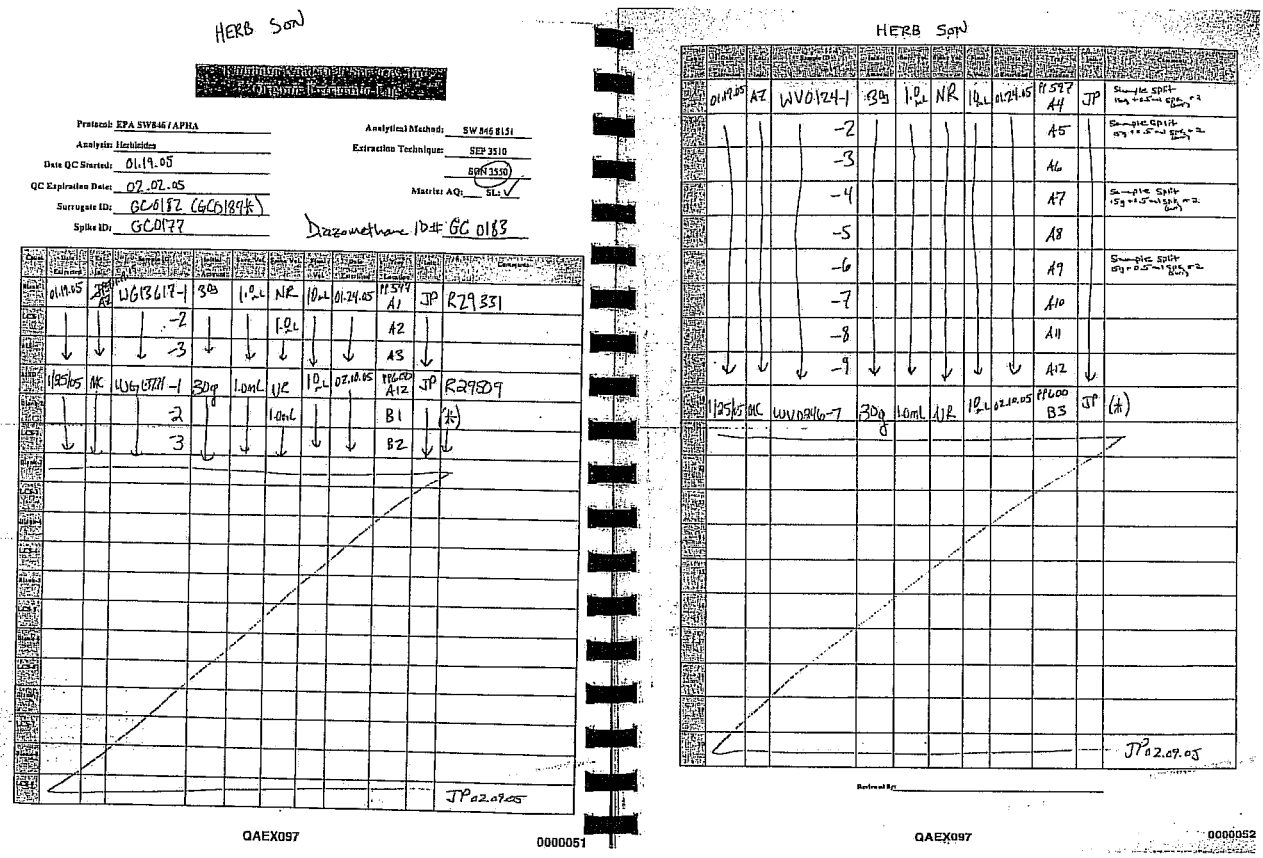
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-517-01	METHOD 8151
Apparatus/Materials	Use nitrogen blowdown technique.	Use two ball micro Snyder column.
Reagents	5.2 Sodium sulfate Do not bake. Do not use ether. Do not store at 130 °C.	5.10 Sodium sulfate –
Sample preservation/ handling		
Procedures	7.10 - 7.12 pH adjusted after spiking and addition of solvent.	7.2.1 pH adjusted prior to spiking and solvent addition.
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: PREPARATION OF SOLID SAMPLES FOR HERBICIDE ANALYSIS BY METHOD 8151

FIGURE 1

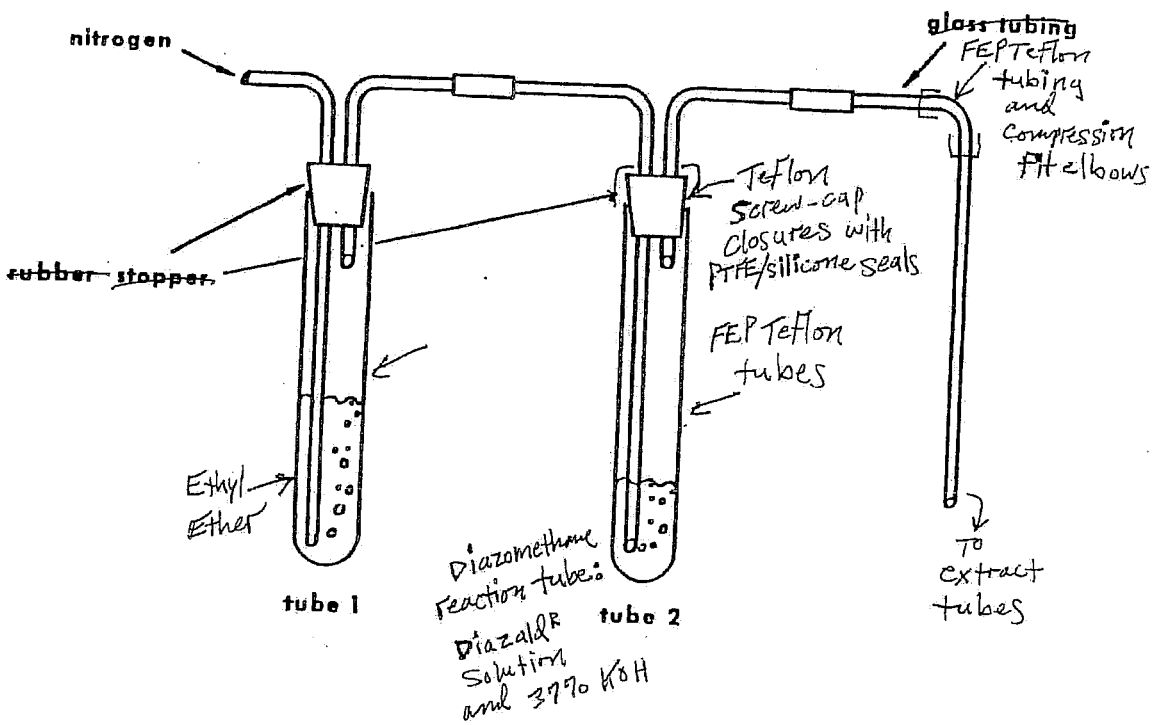
EXAMPLE OF HERBICIDE SOILS EXTRACTIONS LOGBOOK PAGE



TITLE: PREPARATION OF SOLID SAMPLES FOR HERBICIDE ANALYSIS BY METHOD 8151

FIGURE 2

DIAZOMETHANE SOLUTION GENERATOR



KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-524
Revision History
Cover Page
Page 1

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS

Prepared By: Mike Thomas Date: 7/98

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: J Benton Date: 11/15/00

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General Manager: Dennis C. Hufnagel Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	11.15.00	11/15/00
02	Added definitions to section 1.1. Wording changed or added to clarify sections 5, 6, 8, + 9. New figure	MRC	11.08.04	11.08.04
03	Sect. 7.1.2 - adding the step to rinse forceps also. 7.10 adding condenser temperature and output voltage of variable transformer	LAD	04/06	04/06
04	Added generated waste information. Updated spike list. Added LCSD. Reworded Sect. 7.10 and 7.11 for clarification. Updated Table 1 Replaced Figure 1	LAD	09/07	09/07

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

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

I acknowledge receipt of copy ___ of document **SOP CA-524-04**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS.**

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KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-524-04**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure for extracting pesticides/PCBs from solids such as soils, sludges, and wastes using Method 3540. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures including methods 8081 for pesticides and 8082 for PCB's.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for pesticide/PCB analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this

method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training and Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for pesticide/PCB analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and 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 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the "N-Lo" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.

2.2 The extract is then dried, concentrated, and exchanged into hexane for GC analysis. Sulfuric acid cleanup is performed on extracts for 8082 PCB analysis.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

4.1 a) Soxhlet extractor – 45/50 top joint and 24/40 lower joint.

b) 500 mL flat-bottom boiling flask

c) Allihn cooling water condenser

4.2 Powder Funnels – 100 mm top diameter, 35 mm stem

4.3 Kuderna-Danish (K-D) apparatus

4.3.1 Concentrator tube - 10-mL

4.3.2 Evaporation flask - 500-mL

4.3.3 Snyder column - Three-ball macro

4.4 Nitrogen evaporation (N-EVAP) apparatus.

4.5 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride

4.6 Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

4.7 Vials - Glass, 4, 12, or 16 mL with Teflon-lined screw caps

4.8 Glass wool (fiberglass) - baked at 400°C for a minimum of 4 hours or overnight.

4.9 Heating mantles - Rheostat controlled.

4.10 Disposable glass Pasteur pipets, 5 ¾", and bulbs.

4.11 Drying oven - capable of maintaining 105°C for glassware drying.

4.12 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.13 Beakers, 250 or 400 mL

4.14 Top-loading balance - capable of weighing to 0.1 g.

4.15 Spatulas, stainless-steel

4.16 Long forceps, stainless-steel

4.17 Metal clips – for securing Soxhlets to boiling flasks

5.0 REAGENTS

5.1 Sodium sulfate (granular, anhydrous and powdered, anhydrous) (ACS reagent grade), Na_2SO_4. Certified by the manufacturer/vendor as purified by heating at 400°C for 4 hours prior to receipt by the laboratory.

5.2 Methylene chloride - (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300 mL to 1 mL followed by GC/MS analysis.

5.3 Acetone and hexane – (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300 mL to 1 mL followed by GC/MS and GC analysis.

5.4 Organic-free sand, purified by baking at 400 °C at a minimum of 4 hours or overnight. Method blanks serve as checks on the baked sand.

5.5 Surrogate spiking solution - Prepare a solution of decachlorobiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL in acetone.

5.6 Matrix Spike/Lab Control Sample spiking solution

5.6.1 Pesticide spike solution – prepare in pesticide grade methanol containing the analytes listed below at concentrations of 0.5 ug/mL.

4,4'-DDD
4,4'-DDE
4,4'-DDT
Aldrin
alpha-BHC
beta-BHC
delta-BHC
Dieldrin
Endosulfan I

Endosulfan II
Endosulfan Sulfate
Endrin
Endrin Aldehyde
Endrin Ketone
gamma-BHC (Lindane)
Heptachlor
Heptachlor Epoxide
Methoxychlor
alpha-Chlordane
gamma-chlordane

5.6.2 PCB spike solution – prepare Aroclor 1660 (Aroclor 1016 and 1260) in pesticide grade acetone at a concentration of 5.0 ug/mL each.

5.7 Store the solutions mentioned in sections 5.5 and 5.6 at -10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3540 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

7.1 Preparing the Soxhlet Extraction Apparatus

7.1.1 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.

7.1.2 Add ~ 250 mLs of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Rinse the stainless steel forceps and pre-baked glass wool with Methylene chloride. Working in a hood, place a plug of the

glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample.

7.2 Sample Handling

7.2.1 Sediment/soil samples - Decant and discard any water layer on a sediment sample. Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2 Gummy, fibrous, or oily materials not amenable to mixing should be cut, shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

7.3 Weigh out a 30.0 ± 0.1 g portion of sample into a labeled 400 mL beaker. Record sample weight to nearest 0.1 g in appropriate extraction logbook. Add between 30 to 60 g of powdered sodium sulfate, as required, to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and <u>cover</u> 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 30.0 ± 0.1 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 30.0 ± 0.1 g portion of purified sand in a labeled 400 mL beaker. Add 30 g sodium sulfate and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB LCS's must be prepared (refer to section

5.6). If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30.0 ± 0.1 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.1 g in appropriate extraction logbook. Add 30 g sodium sulfate to each to produce a free-flowing mixture, and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB MS/MSD pairs must be prepared (refer to section 5.6).

7.7 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be careful not to have any of the solid material fall into the extract flask through the large vapor tube.

7.8 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the pesticide/PCBs surrogate spiking solution using a 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in the extraction logbook. Thoroughly rinse syringe with solvent between each use.

7.9 To LCS/LCSD and MS/MSD add 1.0 mL of either the pesticide or PCBs matrix spike/LCS spiking solutions using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification codes in the extraction logbook. Thoroughly rinse syringe with solvent between each use.

7.10 Rinse the joints of the Allihn cooling condensers with Methylene Chloride, collecting the waste in a methylene chloride solvent waste container. Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water condensers into the 45/50 joints of the extractors. The condensers should be set to a temperature of 15°C. Save the pieces of aluminum foil for covering the Soxhlets when the extraction is complete. Switch on the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 55% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until the level reaches that of the small siphon tube and then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 18-24 hours. Be sure the chiller/recirculator temperature is set low enough to provide enough cooling capacity for the number of extractions in the batch.

7.11 When the extraction is complete, allow the extracts to cool before dismantling. Remove the Allihn condenser and replace the aluminum foil on top of the extractor. Move the extractors to a hood and detach the extractor from the extract flask. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Try to drain as much solvent as possible from the extractor into the flask. Cover the flask with aluminum foil and store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

7.12 Immediately remove the extracted soil/sodium sulfate mixtures from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries.

CONCENTRATION OF THE EXTRACTS

7.13 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add a few boiling chips to the K-D. Insert fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels.

7.14 For a solvent exchange, add approximately 50 mL Hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only.

7.15 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.

7.16 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.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

7.17 If samples are not to be GPC'd follow Steps 7.17 through 7.23 to concentrate extracts to final volume of 10.0 mLs. Otherwise proceed to GPC cleanup procedure as described in the current revision of Katahdin SOP CA-513.

7.18 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.19 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. 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.

7.20 Reduce the extract in the concentrator tube to approximately 10 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. <u>During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration.</u> Note any problems or extract losses, if they occur, in the extractions logbook.

7.21 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane extract to 10 mL in either a 12 or 16 mL vial using the appropriate "reference vial" for volume comparison.

7.22 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and "tray location" of the individual extract vials.

7.23 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. All sample extracts for 8081 pesticide analysis do not undergo further cleanup unless requested by the client. All sample extracts for combined 8081/8082 analyses must be split. One portion must be acid cleaned for

8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. 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.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticide and/or PCB) to be performed

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality

Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit 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 3540C, SW-846, Third Edition, Updates I, II, IIA, IIB, and III Revised December 1996, US EPA.

LIST OF TABLES AND FIGURES

Table 1 Summary of Method Modifications
Figure 1 Example of Logbook Page

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-524-04	METHOD 3540, current revision
Apparatus/Materials	1. short stem funnels	1. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. Use 30 grams of sample and 30 grams of sodium sulfate. 2. Use 250 mL of methylene chloride 3. no apparatus height specification for concentration on water bath 4. water bath at 75-85 deg C 5. sample removed from water bath when volume reaches ~6 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane at the start of concentration process	1. Use 10 grams of sample and 10 grams of sodium sulfate. 2. Use 300 mL of methylene chloride 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. water bath at 80-90 deg C 5. sample removed from water bath when volume reaches 1-2 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

PCB SOP

Nitrogen Bath temp 35 °C

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: SW846 3550 (sonication)____ SW846 3540 (soxhlet) X SW846 3545 (ase)____

Analytical Method: SW846 8081____ SW846 8082 X CLP OLM03.1____ CLP OLM04.2____

Date QC Started: 7-30-07 QC Expiration Date: 8-13-07 QC Batch ID____

Surrogate ID: GCO 437 Spike ID: GCO 423 Spike ID:____

Solvent Lot # $MeCl_2$: E26E70 Solvent Lot # Hexane: E20E17 Sonicator Horns Tuned:____

Date Extracted	Ext. Init.	Sample ID	Initial Vol.	Surr. Vol.	Spike Vol.	Fraction Pest	Fraction PCB	Final Vol.	Date Conc.	Tray Location	Initials	Clean-Up GPC	Clean-Up Flor.	Clean-Up Acid Wash	Clean-Up Other	Comments
7-30-07	Km	WG41673-1	30.01	1mL	NR		✓	10mL	7-31-07	PP132 B9	GN			✓		RG7718
		-2	30.00		1mL		✓									Lost - N. Bath
		-3	30.04				✓	10mL	7-31-07	B10	GN			✓		
								GN	7-31-07							

QAEX133

0000133

Date Extracted	Ext. Init.	Sample ID	Initial Vol.	Surr. Vol.	Spike Vol.	Fraction Pest	Fraction PCB	Final Vol.	Date Conc.	Tray Location	Initials	Clean-Up GPC	Clean-Up Flor.	Clean-Up Acid Wash	Clean-Up Other	Comments
7-30-07	Km	SA3963-1	29.96	1mL	NR		✓	10mL	7-31-07	PP132 B11	GN			✓		
		SA3978-1	30.00							B12						
		-2	29.99							C1						
		-3	30.00							C2						
		-4	30.01							C3						
		-5	30.02							C4						
		-6	29.97							C5						
		SA3987-1	30.01							C6						
		-2	30.02							C7						
		-3	29.98							C8						
		-4	30.01							C9						
							GN		7-31-07							

QAEX133

0000134

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-526
Revision History
Cover Page
Page 1

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 C Benton Date: 11/15/00

QA Officer: Deborah J. Nadeau Date: 11·16·00

General Manager: Dennis F. Hughes Date: 11/20/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	11·16·00	11/16/00
02	Definitions added to section 1.1. Wording was added or changed to clarify sections 4, 5, 6, 7, 8 & 9. Minor changes throughout. New figures.	MRC	11.09.04	11.09.04
03 LAD 6-26-06	Updated Sect. 7.0 to include SIM. Updated figures 2 and 3 to include current SVOA ~~spikes~~ compounds used. Updated Sect. 5.0 to include all compounds analyzed for. Updated logbook page. minor edits throughout.	LAD	04/06	04/06
	Added waste generated information. Updated Spikes and Surrogates. Added SIM LCSD and MS/D requirements. Updated Table 1. Added GPC references. Added LCSD after LCS.	LAD	09/07	09/07

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-526-04**, 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-526-04**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for extracting semivolatile organic compounds from solids such as soils, sludges, and wastes using Method 3540. The Soxhlet extraction process ensures intimate contact the sample matrix with the extraction solvent.

This method is applicable to the isolation and concentration of water-insoluble and slightly water soluble organics in preparation for a variety of chromatographic procedures.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to

Katahdin SOP QA-805, current revision, "Personnel Training and Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, to assure that their work is properly documented, and to indicate periodic review of the pertinent logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and 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 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste,

and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.

2.2 The extract is then dried and concentrated for subsequent 8270 Semivolatile Organics analysis.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be needed to minimize interferences.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

4.0 APPARATUS AND MATERIALS

4.1 Soxhlet apparatus:
- a) Soxhlet extractor – 45/50 top joint and 24/40 lower joint.
- b) 500 mL flat-bottom boiling flask
- c) Allihn cooling water condenser

4.2 Powder Funnels – 100 mm top diameter, 35 mm stem

4.3 Kuderna-Danish (K-D) apparatus

4.3.1 Concentrator tube - 10-mL

4.3.2 Evaporation flask - 500-mL

4.3.3 Snyder column - Three-ball macro

4.4 Nitrogen evaporation (N-EVAP) apparatus.

4.5 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride

4.6 Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

4.7 Vials - Glass, 1.8-mL capacity, with polytetrafluoroethylene (PTFE)-lined septum vials, and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.

4.8 Glass wool (fiberglass) - baked at 400°C for a minimum of 4 hours or overnight.

4.9 Heating mantles - Rheostat controlled.

4.10 Disposable glass pasteur pipets, 5 ¾" and bulbs.

4.11 Drying oven - capable of maintaining 105°C for glassware drying.

4.12 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.13 Beakers, 250 or 400 mL

4.14 Top-loading balance - capable of weighing to 0.1 g.

4.15 Spatulas, stainless-steel

4.16 Long forceps, stainless-steel

4.17 Metal clips – for securing Soxhlets to boiling flasks

5.0 REAGENTS

5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, certified by the manufacturer/vendor as purified heating to 400°C prior to receipt by the laboratory.

5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated, by lot, prior to use, by concentration of approximately 400 mL to 1.0 mL followed by GC/MS analysis.

5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand.

5.4 Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound	Conc.
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.6 Base/Neutral and Acid (SVOA) Lab Control Sample / Matrix Spike Spiking Solution - Prepare a spiking solution in methanol that contains the following mixes listed in Figure 2 at a concentration of 50 ug/ml for the base/neutral compounds and 100 ug/ml for the acid compounds. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.7 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2 ug/mL for base/neutral. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL in methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.8 Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution – Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at concentrations of 100 ug/ml. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3540 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

Some solid samples may need to be cleaned up to reduce matrix interferences. The cleanup procedure employed is gel permeation chromatography (GPC). The organic department manager should be consulted to determine if a particular sample should be subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs.

Sign chain-of-custody when removing and replacing samples in storage locations, and fill out the sample preparation/extraction log with the necessary information before starting the extraction. Prerinse all glassware three times with methylene chloride.

7.1 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.

7.2 Sample Handling

7.2.1 Sediment/soil samples - Decant and discard any water layer on a sediment sample. Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2 Gummy, fibrous, or oily materials not amenable to mixing should be cut,

shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

7.3 The following steps should be performed rapidly to avoid loss of the more volatile extractables. Weigh out a 30.0 ± 0.1 g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.1 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 30.0 ± 0.1 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 30.0 ± 0.1 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 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. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis.

7.7 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be

careful not to have any of the solid material fall into the extract flask through the large vapor tube.

7.8 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the appropriate base/neutral and acid surrogate spiking solution listed below using the pre-rinsed 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution.

7.8.1 If the request is for SVOA, use the SVOA surrogate solution (sect. 5.4)

7.8.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5).

7.8.3 If the request is for SIM-SVOA, use both the SIM and SVOA surrogate solutions. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.9 To the LCS/LCSD and the MS/MSD add 1.0 mL of the appropriate base/neutral and acid (SVOA) matrix spike/LCS spiking solution listed below using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent when spiking is completed.

7.9.1 If the request is for SVOA, add 1 mL of the SVOA spiking solution (sect. 5.6).

7.9.2 If the request is for SIM, add 1 mL of the SIM Spiking solution (sect. 5.7).

7.9.3 If the request is for SVOA/SIM, add 1 mL of the SVOA spiking solution and 1 mL of the SIM Spiking solution (sect's 5.6 and 5.7).

7.9.4 If the request is for SVOA Appendix IX, add 1 mL of the SVOA Appendix IX spiking solution and 1 mL of the SVOA spiking solution (sect's 5.6 and 5.8).

7.10 Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water condensers into the 45/50 joints of the extractors. Save the pieces of aluminum foil for covering the Soxhlets when the extraction is complete. Switch on the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 55-60% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until the level reaches that of the small siphon tube and then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 18-24 hours. Be sure the chiller/recirculator temperature

is set low enough to provide enough cooling capacity for the number of extractions in the batch.

7.11 When the extraction is complete, allow the extracts to cool before dismantling. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Remove the Allihn condenser and replace the aluminum foil on top of the extractor. Move the extractors to a hood and detach the extractor from the extract flask. Try to drain as much solvent as possible from the extractor into the flask. 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.

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 to drain.

7.15 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.

7.16 If samples are not to be GPC'd follow Steps 7.17 through 7.22 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.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

7.17 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.18 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 4-6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-extraction must occur immediately.** Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.19 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.

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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8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of extractable semivolatile organics for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases

the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs 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 3540C, SW-846, Third Edition, Updates I, II, IIA, IIB, and III Revised December 1996, US EPA.

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TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

TABLE 1

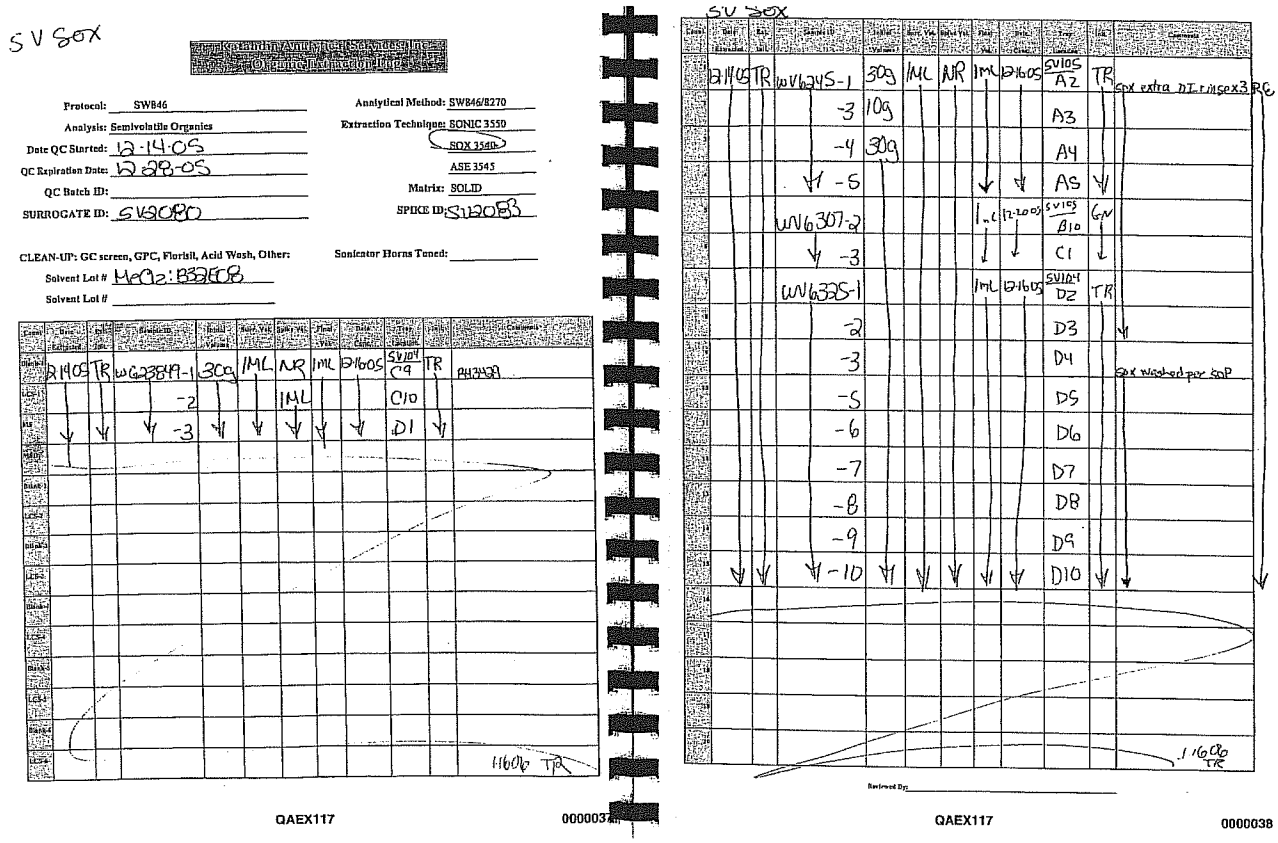
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-526-04	METHOD 3540, current revision
Apparatus/Materials	1. short stem funnels	2. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. Use 30 grams of sample and 30 grams of sodium sulfate 2. Place a plug of glass wool in soxhlet then add sample 3. Use 250 mL of methylene chloride for extraction 4. Extract the sample for 18 - 24 hours 5. Extract dried using Na_2SO_4 in short stem funnels 6. Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 7. no apparatus height specification for concentration on water bath 8. Water bath at 75-85 deg C 9. Sample removed from water bath when volume reaches ~6 mL	1. Use 10 grams of sample and 10 grams of sodium sulfate. 2. Place sample between 2 plugs of glass wool 3. Use 300 mL of methylene chloride for extraction 4. Extract the sample for 16 - 24 hours at 4 - 6 cycles/hour 5. Extract dried using Na_2SO_4 in drying columns 6. Wash the extractor flask and sodium sulfate column with 100 to 125 mL of extraction solvent to complete the quantitative transfer 7. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20min 8. Water bath at 15-20 deg C above solvent boiling point 9. Sample removed from water bath when volume reaches 1-2 mL
QC - Spikes	1. Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1. Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1. Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1. Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC – MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Benzyl alcohol
1,1-Biphenyl	Bis (2-chloroethoxy) methane
1,2,4-Trichlorobenzene	Bis (2-chloroethyl) ether
1,2-Dichlorobenzene	Bis (2-Chloroisopropyl) ether)
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	Dimethyl phthalate
3-Nitroaniline	Di-n-butylphthalate
4-Bromophenylphenyl ether	Di-n-octyl phthalate
4-Chloroaniline	Fluoranthene
4-Chlorophenylphenyl ether	Fluorene
4-Nitroaniline	Hexachlorobenzene
Acenaphthene	Hexachlorobutadiene
Acenaphthylene	Hexachlorocyclopentadiene
Acetophenone	Hexachloroethane
Aniline	Indeno (1,2,3-cd) pyrene
Anthracene	Isophorone
Atrazine	Naphthalene
Azobenzene	Nitrobenzene
Benzaldehyde	N-Nitrosodimethylamine
Benzidine	N-Nitroso-di-n-propylamine
Benzo (a) Anthracene	N-Nitrosodiphenylamine
Benzo (a) pyrene	Phenanthrene
Benzo (b) fluoranthene	p-toluidine
Benzo (ghi) perylene	Pyrene
Benzo (k) fluoranthene	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

TITLE: 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: Deborah J. Nadeau Date: 1·22·01

General Manager: Dennis 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 digestates for ICP-MS analysis. Added use of block digester as primary heating source & adjusted volumes. Revised standard solution names & concs. in Figures 3&4.	DN	8·29·02	8·29·02
03	Added Uranium to spiking solutions for LCS & MS/D. Removed the Internal Custody Record for Metals Digestates figure and reference.	LAD	04/06	04/06

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-604-03**, 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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STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **SOP CA-604-03**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure utilized by Katahdin Analytical Services, Inc. personnel to solubilize metals in aqueous samples, wastes that contain suspended solids, and mobility-procedure extracts prior to analysis by inductively coupled plasma atomic emission spectroscopy (ICP) and inductively coupled plasma mass spectrometry (ICP-MS). This SOP applies to samples prepared by EPA Method 3010, with the method modifications mentioned in Table 2.

1.1 Definitions - none.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of aqueous samples by EPA Method 3010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of aqueous samples using EPA Method 3010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their work in the appropriate lab notebook. Any deviations from the method or irregularities with the samples should also be recorded in the lab notebook and reported to the Supervisor or designated qualified data reviewer responsible for these data.

It is the responsibility of the Supervisor to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, rubber aprons, dust masks, and rubber shoe protectors, is available in the metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever beakers are being heated in the hood. Use caution when handling hot beakers.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Chemical Hygiene Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Any other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Chemical Hygiene Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision.

2.0 SUMMARY OF METHOD

The aqueous sample is refluxed with nitric acid in a covered digestion vessel. Additional nitric acid is added until the color of the digestate has stabilized. After the digestate has been evaporated to a low volume, it is refluxed with hydrochloric acid and diluted to the appropriate final volume with reagent water.

Samples may be concentrated (i.e. final digestate volume less than initial sample volume) during digestion if lower detection limits are required. Volumes of reagents and spiking standards must be added in proportion to the final volume of the digestate. Because

concentration of samples during digestion increases the concentrations of dissolved solids and may exacerbate analytical interferences, concentration factors greater than 5 are not recommended.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1. 250 mL and 400 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning") for digestion using a hot plate. If digestion will be performed using a block digester, 70ml graduated, polyethylene block digester tubes (with attached snap caps) will be used instead of glass beakers.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter and 100 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate, block digester, or other heating source - adjustable and capable of maintaining a temperature of 90-95°C. Hot plates must be numbered for easy identification.

4.6 Device for measuring hot plate temperature. This may consist of a heat-resistant 100ml beaker containing reagent water in which a thermometer is immersed. When using a block digester, a digestion tube containing reagent water in which a thermometer is immersed may be used. The temperature of one hot plate is measured each day, on a rotating basis. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity. These are not necessary when using the block digester since the final digestates are stored in the digestion tubes.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid and 1:1 HCl.

5.0 REAGENTS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Hydrochloric acid, 1:1. Add a volume of concentrated hydrochloric acid to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 Multi-element spiking solutions (as listed in Figure 3).

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

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.

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 and print labels for the digestate containers. Print out a copy of the spreadsheet. Attach labels to the polyethylene sample containers that will contain the digestates.

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digester do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 If digestion is performed using a block digester, the sample aliquot may be measured in the digestion vessel using the graduations on the digestion tubes. Measure 50 ml of well-mixed sample into a 70 ml block digestion tube. A larger sample aliquot may be used (up to 250 mL) if concentration of the sample during digestion is desired. Sample volumes larger than 50 mL may be digested in 250 mL beakers. Measure aliquot of well-mixed sample into a graduated specimen cup and transfer into a properly cleaned 250 mL beaker. Sample volumes of more than 50ml may not be digested using the 70ml block digester tubes. The volumes of reagents and spiking solutions used must be adjusted in proportion to the final digestate volume. The reagent and spiking solution volumes listed below are based on a final volume of 50 mL.

7.4 Add spike solutions to matrix spike samples and laboratory control samples (refer to Figure 3 for spiking instructions).

7.5 Use a repipetter to add 1.5 mL of concentrated HNO_3 (per 50 mL final volume) to the sample. Cover with a ribbed watch glass and place on heatsource. Heat cautiously, without boiling the sample, and evaporate to a low volume (10 - 15 mL).

NOTE: Do not allow any portion of the bottom of the digestion vessel to go dry during any part of the digestion. If a sample is allowed to go to dryness, low

recoveries may result. Should this occur, discard the digestate and re-prepare the sample.

7.6 Cool the sample and add another 1.5 mL aliquot (per 50 mL final volume) of concentrated HNO_3. Cover and resume heating, increasing the temperature until a gentle reflux action occurs.

7.7 Continue heating, adding additional acid as necessary, until the digestate is light in color or does not change in appearance with continued refluxing.

7.8 Evaporate digestate to a low volume (10 - 15 mL).

7.9 Cool the sample and use a repipetter to add 5 mL (per 50 mL final volume) of 1:1 HCl. Cover the sample and resume heating, refluxing for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

7.10 Allow the sample to cool.

7.11 If the digestate contains visible particulate material, it must be filtered. Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated plastic specimen container or block digester digestion tube. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container or digestion tube, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse.

If the digestion was performed using hot plates and the digestate does not contain particulate material, simply decant the digestate into a clean graduated specimen container (or graduated sample container with attached snap lid), rinse the beaker with reagent water, and add the rinsates to the container.

If the digestion was performed using a block digester and the digestate contains no visible particulate material, the digestate may be brought to final volume and stored in the digestion tube without decanting or rinsing.

7.12 Using the graduations on the specimen container, snap-lid container or digestion tube, dilute to the required final volume with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container or digestion tube has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for analysis.

7.13 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a

corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) reagent lot numbers, spiking information, initial and final volumes, and hot plate temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 1.

7.14 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

7.15 A condensation of the procedure described above is included in this SOP as Table 3. A controlled copy of this table may be posted in the metals preparation laboratory for reference by the analyst.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 At least one preparation blank for waters (PBW) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBW consists of an aliquot of reagent water that is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the PBW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the PBW must also be concentrated). Refer to the appropriate analytical SOP for PBW acceptance criteria and corrective actions.

8.2 At least one laboratory control sample for waters (LCSW) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSW consists of an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations, and is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the LCSW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the LCSW must also be concentrated). Directions for spiking the LCSW are contained in Figures 3 and 4. The measured analyte recoveries for the LCSW are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSW recovery acceptance criteria and corrective actions.

8.3 Matrix spiked samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is spiked with known amounts of all analytes of interest. Matrix spike recoveries are used to assess the effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are

contained in Figures 3 and 4. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

NOTE: Clients may choose specific samples for matrix spike and matrix spike duplicate analysis; otherwise, the choice is left to the person performing the digestion. The sample volumes available may restrict the choice of samples used for matrix spike and duplicate digestion. Field blank samples should not be chosen for matrix spike and matrix spike duplicate analysis.

8.5 The quality control measures and frequencies described above are minimum requirements. They are summarized for reference in Table 1. Individual clients and analytical programs may impose additional QC requirements.

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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TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 1

QC REQUIREMENTS

Analytical Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3010	Preparation Blank for Waters (PBW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Laboratory Control Sample for Waters (LCSW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Matrix Spike Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Matrix Spike Duplicate Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-604-03	EPA METHOD 3010, current revision
Apparatus/Materials	1) Disposable plastic specimen cup used to measure sample volume. 2) Digestion performed in 250 mL, 400 mL Griffin beaker, or 70ml digestion tube to facilitate evaporation. 3) Ribbed watch glass used throughout digestion to reduce contamination.	1) Graduated cylinder used to measure sample volume. 2) Digestion performed in 150 mL Griffin beaker. 3) Ribbed and non-ribbed watch glasses alternated in digestion.
Procedures	1) Digestate may be analyzed for antimony and silver. 2) Sample aliquots larger or smaller than 100 mL may be used. 3) Sample evaporated to 10 - 15 mL.	1) Digestate may not be analyzed for antimony and silver. 2) Requires sample aliquot of 100 mL. 3) Sample evaporated to 5 mL.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 3

PROCEDURE CONDENSATION: EPA METHOD 3010

1. If performing digestion on a hot plate, rinse glass beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with reagent water. If performing digestion with block digester, polyethylene digestion tubes do not require precleaning.
3. Label digestion vessels with sample numbers.
4. 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.
5. Add spike solutions to matrix spike samples and LCSW (refer to Figure 3 of this SOP).
6. Add 1.5 mL (per 50 mL final volume) concentrated HNO_3 to sample.
7. Cover with a ribbed watch glass.
8. Place on heating device (hotplate or block digester) and evaporate to 10 - 15 mL.
9. Cool sample and add another 1.5 mL (per 50 mL final volume) concentrated HNO_3.
10. Resume heating until gentle reflux action occurs.
11. Continue heating, adding additional HNO_3 as necessary until digestion is complete.
12. Evaporate to 10 - 15 mL.
13. Cool sample and add 5 mL (per 50 mL final volume) 1:1 HCl. Resume heating and reflux gently for 15 minutes.
14. Cool sample and filter (if necessary) or decant into a graduated polyetheyne digestion tube. Rinse beaker with reagent water and filter or decant rinsate into specimen container.
13. Dilute to appropriate final volume with reagent water.
14. Cap sample container and shake gently to mix.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 1

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK

Katahdin Analytical Services, Inc. — Metals Preparation Benchsheet

Reagent Information:
JT Baker HNO3: C20022 JT Baker HCL: C07046 Ashland H2O2: NA Method: 3010

Standards/Spiking Information:
I.V. CLPP-SPK-1 (ID/Vol): MS1362 / 0.05 mL NA : NA
CLPP-SPK-INT1 (ID/Vol): MW9975 / 0.5 mL Hot Plate No.: A
CLPP-SPK-INT2 (ID/Vol): MW9930 / 0.5 mL Temp.: 95 °C
U-Spike (ID/Vol): MS1332 / 0.05 mL
Spiking Witnessed by: N/A

REVIEWED
EC 06-27-06
KATAHDIN ANALYTICAL METALS SECTION

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LC2WWF27ICW1	WF27ICW1	0.05	L	0.05	L	AQ	IC	DJJ	06/27/2006	N/A	N/A	N/A	N/A	N/A	N/A
LCSWWF27ICW1	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006	N/A	N/A	N/A	N/A		
PBWWF27ICW1	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006	N/A	N/A	N/A	N/A		
WW3165-001	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-002	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-003	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-004	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-005	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-006	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-007	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-008	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-009	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-010	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-011	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-012	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-013	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-014	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-015	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-016	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-017	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-018	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-019	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-020	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						

DJJ 6/27/06

Digestion performed by: DJJ On: 6/27/06 Page: WF061 Revision: 00

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 2

PREPARATION OF MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 50 mL Final Volume (mL)
Laboratory Control Sample (LCSW) and Matrix Spike	CLPP-SPK-1	Inorganic Ventures	0.050
	CLPP-SPK-INT1	Lab Prepared (see below)	0.50
	CLPP-SPK-INT2	Lab Prepared (see below)	0.50
	1000 mg/L Uranium Standard	Inorganic Ventures	0.050

Sample or 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
CLPP-SPK-INT2	2007ICS-1	Inorganic Ventures	10.0
	1000 mg/L Sr	High Purity Standards	5.0
	1000 mg/L Sn	High Purity Standards	5.0
	10000 mg/L Si	High Purity Standards	5.0

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 3

ELEMENT CONCENTRATIONS IN MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Element	CONCENTRATION IN SOLUTION, mg/L								
	Matrix Spike	LCSW	CLPP-SPK-1	CLPP-SPK-4	CLPP-SPK-INT1	CLPP-SPK-INT2	QCP-CICV-3	2007 ICS-1	1000 mg/L U
Aluminum	2.000	2.000	2000						
Antimony	0.500	0.500		100	50				
Arsenic	2.000	2.000		40	200		500		
Barium	2.000	2.000	2000						
Beryllium	0.050	0.050	50						
Boron	0.500	0.500				50		500	
Cadmium	0.050	0.050		50	5		250		
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		20	50		500		
Magnesium	5.000	5.000			500				
Manganese	0.500	0.500	500						
Molybdenum	0.300	0.300				30		300	
Nickel	0.500	0.500	500						
Potassium	25.000	25.000			2500				
Selenium	2.000	2.000		10	200		500		
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			
Thallium	2.000	2.000		50	200		500		
Tin	0.500	0.500				50			
Titanium	1.000	1.000				100		1000	
Uranium	1.000	1.000							1000
Vanadium	0.500	0.500	500						
Zinc	0.500	0.500	500						

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND GFAA

Prepared By: George Brewer Date: 3/98

Approved By:

Group Supervisor: George Brewer Date: 01/24/01

Operations Manager: J. C. Burton Date: 1/24/01

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Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3050B	Format changes, added pollution prevention, added MSD, added spiking instruction tables.	DN	1.24.01	1/24/01
02 3050B	Removed all references/procedures devoted to GFAA. Added use of digestates for ICP-MS analysis. Revised standard solution names + concs. in Tables 3 + 4 to reflect current practice.	DN	8.29.02	8.29.02

TITLE: **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

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

I acknowledge receipt of copy ___ of document **SOP CA-605-02**, titled **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS.**

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STANDARD OPERATING PROCEDURE

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TITLE: **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the Katahdin Analytical Services, Inc. procedure utilized to dissolve solid matrices and solubilize metals from solid samples prior to analysis for metals by ICP-AES and ICP-MS. This SOP applies to samples prepared by EPA Method 3050, with method modifications as summarized in Table 2.

This procedure applies to all solid sample (e.g. sediments, sludges, soils, and ashes) preparations for ICP-AES and ICP-MS analyses. This method is not a total digestion technique for most samples. It is a very strong acid digestion that will dissolve almost all elements that could become "environmentally available". By design, elements bound in silicate structures are not normally dissolved by this procedure as they are not usually mobile in the environment.

1.1 Definitions

ICP-AES – Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICP-MS – Inductively Coupled Plama Mass Spectrometry.

LCSS – Laboratory Control Sample for Solids – A standard or solid reference material that has been brought through the sample preparation process.

Matrix Spike – An aliquot of a sample to which a known amount of analyte has been added before digestion.

PBS – Preparation Blank for Solids – An aliquot of reagent water that has been brought through the sample preparation process.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of solid samples by USEPA Method 3050 for metals analysis. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Training".

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of solid samples by USEPA Method 3050 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the procedure or irregularities with the samples should also be recorded in the lab notebook and reported to the responsible Supervisor or designated qualified data reviewer.

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, aprons, dust masks, and shoe protectors, is available in the Metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully, while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever beakers are being heated on a hot plate. Use caution when handling hot beakers.

Personnel are required to read the Katahdin Chemical Hygiene Plan and Safety Manual before performing this procedure, and must be familiar with the general rules for laboratory safety, personal hygiene, housekeeping, and use of protective clothing and equipment.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin 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 Katahdin Chemical Hygiene Plan and Safety Manual.

TITLE: **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

2.0 SUMMARY OF METHOD

A representative 1 to 2 g (wet weight) sample is digested with repeated additions of nitric acid and hydrogen peroxide. Hydrochloric acid is added to the initial digestate and the sample is refluxed. The digestate is then filtered and diluted to a final volume of 100 mL.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1 100 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning").

4.2 Ribbed watch glasses, 75 mm diameter, pre-cleaned as above.

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, griddle, or other heating source - adjustable and capable of maintaining a temperature of 95°C ± 5°C. Hot plates must be numbered for easy identification.

4.6 Device for measuring hot plate temperature. This may consist of a heat-resistant Erlenmeyer flask containing reagent water in which a thermometer is immersed. The temperature of each hot plate used is measured and recordedeach day. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of

5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water, again allowing each rinse to drain completely. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, 1:1 HNO_3, and concentrated HCl.

4.13 Analytical balance capable of reading to 0.01 gram.

4.14 Spatulas, scoops, or spoons; plastic or stainless steel, rinsed with 5% HNO_3 and reagent water.

5.0 REAGENTS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Nitric acid, 1:1. Add a volume of concentrated HNO_3 to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 30% hydrogen peroxide (H_2O_2) - spectrometric grade.

5.7 Multielement spiking solutions (see Table 3 for a list of required spiking solutions).

5.8 Solid reference material – a soil containing all the elements of interest, with empirically established method-specific recoveries and acceptance limits for all analytes. Solid reference materials are purchased with documentation of analysis provided by the vendor. See Figure 4 for an example certificate of analysis for a solid reference material.

TITLE: **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples should be collected in clean plastic or glass containers. Samples must be refrigerated (4°C ±2°C) upon receipt by the laboratory. The holding time for solid samples is 6 months from the date of sample collection.

7.0 PROCEDURE

The procedure described below is condensed for quick reference in Table 3.

SAMPLE PREPARATION

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet (see Figure 2 for an example). Print labels for the digestate containers, and attach them to the polyethylene sample containers that will contain the digestates.

7.2 Submerge previously cleaned beakers and watch glasses three times into a 10% nitric acid bath, then rinse three times with reagent water. Label beakers with sample numbers.

7.3 Weigh 1 to 2 g of well-mixed sample into a properly cleaned, labeled, and tared Griffin beaker. Record the weight of each sample on the printout of the digestion spreadsheet.

7.4 Weigh an appropriate amount of solid reference material to a clean, labeled, and tared Griffin beaker to serve as a laboratory control sample.

7.5 Add spike solutions to matrix spike samples (refer to Tables 3 and 4 for spiking instructions).

7.6 Add 10 mL of 1:1 HNO_3, mix the slurry, and cover the beaker with a ribbed watch glass. Place beaker on a hot plate; gently heat the sample to 95°C ± 5 °C and reflux for 10 to 15 minutes without boiling. Remove the beaker from the hot plate and cool the sample.

7.7 Add 5 mL of concentrated HNO_3 to the sample, replace the watch glass, and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL of concentrated HNO_3) until no brown fumes are given off by the sample, indicating complete reaction by HNO_3.

7.8 Continue heating the sample at 95°C ± 5°C without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the beaker from the hot plate and cool the sample.

7.9 Add 2 mL of reagent water and 3 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the hot plate to start the peroxide reaction. Continue heating until effervescence subsides.

7.10 Add an additional 7 mL of 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

7.11 Continue heating the sample at 95°C ± 5°C without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the beaker from the hot plate and cool the sample.

7.12 Add 10 mL of concentrated HCl to the digest from 7.11, replace the watch glass, and reflux at 95°C ± 5°C for 15 minutes. Remove the beaker from the hot plate and cool the sample.

7.13 Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated polystyrene specimen container or graduated polyethylene sample container with attached snap lid. Using a wash bottle, rinse the beaker with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse. Using the graduations on the specimen container or snap-lid container, dilute to 100 mL with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for ICP-AES or ICP-MS analysis.

7.14 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) reagent lot numbers, spiking information, and 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 2. 7.15 Reopen the electronic ACCESS spreadsheet for the digestion and transcribe the sample weights from the handwritten, bound copy into the electronic copy. The information in this electronic

spreadsheet will later be imported into the ACCESS metals database and used to calculate sample concentrations on a weight basis.

7.15 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. Log the digestion batch into the Internal Custody Log for Metals Digestates (see Figure 3).

CALCULATIONS

7.16 Analytical results for solid samples are reported on a dry weight basis. Total solids are determined by the Wet Chemistry Group, and are recorded in spreadsheets that are electronically imported into the Access metals database. Final dry weight concentrations are calculated by the Access database as follows:

$$\text{Concentration (mg/kg dry weight)} = (C \times V) / (W \times S)$$

where: C = Measured concentration (mg/L)
V = Digestate final volume (L)
W = Sample wet weight (kg)
S = % Solids/100

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 3050 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The 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.

8.1 At least one preparation blank for soils (PBS) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBS consists of a 1.0 g aliquot of reagent water that is digested using the same reagents as those used to digest associated samples. Refer to the appropriate analytical SOP for PBS acceptance criteria and corrective actions.

8.2 At least one laboratory control sample for soils (LCSS) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSS consists of an aliquot of a solid reference material for which the concentrations of the analytes of interest have been empirically established (solid-matrix LCSS), or an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations (aqueous-matrix LCSS). The solid reference material should normally be used as the LCSS, unless a particular client or analytical program requires that spiked reagent water be used. The LCSS is digested using the same reagents as those used to digest associated samples. Directions for spiking the aqueous-matrix LCSS are contained in Figure 3. The measured analyte recoveries for the LCSS are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSS recovery acceptance criteria and corrective actions.

8.3 Matrix spike samples are processed along with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is fortified with known amounts of all analytes of interest prior to digestion. Matrix spike recoveries are used to assess the biasing effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figure 2. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

NOTE: Clients may choose specific samples for matrix spike and duplicate analysis; otherwise, the choice is left to the person performing the digestion.

8.5 The quality control measures and frequencies described above are minimum requirements. Individual clients and analytical programs may impose additional QC requirements.

9.0 METHOD PERFORMANCE

Refer to the applicable instrumental analysis SOP for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste," United States Environmental Protection Agency, SW-846, Third Edition, Final Update III, 12/96, Method 3050B.

TITLE: **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

LIST OF TABLES AND FIGURES

Table 1 QC Requirements – Method 3050
Table 2 Summary of Method Modifications – Method 3050
Table 3 Preparation of Matrix Spikes and Spiking Solutions
Table 4 Element Concentrations in ICP-AES Matrix Spikes and Their Component Spiking Solutions
Figure 1 Procedure Condensation – Method 3050
Figure 2 Example Page from Metals Sample Preparation Logbook
Figure 3 Example Page from Internal Custody Log for Metals Digestates
Figure 4 Example Certificate of Analysis for Solid Reference Material

TITLE: **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

TABLE 1

QC REQUIREMENTS – METHOD 3050

Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3050	Preparation Blank for Solids (PBS)	One per prep batch of 20 or fewer samples.	Refer to analytical method.	Refer to analytical method.
	Laboratory Control Sample for Solids (LCSS)	One per prep batch of 20 or fewer samples.	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Duplicate Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Demonstration of analyst proficiency	One-time demonstration by each analyst performing the method.	Must pass all applicable QC for method.	Repeat analysis until able to perform passing QC; document successful performance in personal training file.

TITLE: **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS – METHOD 3050

TOPIC	KATAHDIN SOP CA-605-02	METHOD 3050, current revision
Apparatus /Materials	1) Digestion performed in 100 mL Griffin beaker. 2) Graduated disposable plastic cup used to bring digestate to final volume.	1) Digestion performed in 250 mL Griffin beaker. 2) Volumetric flask used to bring digestate to final volume.
Procedure	1) Digestate volume reduced to 5 to 10 mL prior to filtering. 2) Samples are not cooled after the initial addition of H_2O_2. 3) After filtration, the filters are rinsed three times with reagent water.	1) Digestate volume reduced to 5 mL prior to filtering. 2) Samples are cooled after the initial addition of H_2O_2. 3) After filtration, the filters are rinsed twice with reagent water.

TITLE: **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

TABLE 3

PREPARATION OF MATRIX SPIKES AND SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Matrix Spike for ICP-AES	CLPP-SPK-1	Inorganic Ventures	0.10
	CLPP-SPK-INT1	Lab Prepared (see below)	1.00
	CLPP-SPK-INT2	Lab Prepared (see below)	1.00

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	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
CLPP-SPK-INT2	2007ICS-1	Inorganic Ventures	10.0
	1000 mg/L Sr	High Purity Standards	5.0
	1000 mg/L Sn	High Purity Standards	5.0
	10000 mg/L Si	High Purity Standards	5.0

TITLE: **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

TABLE 4

ELEMENT CONCENTRATIONS IN ICP-AES MATRIX SPIKES AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY METHOD 3050

Element	CONCENTRATION IN SOLUTION, mg/L						
	Matrix Spike (ICP-AES)	CLPP-SPK-1	CLPP-SPK-4	CLPP-SPK-INT1	CLPP-SPK-INT2	QCP-CICV-3 SPK-3	2007ICS-1
Aluminum	2.000	2000					
Antimony	0.500		100	50			
Arsenic	2.000		40	200		500	
Barium	2.000	2000					
Beryllium	0.050	50					
Boron	0.500				50		500
Cadmium	0.050		50	5		250	
Calcium	2.500			250			
Chromium	0.200	200					
Cobalt	0.500	500					
Copper	0.250	250					
Iron	1.000	1000					
Lead	0.500		20	50		500	
Magnesium	5.000			500			
Manganese	0.500	500					
Molybdenum	0.300				30		300
Nickel	0.500	500					
Potassium	25.000			2500			
Selenium	2.000		10	200		500	
Silicon	5.230				523		230
Silver	0.050	50					
Sodium	7.500			750			
Strontium	0.500				50		
Thallium	2.000		50	200		500	
Tin	0.500				50		
Titanium	1.000				100		1000
Vanadium	0.500	500					
Zinc	0.500	500					

TITLE: **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

FIGURE 1

PROCEDURE CONDENSATION – METHOD 3050

1. Prepare and print out ACCESS spreadsheet. Label plastic sample containers.
2. Rinse beakers and watch glasses 3 times in 10% HNO_3 bath.
3. Rinse beakers and watch glasses 3 times with reagent water.
4. Label beakers with sample numbers.
5. Weigh 1 to 2 g of well-mixed sample into tared beaker. Record sample weights.
6. Add spike solutions to matrix spike samples.
7. Add 10 mL 1:1 HNO_3 to samples and cover with watch glasses.
8. Reflux for 10 to 15 minutes at $95^{\circ} \pm 5^{\circ}$ C. without boiling. Cool samples.
9. Add 5 mL conc. HNO_3, cover beakers, and reflux for 30 minutes.
10. Repeat Step 9 as necessary until digestion is complete.
11. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
12. Cool sample and add 3 mL 30% H_2O_2 and 2 mL reagent water. Heat gently until effervescence subsides.
14. Cool samples and add 7 mL of 30% H_2O_2 in 1 mL aliquots. Heat gently until effervescence subsides.
15. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
16. Add 10 mL conc. HCl and reflux for 10 to 15 minutes at $95^{\circ} \pm 5^{\circ}$ C.
17. Cool sample and filter into graduated specimen container. Bring to volume with reagent water and transfer to labeled polyethylene bottle.
18. Enter sample weights into ACCESS spreadsheet.

TITLE: **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

FIGURE 2

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK

Katahdin Analytical Services, Inc.

Metals Preparation Benchsheet

Reagent Information:
JT Baker HNO3: T324060 JT Baker HCL: T31035 Ashland H2O2: 004A008E

Method: 3050H
LCSS: E.R.A. Priority Pollutant/CLP Lot: S-824 LOT # 243

Standards Information:
I.V. CLPP-SPK-1: S895 0.1 ml
CLPP-SPK-INT1: MW5476 1.0
CLPP-SPK-INT2: MW5463 1.0
NA : NA
NA : NA
Hot Plate No.: 6
Temp.: 99 °C

REVIEWED
Ekm 12-9-00
KATAHDIN ANALYTICAL METALS SECTION

Spiking Witnessed by: WITNESS NA

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Texture	Final Color	Final Clarity	Artifacts	Bottle
LCSSQL08ICS0	QL08ICS0	0.50	g	0.1	L	SL	IC	DCP	12/08/2000	N/A	N/A	N/A	N/A	NA	NA
PBSQL08ICS0	QL08ICS0	1.00	g		L	SL	IC	DCP	12/08/2000	N/A	N/A	N/A	N/A		
WQ3697-001	QL08ICS0	0.52	g		L	FP	IC	DCP	12/08/2000	NA	NA	NA	NA		
WQ3697-002	QL08ICS0	0.59	g		L	FP	IC	DCP	12/08/2000						
WQ3705-011	QL08ICS0	1.13	g		L	SL	IC	DCP	12/08/2000						
WQ3705-021	QL08ICS0	1.03	g		L	SL	IC	DCP	12/08/2000						
WQ3705-022	QL08ICS0	1.00	g		L	SL	IC	DCP	12/08/2000						
WQ3719-003	QL08ICS0	1.17	g		L	SL	IC	DCP	12/08/2000						
WQ3719-003P	QL08ICS0	1.20	g		L	SL	IC	DCP	12/08/2000						
WQ3719-003S	QL08ICS0	1.19	g		L	SL	IC	DCP	12/08/2000						
WQ3720-001	QL08ICS0	0.56	g		L	FP	IC	DCP	12/08/2000						
WQ3728-001	QL08ICS0	1.02	g		L	SL	IC	DCP	12/08/2000						
WQ3729-001	QL08ICS0	1.30	g		L	SL	IC	DCP	12/08/2000						

DCP 12/8/00

Digestion performed by: DCP On: 12/8/00

Page: QL027 Revision: 00

TITLE: **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

FIGURE 3

EXAMPLE PAGE FROM INTERNAL CUSTODY LOG FOR METALS DIGESTATES

Katahdin Analytical Services, Inc.
Internal Custody Record for Metals Digestates

	Removed By			Returned By			
	Initials	Date	Time	Initials	Date	Time	Comments
QC BATCH ID: QL19ICWD	[illegible]	12-20-00	1170	EAm	1-2-01	8:20	
Logged In By: DCP							
Log-In Date: 12/19/00							
Log-In Time: 1800							
QC BATCH ID: QL19ICSO	[illegible]	12-20-00	1140	EAm	1-2-01	8:20	
Logged In By: DCP							
Log-In Date: 12/19/00							
Log-In Time: 1800							
QC BATCH ID: QL20ICWD	[illegible]	12-21-00	1130	EAm	1-2-01	8:20	
	EAm	1-5-01	8:25	EAm	1-22-01	13:15	
Logged In By: DCP							
Log-In Date: 12/20/00	EAm 12-20-00						
Log-In Time: 1700							
QC BATCH ID: QL20ICSO	[illegible]	12-21-00	1130	EAm	1-2-01	8:20	
Logged In By: DCP							
Log-In Date: 12/20/00							
Log-In Time: 1700							

DISK: METALSFORMS FILE CUSTODY.WK1

TITLE: **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

FIGURE 4

EXAMPLE CERTIFICATE OF ANALYSIS FOR SOLID REFERENCE MATERIAL

ENVIRONMENTAL RESOURCE ASSOCIATES
ARVADA, COLORADO 1-800-372-0122

Recd 12/08/06

Certification

PriorityPollutnT™/CLP Inorganic Soils - Hot Plate Digestions Quality Control Standards

Catalog No PPS-46

Lot No. 245

Parameter	Total Concentration[1]	Method 3050 ICP-OES/FLAA: Certified Value[2]	Method 3050 ICP-OES/FLAA: Performance Acceptance Limits™ [3]	Method 3050 ICP-MS/GFAA: Certified Value[2]	Method 3050 ICP-MS/GFAA: Performance Acceptance Limits™ [3]
TRACE METALS PriorityPollutnT™ (Catalog No 540)	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg
Aluminum	53800*	8830	3780 - 13900	9100	5980 - 12200
Antimony	198	68.9	18.8 - 119	8.10	D.L. - 27.7
Arsenic	152	136	101 - 171	136	101 - 171
Barium	894*	124	95.3 - 152	132	102 - 163
Beryllium	103	95.3	74.7 - 116	96.1	75.0 - 117
Boron	158	115	79.2 - 151	119	69.0 - 169
Cadmium	131	118	90.4 - 145	121	93.0 - 149
Calcium	16100*	11500	8590 - 14400	11600	8670 - 14500
Chromium	135	89.3	71.3 - 107	89.7	71.6 - 108
Cobalt	125	110	87.2 - 132	121	93.2 - 148
Copper	136	117	95.7 - 138	118	90.4 - 146
Iron	26400*	13700	8340 - 19100	11500	7000 - 16000
Lead	172	138	105 - 170	144	110 - 179
Magnesium	5520*	3040	2150 - 3930	3000	2420 - 3590
Manganese	520*	341	272 - 409	319	258 - 381
Mercury	2.60	2.48	1.69 - 3.27	2.48	1.69 - 3.27
Molybdenum	111	94.1	72.0 - 116	82.6	63.2 - 102
Nickel	186	156	122 - 190	161	117 - 204
Potassium	31000*	3430	2670 - 4190	3290	2570 - 4010
Selenium	105	87.6	64.9 - 110	95.0	70.4 - 120
Silver	132	119	88.8 - 150	87.1	47.3 - 127
Sodium	12100*	853	578 - 1130	809	549 - 1070
Strontium	294*	83.4	60.5 - 106	80.4	58.4 - 103
Thallium	154	139	79.6 - 199	144	82.4 - 205
Tin	117	96.3	72.1 - 121	29.8	13.3 - 46.3
Titanium	2900*	276	86.2 - 465	160	103 - 218
Vanadium	131	79.1	54.0 - 104	71.3	48.7 - 93.9
Zinc	100	66.0	42.9 - 89.1	72.1	49.2 - 95.0

CONTINUED ON BACK

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

Prepared By: George Brewer Date: 7/98

Approved By:

Group Supervisor: George Brewer Date: 01/23/01

Operations Manager: John C. Burton Date: 1/23/01

QA Officer: Deborah J. Nadeau Date: 1.23.01

General Manager: Duncan F. Hufgath Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 6010B	Format changes, added pollution prevention, expanded procedure and QC sections. Added tables.	DN	1.23.01	1/23/01
02 6010B	Calibration begins with analysis of S0 (cal. blank) followed by S1 (Mixed Cal. Std.) Changes to section 7.5 and Table 8 to reflect this. Made changes to element concs. in Tables 3,4,5,6 to reflect current practices.	DN	10.21.02	10.21.02
03 6010B	Added MN-IEC to standards run. Changed frequency of LRS. Changed concentration of HNO_3 in calibration blank. CRI changed from three separate solutions to one. Changed CRI vendor.	MRC	04.15.04	04.15.04
04	Updated ICV, CCV, ICB, PQL Chk std. PBW, PBS, MS & MSD acceptance criteria updated Table 1	LAD	05/06	05/06
05	Updated Tables 3,4,5,6 and 7 with current standard concentrations and prep. Updated Table 1 with current practices including NAVY audit findings. Updated sections 2, 7.2, 7.6 and Table 1 with new ICP information. Updated Table 8 with current sequence requirements.	LAD	07/07	07/07

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added hardness definition and calculation (APP. 1)	LAD	09/07	09/07

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-608-06**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-608-06**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

Recipient: _____ Date: _____

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma atomic-emission spectroscopy (ICP-AES) determines trace elements, including metals, in solution. The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services, Inc. personnel to analyze aqueous and solid samples for trace metals by USEPA Method 6010 (Test Methods for Evaluating Solid Waste, Physical/ Chemical Methods, USEPA SW846).

Sample types that may be analyzed using these methods include drinking waters, ground waters, aqueous samples, TCLP, SPLP and EP Toxicity extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes. The following elements may be analyzed under this SOP: Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Si, Ag, Na, Sn, Sr, Tl, Ti, V, and Zn.

All samples, except filtered ground water samples, analyzed under USEPA Method 6010 require digestion prior to analysis. USEPA Methods 3005, 3010, and 3050 describe appropriate digestion procedures for samples to be analyzed by ICP-AES under EPA Method 6010. Refer to current revisions of Katahdin SOPs CA-604 and CA-605, current revisions, for sample digestion procedures.

1.1 Definitions

Analytical Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

CRI - Contract Required detection limit sample for ICP - A low concentration standard used to verify calibration accuracy near the low end of the calibration range.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-AES - Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICS - Interference Check Sample - Two standards (ICSA and ICSAB) used to verify the effectiveness of interelement correction and background correction. Solution ICSA contains only interferents (Al, Ca, Fe, and Mg) at high concentrations (200 to

500 mg/L); solution ICSAB contains interferents at the same concentrations as well as analytes at low (20 mg/L or less) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 99% confidence.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LRS - Linear Range Standard - A high-concentration standard used to determine the upper reporting limit of the ICP calibration.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

Hardness – The sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in mg/L.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP analysis by EPA Method 6010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in ICP analysis by Method 6010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the group supervisor or designated qualified data reviewer responsible for this data.

It is the responsibility of the Group Supervisor to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and 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.

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 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

This method describes multielemental determinations by ICP-AES using simultaneous optical systems and radial or 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 spectrometer and the intensities of the emitted lines are monitored by a charge injection device (CID). Signals from the CID are measured simultaneously by a computer system. Element concentrations of unknown samples are quantitated by comparison of sample emission intensities to emission intensities of standards of known concentration. A background correction technique is used to compensate for variable background contribution to the determination of trace elements. Background is measured adjacent to the analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, has been determined by the complexity of the spectrum adjacent to the analytical line. The position used must be relatively free of spectral interference and must reflect the same change in background intensity as occurs at the analyte wavelength. Physical interferences are corrected through the use of an internal standard (yttrium) that is automatically added to all samples and standards prior to nebulization. The possibility of additional interferences (noted in Section 3) must be recognized and appropriate corrections applied.

3.0 INTERFERENCES

Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as spectral interferences, physical interferences, and chemical interferences.

Spectral interferences can be categorized as 1) overlap of a spectral line from another element; 2) unresolved overlap of molecular band spectra; 3) background contribution from continuous or recombination phenomena; and 4) background from stray light from the line emission of high concentration elements. The first of these effects is compensated by utilizing the computer correction of raw data, requiring the monitoring and measurement of the interfering element (interelement correction). The second effect is controlled by choosing analytical wavelengths that are free from overlapping molecular emission spectra. The third and fourth effects are usually compensated by a background correction adjacent to the analyte line. Uncorrected spectral interferences may be detected through examination of serial dilution and matrix spike data.

Physical interferences are generally considered to be effects associated with sample nebulization and transport processes. Such properties as changes in viscosity and surface tension can cause significant inaccuracies, especially in samples that may contain high dissolved solids and/or acid concentrations. Matrix matching of standards and samples and the use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem that can occur from high

dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow rate causing instrumental drift. Regular cleaning of nebulizer tips and dilution of samples with high dissolved solids contents are used to control this problem. Physical interferences are also corrected by this laboratory through the use of an internal standard. Uncorrected physical interferences may be detected through examination of serial dilution and matrix spike data. Instrument drift caused by the salting up of nebulizer tips may also be detected by looking for oriented drift in calibration verification standards analyzed regularly throughout the run.

Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not pronounced with the ICP technique; however, if observed they can be minimized by careful selection of operating conditions (i.e., incident power, observation position, etc.), by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element. Uncorrected chemical interferences may be detected through examination of serial dilution data.

4.0 APPARATUS AND MATERIALS

4.1 Computer-controlled inductively-coupled plasma atomic emission spectrometer (plasma viewed radially or axially) equipped for internal standardization, and capable of performing automatic background correction and interelement correction.

4.2 Computer-controlled autosampler.

4.3 Argon gas supply – high purity.

4.4 Volumetric glassware of suitable precision and accuracy.

4.5 Automatic pipets of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

Refer to the appropriate instrument-specific SOP for additional required equipment.

5.0 REAGENTS

5.1 Hydrochloric acid, concentrated (HCl) – spectroscopic grade.

5.2 Nitric acid, concentrated (HNO_3) – spectroscopic grade.

5.3 Reagent water, trace metals free.

5.4 Calibration blank – reagent water containing HCl (5% v/v) and HNO_3 (5% v/v). Calibration blank solution is prepared in large volumes (up to 20 liters) and stored in a carboy. Calibration blank solution is used in establishing the analytical curve, and in all initial and continuing calibration blank determinations. This solution is also used to flush the system between standards and samples. Intermediate and working standards are prepared by diluting stock standards and intermediate standards with calibration blank solution so that all standards and blanks are acid matrix-matched to sample digestates.

5.5 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals, and supplied by the vendors with certificates of purity and analysis. Refer to Tables 3 and 4 for a listing of stock standards required, and to Table 7 for element concentrations in stock standards.

5.6 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 4 for a listing of intermediate standards required and for preparation instructions. Refer to Table 6 for element concentrations in intermediate standards.

5.7 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument and to perform all necessary QC checks. Refer to Table 3 for a listing of working standards and for preparation instructions. Refer to Table 5 for element concentrations in working standards.

5.8 5 mg/L yttrium internal standard solution – add 0.5 mL 10000 mg/L yttrium stock standard to a 1000 mL volumetric flask half filled with calibration blank solution. Bring to volume with calibration blank solution.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP should be collected and preserved as described in the following table.

Matrix	Container [1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months

[1] P = polyethylene or , G = glass

7.0 PROCEDURES

7.1 Background correction positions, dynamic linear ranges, and interelement correction factors must be established for each analytical wavelength used on each instrument prior to beginning sample analysis. Refer to Section 8 of this SOP and to the appropriate instrument operation SOP (current revisions of Katahdin SOPs CA-609 or CA-612).

7.2 Ignite the plasma and allow the instrument to aspirate calibration blank solution (refer to the appropriate instrument operation SOP). Wait 15 minutes for the instrument to become thermally stable before proceeding with analysis.

7.3 Profile the instrument as described in the appropriate instrument operation SOP.

7.4 Sample analysis will be performed through the use of an autosampler. Using the instructions given in the appropriate instrument operation SOP, prepare an autosampler table listing all standards and samples to be analyzed, in the appropriate order. Print out the autosampler table and fill the autosampler racks with tubes containing samples and standards in the positions listed in the autosampler table.

7.5 Initiate sample analysis using the autosampler, as described in the appropriate instrument operation SOP. 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 a calibration blank solution (Blank) followed by the analysis of a multielement calibration standard (S1 in Table 3) to calibrate the instrument. The system is flushed with calibration blank for one minute between each sample and standard, and each sample and standard is aspirated for one minute prior to the beginning of emission measurements.

7.6 Interelement correction standards, which are used to update the interelement correction (IEC) factors, are analyzed daily. The solution "AL_IEC" contains 500 mg/L aluminum, and the IEC factors for aluminum should be updated after analysis of this solution. The solution "FE_IEC" contains 200 mg/L iron, and the IEC factors for iron should be updated after analysis of this solution. IEC factors for other elements are updated as necessary, at least every six months.

7.7 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.

7.8 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.

7.9 Interference check standard solutions ICSA and ICSAB must be analyzed at the beginning and end of each run to verify the accuracy of the IEC factors. Refer to Section 8 and Table 1 for additional information.

7.10 A practical quantitation limit standard (PQL) must be analyzed at the beginning and end of each run to determine the accuracy of the calibration at the reporting limit. Refer to Section 8 and Table 1 for additional information.

7.11 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a QC sample (ICV, ICB, CCV, CCB, ICSA, or ICSAB) for that element must not be reported. The sample must be reanalyzed for the element in question.

7.12 All samples that exceed the linear calibration 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 calibration range and the lead result must be reported from that dilution analysis.

7.13 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.

7.14 All analyses are performed using yttrium as an internal standard to compensate for enhancement or depression of the analytical signal due to matrix effects. Yttrium solution is pumped at a constant rate through one channel of the peristaltic pump. Samples and standards are pumped through a second channel of the pump. The tubing carrying the internal standard is connected to the tubing carrying samples and standards downstream from the pump, and mixing of the two streams is accomplished in a mixing coil downstream from the connection, prior to nebulization.

For each sample or standard, the computer that controls the spectrometer divides the detected emission signal for each element by the detected yttrium emission signal prior to quantitation, thus normalizing all emission signals to that of yttrium.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6010 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

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

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of a reagent water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of a reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the seven replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 The upper limit of the linear dynamic range (LDR) must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing succeedingly higher standard concentrations of the analyte until the observed analyte concentration is no more than 10% below 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 90% of 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.

8.4 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 0.995 or better. Third order fits are not acceptable. Non-linear response curves must be revalidated and recalculated every six months.

ANALYTICAL RUN QC SAMPLES

8.5 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, with relative standard deviation <5% from replicate integrations. If the ICV fails, result for the failing elements may not be reported from the run unless the ICV recovery is greater than 110%and the sample result is less than the PQL.

8.6 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. Also, for failing elements, all samples analyzed after the last passing CCV must be reanalyzed.

8.7 Calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs

must be less than the Practical Quantitation Level (PQL) for each element. If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for a CCB a 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.

8.8 Interference check solutions ICSA and ICSAB (refer to Section 1.1) are analyzed at the beginning of each run to verify interelement correction factors and background correction. ICSA contains interferent elements (Al, Ca, Fe, and Mg) only, at concentrations of 200 mg/L to 500 mg/L. Results for interfering elements in the ICSA must fall within 80% to 120% of the expected values. Results for unspiked elements in ICSA must fall within ± PQL if the PQL is greater than 0.01 mg/L, within ± 2xPQL if the PQL is less than or equal to 0.01 mg/L. 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.

8.9 A Practical Quantitation Limit (PQL) Check Standard is analyzed at the beginning of each run, after the ICV and ICB samples. Element concentrations in this solution are at the laboratories practical quantitation limit. Element recoveries for the PQL check Standard must fall between 80-120% 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 120% and the samples results are less than the PQL.

PREPARATION BATCH QC SAMPLES

8.10 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.

8.11 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. 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, associated sample results that are less than the PQL or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.12 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 witht he following exception. If the LCS fails high, sampleresults less than the PQL may bereported.

SAMPLE MATRIX QC SAMPLES

8.13 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.

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.

8.12 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL), 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.

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 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

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846 Updates I, II, IIA, and IIB, Revised December 1996., Method 6010B.

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TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010	Initial Calibration, minimum 1 point plus a calibration blank.	Daily prior to sample analysis.		
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value, and RSD < 5%.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL.	1) Do not use results if ≥ PQL and < 10x CCB level. 2) Investigate and correct problem.
	Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within ± 10% of true value, and RSD < 5%.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Continuing Calibration Blank (CCB)	After every 10 samples and at end of the run.	Absolute value of CCB < PQL.	1) Do not use results if ≥ PQL and < 10x CCB level. 2) Investigate and correct problem.
	Practical Quantitation Level Check Standard (PQL)	At beginning and end of run.	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.
	Interference Check Solution A (ICSA)	At beginning and end of run.	For Al, Ca, Fe, and Mg, recovery within ± 20% of true value. For analytes not spiked, ± PQL, or, if PQL ≤ 0.01 mg/L, ± 2x PQL.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Interference Check Solution AB (ICSAB)	At beginning and end of run.	Recovery of each analyte within ± 20% of true value.	1) Do not use results for failing elements. 2) Investigate and correct problem.

TABLE 1, CONTINUED

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010, continued	Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and <10x the blank concentration.
	Laboratory Control Sample (LCSW/LCSS)	One per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added.	Flag results.
	Matrix Spike Duplicate Sample (P)	One per digestion batch of 20 or fewer samples.	1) Recovery ± 25% of true value, if sample < 4x spike added. 2) RPD ≤20% for duplicate spikes.	Flag results
	Serial Dilution (L)	One per digestion batch.	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag result or dilute and reanalyzed sample to eliminate interference.
	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	Annually.	M PQL > 2-3 * the MDL	1) Repeat MDL study. 2) Raise PQL.
	Linear Range Study	Every six months	Run succeedingly higher stds until recovery not within ± 10%. Use highest passing concentration as upper limit of linear range.	Only accept data to highest passing concentration until next linear range study.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-608-06	METHOD 6010, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6010: ± PQL	Acceptance criteria stated in 6010: mean blank value ± 3 standard deviations

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 Internal Std.	Lab Prepared (see Table 4)	10
	QCS-26	High Purity Standards	1
Initial Calibration Verification (ICV)	IV-28	Inorganic Ventures	0.4
	CL-CAL-3	SPEX	0.96
	10000 mg/L K	Inorganic Ventures	0.98
	1000 mg/L Al	IV	0.96
	1000 mg/L Sn	Inorganic Ventures	0.05
Practical Quantitation Limit Sample (PQL)	PQL-INT	Lab Prepared (see Table 4)	1
AL_IEC	10000 mg/L Al	Inorganic Ventures	5
FE_IEC	10000 mg/L Fe	Inorganic Ventures	2
MN_IEC	1000 mg/L Mn	High-Purity Standards	2
Interference Check Solution A (ICSA)	CLPP-ICS-A	Inorganic Ventures	10
Interference Check Solution AB (ICSAB)	CLPP-ICS-A	Inorganic Ventures	10
	CLPP-ICS-B4	Inorganic Ventures	1
	ICSAB-INT	Lab Prepared (see Table 4)	5
Continuing Calibration Verification (CCV)	ICP intermediate std.	Lab Prepared (see Table 4)	5
	QCS-26	High Purity Standards	0.5

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)
ICP Intermediate Std.	10000 mg/L Si	High Purity Standards	2.5 each
	10000 mg/L Al, Ca, Fe	High Purity Standards	2.4 each
	10000 mg/L mg, Na	High Purity Standards	2.4each
	10000 mg/L K	High Purity Standards	1.5 each
	1000 mg/L Sn, Sr	High Purity Standards	1.0 each
PQL-INT	1000 mg/L B,Mo,Sn,Sr	High Purity or Inorganic Ventures	1.0 each
	10000 mg/L K	High Purity or Inorganic Ventures	1.0
	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
	10000 mg/L Si	High Purity Standards	0.2
	1000 mg/L Cr,Ti,Tl,Ag	High Purity Standards	0.15 each
	1000 mg/L Cd,Se	High Purity Standards	0.1 each
	10000 mg/L Al	High Purity Standards	0.3
	10000 mg/L Na	High Purity or Inorganic Ventures	1.0
	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	High Purity or Inorganic Ventures	4.0 each
	10000 mg/L B,Mo,Sr,Sn,Ti	High Purity Standards	1.0 each
	10000 mg/L Si	High Purity Standards	0.4

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	AL_IEC	FE_IEC	ICSA	ICSAB	CCV
Aluminum	25	10	0.3	500		500	500	12.5
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	200	12.5
Lead	1	0.4	0.005				0.05	0.5
Magnesium	25	10	0.05			500	500	12.5
Manganese	1	0.4	0.005				0.5	0.5
Molybdenum	1	0.4	0.1				0.5	0.5
Nickel	1	0.4	0.04				1.0	0.5
Potassium	25	13.6	1.0				20	12.5
Selenium	1	0.4	0.01				0.05	0.5
Silicon	25	10	0.2				10	12.5
Silver	1	0.4	0.015				0.2	0.5
Sodium	25	10	1.0				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
Vanadium	1	0.4	0.025				0.5	0.5
Zinc	1	0.4	0.025				1.0	0.5

TABLE 6

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L		
	ICP-INT	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	
Magnesium	240	5.0	
Manganese		0.5	
Molybdenum		10	10
Nickel		4.0	
Potassium	150	100	400
Selenium		1.0	
Silicon	250	20	40
Silver		1.5	
Sodium	240	100	40
Strontium	10	10	10
Thallium		1.5	
Tin	10	10	10
Titanium		1.5	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	
	CLPP-ICS-A	CLPP-ICS-B4
Aluminum	5000	
Antimony		60
Arsenic		10
Barium		50
Beryllium		50
Boron		
Cadmium		100
Calcium	5000	
Chromium		50
Cobalt		50
Copper		50
Iron	2000	
Lead		5
Magnesium	5000	
Manganese		50
Molybdenum		
Nickel		100
Potassium		
Selenium		5
Silicon		
Silver		20
Sodium		
Strontium		
Thallium		10
Tin		
Titanium		
Vanadium		50
Zinc		100

TABLE 8

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Blank (Calibration Blank)	Initial calibration
2	S1 (Calibration Standard)	Initial calibration
3	ICV (Initial Calibration Verification)	Check calibration accuracy
4	ICB (Initial Calibration Blank)	Check calibration accuracy
5	PQL (Practical Quantitation Level Sample)	Check calibration accuracy near PQL, repeat before final CCV, CCB
6	ICSA (Interference Check Solution A)	Verify accuracy of IEC factors, repeat before final CCV, CCB
7	ICSAB (Interference Check Solution AB)	Verify accuracy of IEC factors, repeat before final CCV, CCB
8	CCV (Continuing Calibration Verification)	Check calibration stability
9	CCB (Continuing Calibration Blank)	Check calibration stability
10-19	Analyze up to 10 samples	
20	CCV (Continuing Calibration Verification)	Check calibration stability
25	CCB (Continuing Calibration Blank)	Check calibration stability
...	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	

ATTACHMENT 1

HARDNESS BY CALCULATION

As referenced in "Standard Methods for the Examination if Water and Wastewater," Methods 2340 A & B, Hardness Introduction and Hardness by Calculation, American Public Health Association, 18th Edition, Revised 1992, total hardness is the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter.

Once the calcium and magnesium concentrations have been determined by EPA methods 6010, 6020, 200.7 or 200.8, the total hardness of an aqueous sample may be calculated as follows:

$$\text{Total Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L) + 4.118\ (Mg, mg/L)$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L)$$

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-611
Revision History
Cover Page
Page 1

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Prepared By: George Brewer Date: 12/97

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: John C. Burton Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1.29.01

General Manager: Dennis F. Hufnah Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
02 7471A	Format changes, added pollution prevention, other minor changes to sections 7, 8 and QA Table.	DN	1.29.01	1/29/01
03 7471A	Changed Leeman PS200 Automated Mercury Analyzer to Cetac M6100 Mercury analyzer. Revised Sect. 10 to show correct reference material. Removed fig. 2 Revised sect. 4.8, 5.7 and 8.9 to reflect current practises. minor changes through out	LAD	021605	021605

TITLE: **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

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

I acknowledge receipt of copy ___ of document **SOP CA-611-03,** titled **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471.**

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STANDARD OPERATING PROCEDURE

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TITLE: **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services, Inc. personnel for the digestion and analysis solid samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in soils, sediments, bottom deposits, and sludges under USEPA Method 7471 (Test Method for Evaluating Solid Wastes, USEPA SW 846, Third Edition).

1.1 Definitions

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory reagent grade water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory reagent grade water used to verify calibration accuracy.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

PB - Preparation Blank - Laboratory reagent grade water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7471. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7471 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Management Plan and Safety Manual 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 there waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in cold vapor atomic absorption may contain high concentrations of acids, mercury, and other toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested mercury samples and standards and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled "Waste Stream A") that is located in the Metals Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's 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.

3.0 INTERFERENCES

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low bias for analytical results. Certain volatile organic materials will also non-specifically absorb

radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Samples that are high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine, which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

4.0 APPARATUS AND MATERIALS

4.1 250 mL Pyrex media bottles with plastic screw caps, for use as digestion vessels.

4.2 Water bath capable of maintaining a constant temperature of 95° C.

4.3 Analytical balance capable of weighing to 0.01 g.

4.4 Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents.

4.6 Spirit-filled thermometer, NIST-traceable, covering the range from 20° to 110° C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.

4.7 Disposable graduated polystyrene sample cups, 200 mL capacity.

4.8 CETAC M6100 Mercury Analyzer and associated peripherals and parts.

Refer to Katahdin SOP CA-629, current revision, "Operation and Maintenance of the CETAC M6100 Mercury Analyzer" for additional required materials.

TITLE: **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

5.0 REAGENTS

5.1 Laboratory reagent grade water – mercury-free water which has been treated by the Culligan Water System and the Millipore Mill-QÔ Water System and meets the specifications of ASTM Type II water

5.2 Concentrated nitric acid (HNO_3), trace metal grade

5.3 Concentrated hydrochloric acid (HCl), trace metal grade

5.4 Aqua regia: Prepare an appropriate amount immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3 in a heat-proof beaker or flask. Preparation of aqua regia must be performed in a fume hood.

5.5 Potassium permanganate solution, 5% w/v: Dissolve 50 g of potassium permanganate in 1 L laboratory reagent grade water. The source reagent should be labeled as suitable for use in mercury determination.

5.6 Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in laboratory reagent grade water and dilute to a final volume of 1 L.

5.7 Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory reagent grade water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. Reagent should be labeled as suitable for use in mercury determination.

5.8 Mercury Stock Standards: Two 10.0 mg/L mercury stock standards, obtained from separate sources, are required. The mercury concentrations of these standards must be certified by the manufacturers as traceable to NIST reference standards.

5.9 Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. This intermediate standard is used to prepare calibration standards, matrix spikes, CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard and instructions for its dilution may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard A must be prepared fresh daily, and disposed of appropriately after use. (Note: the concentrations of all stock standards must be certified by the vendors as traceable to NIST reference materials).

5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. The source of the stock standard used to prepare Intermediate Mercury Standard B must be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8.0). The identity of the stock standard currently used to prepare this intermediate standard and instructions for its dilution may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard B must be prepared fresh daily, and disposed of appropriately after use.

5.11 Solid Reference Material: A soil with a known or empirically-established mercury concentration for use in preparing the laboratory control sample for soils. Solid reference materials should be purchased with certificates listing reference values and quality control acceptance limits. See Figure 3 for an example certificate of analysis for a solid reference material.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Soil samples to be analyzed for mercury should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Solid	P, G	40 g	Cool to 4°C ± 2°	28 days

[1] P = polyethylene, G = glass

7.0 PROCEDURES

BOTTLE PREPARATION

7.1 Mercury digestion bottles are reused, and must be cleaned between uses. After the previous contents of the bottles have been discarded, bottles are segregated according to whether the measured mercury concentrations of the previous contents were above the PQL (contaminated bottles) or below the PQL (uncontaminated bottles). Labels are removed from the bottles by wiping with a paper towel saturated with toluene. Both contaminated and uncontaminated bottles

are then cleaned with Liquinox and water, if necessary, to remove visible grime, and rinsed thoroughly with tap water.

7.2 Uncontaminated bottles are then triple-rinsed with laboratory reagent grade water, and are ready for reuse.

7.3 Contaminated bottles are placed in a bath containing 10% HCl for at least 12 hours. After acid-leaching, these bottles are triple rinsed with laboratory reagent grade water, and are then ready for reuse.

PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

7.4 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer database and print out a copy of the benchsheet. All necessary details of sample preparation (standards preparation information, digestion times, initial weights and final volumes, pertinent observations, etc.) must be recorded on this benchsheet, which will be bound in the Mercury Preparation Logbook. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.

7.5 Using a silver paint marker, label clean digestion bottles with the appropriate sample numbers and standard identifications for each sample and standard to be digested.

7.6 Using calibrated adjustable pipettes, prepare calibration standards by adding 0 uL, 20 uL, 50 uL, 100 uL, 500 uL, and 1000 uL of Intermediate Mercury Standard A to separate appropriately-labeled digestion bottles. The mercury concentrations of these calibration standards will be, respectively, 0 ug/L (calibration blank), 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L, and 10.0 ug/L. The 0.2 ug/L and 0.5 ug/L standards are analyzed during analysis 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.

7.7 Using a calibrated adjustable pipette, prepare the initial calibration verification (ICV) standard (refer to Section 8) by adding 600 uL of Intermediate Mercury Standard B to an appropriately labeled digestion bottle. The mercury concentration of the ICV will be 6.0 ug/L.

7.8 Prepare an appropriate number of calibration blanks (ICB/CCB) and preparation blanks (PBW) by adding 1.0 mL of laboratory reagent grade water to labeled digestion bottles.

7.9 Prepare an appropriate number of laboratory control samples (LCSS) by weighing appropriate masses of solid reference material into labeled digestion bottles. The

mercury concentration of these LCSSs will depend on the solid reference material used, and the mass of each aliquot. Refer to Figure 4 for an example certificate of analysis for a solid reference material.

7.10 Matrix spikes are prepared by adding 100 uL of Intermediate Mercury Std A to each matrix spike sample. The amount of mercury added to each matrix spike increases the final digestate concentration by 1.0 ug/L.

7.11 All calibration standards, QC samples, and blanks are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, Steps 7.12 through 7.16 of this SOP.

SAMPLE PREPARATION AND DIGESTION

7.12 Weigh three approximate 0.2 g portions (a total of approximately 0.6 g) of untreated sample from different parts of the sample container and place them in the bottom of a labeled digestion bottle. The purpose of using three portions is to obtain a representative sample from the sample container.

7.13 Add 5 mL of laboratory reagent grade water and 5 mL of aqua regia to each sample, standard, and QC sample. Place bottles in a water bath located in a fume hood and heat for 2 minutes at 95° C. Remove the bottles from the water bath and allow them to cool in a fume hood.

7.14 Add 50 mL of laboratory reagent grade water and 15 mL of potassium permanganate solution to each digestion bottle, swirl to mix, and allow to stand for at least 15 minutes. Samples that contain large amounts of oxidizable organic matter may require additional 15 mL aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 15 mL aliquots to samples as necessary until the purple color persists for 15 minutes. If any of the samples requires these additional aliquots of permanganate, note that fact on the mercury preparation benchsheet and accordingly adjust the final volumes recorded on the benchsheet for those samples.

When a persistent purple color has been obtained for all samples, place the digestion bottles in the water bath and heat for 30 minutes at 95° C.

7.15 Remove the bottles from water bath and allow them to cool in a fume hood. If any of the samples have become colorless during heating, add additional 15 mL aliquots of potassium permanganate solution as necessary to obtain a persistent purple color and heat for an additional 30 minutes at 95° C. Record any information regarding additional permanganate aliquots on the mercury preparation benchsheet

and accordingly adjust the final volumes recorded on the benchsheet for the samples affected.

7.16 Add 6 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion bottle and swirl to mix. Perform this addition in a fume hood, as chlorine gas may be evolved. This will reduce the excess permanganate, and the sample will change from purple to colorless. Add 50 mL of laboratory reagent grade water to each bottle. Wait at least 30 seconds before proceeding with analysis.

INSTRUMENTAL ANALYSIS

7.17 Digested mercury samples are analyzed using the CETAC M6100 Mercury Analyzer. Analysis is automated and is controlled by the QuickTrace software running on a dedicated PC. Detailed instructions for setting up the instrument and running samples are given Katahdin SOP CA-629, "Operation and Maintenance of the CETAC M6100 Mercury Analyzer". The following information specifically pertains to analysis of digested samples in accordance with USEPA Method 7471, and should be used in conjunction with the instructions given in Katahdin SOP CA-629.

7.18 Instrument operating conditions and quality control acceptance limits are specified in the instrument software in "templates". The template that is used to analyze digested samples in accordance with USEPA Method 7471 is named "SW846-7470-7471".

7.19 Prior to analysis, digested samples, standards, and QC samples are decanted into autosampler tubes which are placed in racks on the instrument's autosampler. The "standards" autosampler rack has 10 positions for 25 x 100 mm autosampler tubes (50 mL capacity). Tubes containing the calibration standards, the ICV, the CCV, the ICB/CCB, and the PQL standard are placed in the appropriately labeled positions in this autosampler rack.

7.20 Client samples, batch QC samples (preparation blanks and laboratory control samples), and matrix QC samples (duplicates and matrix spikes) are decanted into 17 x 100 mm autosampler tubes (15 mL capacity), which are placed in the one of the "samples" autosampler racks. The "samples" autosampler racks have 60 positions for 17 x 100 mm autosampler tubes. Instructions for filling the "samples" autosampler racks, including recording the rack position of each sample, are contained in Katahdin SOP CA-629, "Operation and Maintenance of the CETAC M6100 Mercury Analyzer".

METHOD OF STANDARD ADDITIONS

7.21 The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

7.21.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration C_x is calculated:

$$C_X = \frac{S_B V_S C_S}{(S_A - S_B) V_X}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

7.21.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 3. A linear regression program may be used to obtain the intercept concentration.

7.21.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

1) 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.

2) 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.

3) The determination must be free of spectral interference and corrected for nonspecific background interference.

DATA REDUCTION AND REPORTING

7.22 Results are obtained in units of ug/L in the digestate. Results that exceed the calibration range of the instrument may not be reported - the sample must be appropriately diluted and reanalyzed. Results for diluted samples must be multiplied by the dilution factor prior to reporting. If additional aliquots of potassium permanganate were added during digestion, the change in digestate final volume must be taken into account in calculating the final result. Mercury results for solid samples are reported in units of ug/g, calculated on a dry weight basis. Calculation of mercury results for solid samples is performed automatically by the Metals reporting database, as follows:

$$\text{Mercury Concentration in Solid (mg/kg dry wt.)} = \frac{(C) \times (DF) \times (FV) \times 100}{(W) \times (TS)}$$

where C = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Digestate final volume (L)
W = Digested wet sample weight (g)
TS = Total Solids (%)

7.23 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported as "<PQL".

TITLE: **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 7471 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.6 through 7.10 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The supervisor, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument by each method. This determination requires seven replicate analyses of laboratory reagent grade water spiked 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 reagent grade water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value

to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

ANALYTICAL RUN QC

8.3 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). Because mercury may be adsorbed onto the walls of glass and plastic containers, the intermediate standard used to prepare 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.

8.4 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared from a standard source different than that of the calibration standard and at a concentration within the working range of the instrument. The result of the ICV must fall within 90% to 110% of the expected value. If the ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.

8.5 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.

8.6 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 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.

8.7 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 50% to 150% of the expected values. No corrective action has been established at this time.

PREPARATION BATCH QC SAMPLES

8.8 A preparation blank (PBS), consisting of laboratory reagent grade water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL). If a preparation blank fails, results may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL, associated sample results that are greater than or equal to ten times the measured preparation blank concentration may be reported with "B" notation. Associated sample results that are below the PQL may be reported without notation.

8.9 A laboratory control sample (LCSS), consisting of solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested. The laboratory uses a reference value and statistical acceptance limits for laboratory control samples are supplied by the vendor of the solid reference material.

SAMPLE MATRIX QC SAMPLES

8.10 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.

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where:

D_1 = Spike sample result
D_2= Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

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 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 7471 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Wastes, United States Environmental Protection Agency, USEPA SW 846, Third Edition, Final Update III (12/96), Method 7471A

QuickTrace M6100 Mercury Analyzer Operator Manual Version 1.0.1, CETAC Technologies

QuickTrace Mercury Analyzer Software Manual, CETAC Technologies.

TITLE: **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

List of Tables and Figures

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Table 2 Method Modifications
Figure 1 Example Mercury Preparation Logbook Page
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Figure 3 Example Certificate of Analysis for a Solid Reference Material

TITLE: **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA 7471	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ≥ 0.995.	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL.	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within ± 50% of true value.	No corrective action required at this time.
	Continuing Calibration Verification (CCV)	At beginning or run, after every 10 samples, and at end of the run	Recovery within ± 20% of true value	Repeat calibration and reanalyze all samples analyzed since the last successful CCV.
	Continuing Calibration Blank (CCB)	At beginning or run, after every 10 samples, and at end of the run	Less than PQL.	Repeat calibration and reanalyze all samples analyzed since the last successful CCB.
	Preparation Blank (PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and < 10x the blank concentration.
	Laboratory Control Sample (LCSS)	One per digestion batch of 20 or fewer samples.	Recovery within vendor-supplied acceptance limits.	Redigest all affected samples.

TITLE: **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

TABLE 1, cont'd

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA 7471	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ±25% of true value, if sample > 4x spike value.	Flag results.
	Matrix Spike Duplicate Sample (P)	One per digestion batch of 20 or fewer samples.	1) Recovery ± 25% of true value, if sample < 4x spike added. 2) RPD 20% for duplicate spikes.	Flag results
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < PQL	1) Repeat IDL study. 2) Raise PQL.
	Method Detection Limit (MDL) Study	Annually.	Ideally, PQL = at least 3 X the MDL.	1) Repeat MDL study. 2) Raise PQL.

TITLE: **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-611-03	USEPA METHOD 7471, current revision
Reagents	Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	Sampling and gas stream switching performed automatically by mercury analyzer.	Sampling and gas stream switching performed manually by analyst.
QC – Calibration Verification	1) Known reference sample (ICV) analyzed daily. 2) Calibration verified after every 10 samples with CCV.	1) Known reference sample analyzed quarterly. 2) Calibration verified after every 20 samples.
QC - Calibration Blanks	Acceptance criteria employed for 7471: ± PQL	Acceptance criteria stated in 7471: ± MDL

TITLE: **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK

Katahdin Analytical Services, Inc.

Metals Preparation Benchsheet

Reagent Information:
JT Baker HNO3: Y42044 JT Baker HCL: N/A JT Baker H2SO4: Y19042
JT Baker KMNO4: MR164 JT Baker K2S2O8: MR156 JT Baker NH2OH-HCl: MR167

Method: 7470

Standards Information:
1ppm A = MW7971
1ppm B = MW7972
LCSW = 125uL of 1ppm B to 25mL
Spike(S/P) = 25uL of 1ppm B to 25mL
TCLPMS(M) = 200uL of 1ppm N/A to 100mL
ICV = 600uL of 1ppm A to 100 mL
S0.2 = 20uL of 1ppm B to 100 mL
S0.5 = 50uL of 1ppm B to 100 mL
S1.0 = 100uL of 1ppm B to 100 mL
S5.0 = 500uL of 1ppm B to 100 mL
S10.0 = 1000uL of 1ppm B to 100 mL

REVIEWED
MJF 1.14.04
KATAHDIN ANALYTICAL
METALS SECTION

Digestion Start Time (@ 95 °C): 13:30 Digestion End Time: 15:30

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LCSWUA13HGW0	UA13HGW0	0.025	L	0.025	L	AQ	HG	EAM	01/13/2004	N/A	N/A	N/A	N/A		
PBWUA13HGW0	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004	N/A	N/A	N/A	N/A		
WU0041-001	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-001P	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-001S	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-002	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-003	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-004	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-005	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-006	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-007	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-001	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-002	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-003	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-004	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-005	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-006	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-007	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-008	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-009	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-010	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						

MJF 1.14.04

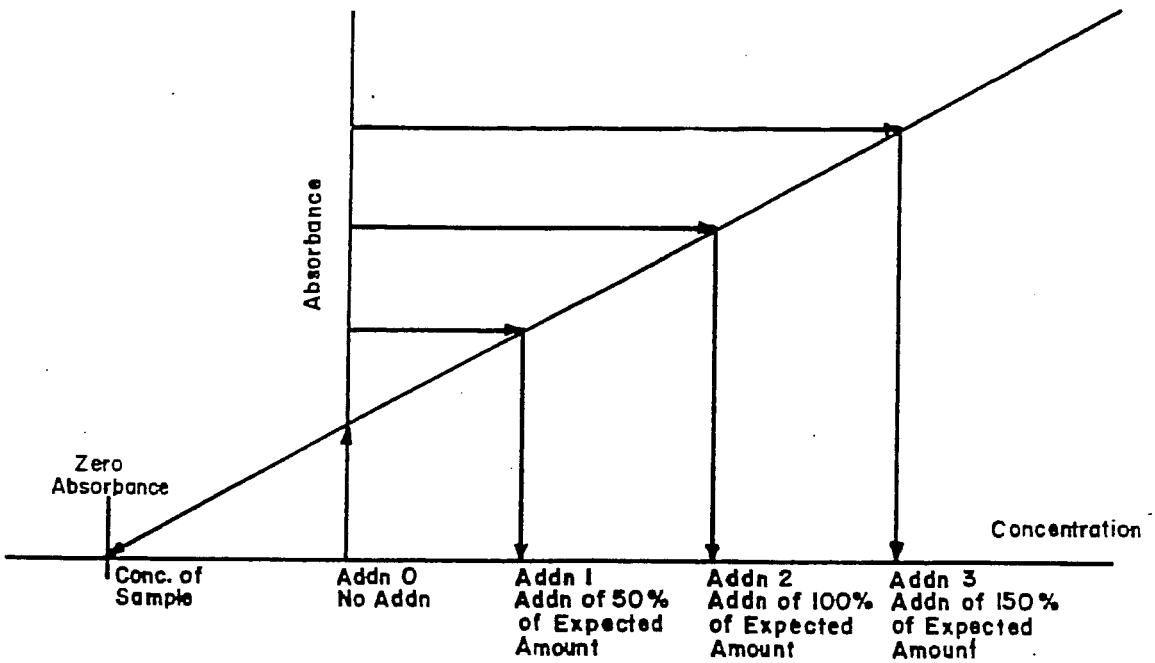
Digestion performed by: [illegible] On: 01-13-04

Page: UA023 Revision: 00

TITLE: **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

FIGURE 2

STANDARD ADDITIONS PLOT



TITLE: **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

FIGURE 3

EXAMPLE CERTIFICATE OF ANALYSIS FOR A SOLID REFERENCE MATERIAL

Rec'd 12/08/00

ENVIRONMENTAL RESOURCE ASSOCIATES
ARVADA, COLORADO 1-800-372-0122

Certification

PriorityPollutnT™/CLP Inorganic Soils - Microwave Digestions Quality Control Standards

Catalog No PPS-46

Lot No. 245

Parameter	Total Concentration[1]	Method 3051 HNO3 only: Certified Value[2]	Method 3051 HNO3 only: Performance Acceptance Limits™ [3]	Method 3051 HNO3 and HCl: Certified Value[2]	Method 3051 HNO3 and HCl: Performance Acceptance Limits™ [3]
TRACE METALS PriorityPollutnT™ (Catalog No 540)	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg
Aluminum	53800*	5580	3610 - 7550	6110	3260 - 8960
Antimony	198	29.2	D.L. - 93.7	119	32.6 - 206
Arsenic	152	131	97.3 - 164	137	102 - 172
Barium	894*	115	89.0 - 142	127	98.2 - 157
Beryllium	103	93.7	73.5 - 114	92.1	72.2 - 112
Boron	158	106	78.1 - 134	126	92.5 - 159
Cadmium	131	119	91.8 - 147	119	91.3 - 146
Calcium	16100*	11400	8520 - 14300	11400	8520 - 14300
Chromium	135	83.1	66.4 - 99.8	85.2	68.1 - 102
Cobalt	125	118	93.8 - 142	127	101 - 153
Copper	136	115	93.9 - 135	119	97.4 - 140
Iron	26400*	9190	5590 - 12800	10900	6630 - 15200
Lead	172	139	106 - 172	137	105 - 170
Magnesium	5520*	2430	1960 - 2900	2550	2060 - 3040
Manganese	520*	291	235 - 347	320	259 - 382
Mercury	2.60	2.48	1.69 - 3.27	2.48	1.69 - 3.27
Molybdenum	111	88.1	59.0 - 117	108	82.9 - 134
Nickel	186	160	126 - 195	167	131 - 204
Potassium	31000*	2920	2280 - 3560	3040	2370 - 3710
Selenium	105	88.3	65.4 - 111	88.0	65.2 - 111
Silver	132	56.9	11.9 - 102	46.1	31.0 - 61.1
Sodium	12100*	771	523 - 1020	772	524 - 1020
Strontium	294*	76.1	55.2 - 96.9	78.7	57.1 - 100
Thallium	154	137	78.7 - 196	113	64.8 - 162
Tin	117	57.7	D.L. - 129	119	88.8 - 149
Titanium	2900*	82.6	53.9 - 111	115	75.3 - 156
Vanadium	131	65.8	44.9 - 86.6	72.8	49.6 - 95.9
Zinc	100	72.8	11.0 - 135	69.2	53.5 - 84.9

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TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Prepared By: George Brewer Date: 01/01

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: Joel C. Burton Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1·29·01

General Manager: Dennis F. Hufnagle Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
00 7470A	NA	DN	1·29·01	1/29/01
01	Revised Sect. 4, 5 and 7 to reflect current practice. Revised Sect. 8 to reflect current QC Limits. Revised sect. 10 to reflect current Applicable Documents and references. Removed figure 2. Update table 1 to reflect current QC Limits. Minor changes throughout	LAD	02-16-05	02-16-05

TITLE: **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

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

I acknowledge receipt of copy ___ of document **SOP CA-615-01,** titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-615-01,** titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470.**

Recipient: _____ Date: _____

TITLE: **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services, Inc. personnel for the digestion and analysis aqueous samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in groundwaters, aqueous wastes, and mobility-procedure extracts under USEPA Method 7470 (Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods, SW-846, 2nd edition, 1982 (revised 1984), 3rd edition, 1986, and Updates I, II, IIA, and III 1996, Office of Solid Waste and Emergency Response, U.S. EPA.

1.1 Definitions

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

PB - Preparation Blank - Laboratory grade reagent water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7470. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7470 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Rubber gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Management Plan and Safety Manual 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.

3.0 INTERFERENCES

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold

vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low bias for analytical results. Certain volatile organic materials will also non-specifically absorb radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate and potassium persulfate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Sea waters, brines, and industrial effluents high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

4.0 APPARATUS AND MATERIALS

4.1 40 mL VOA vials, for use as digestion vessels.

4.2 250 mL Pyrex media bottles with plastic screw caps, for use in digesting calibration standards.

4.3 Water bath capable of maintaining a constant temperature of 95° C.

4.4 Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents

4.6 Spirit-filled thermometer, NIST-traceable, covering the range from 20° to 110° C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.

4.7 Disposable graduated polystyrene sample cups, 200 mL capacity

4.8 CETAC M-6100 automated mercury analyzer and associated peripherals and parts

4.9 Disposable graduated dose cups, 30 mL capacity

Refer to Katahdin SOP CA-629, current revision, "Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer" for additional required materials.

5.0 REAGENTS

5.1 Laboratory grade reagent water – mercury-free water meeting the specifications of ASTM Type II water

5.2 Concentrated sulfuric acid, trace metals grade

5.3 Concentrated nitric acid, trace metals grade

5.4 Concentrated hydrochloric acid, trace metal grade

5.5 Potassium permanganate solution, 5% w/v: Dissolve 50 g of potassium permanganate in 1 L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.

5.6 Potassium persulfate solution, 5% w/v: Dissolve 50g of potassium permanganate in 1L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.

5.7 Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in laboratory grade reagent water and dilute to a final volume of 1 L.

5.8 Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory grade reagent water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. Reagent should be labeled as suitable for use in mercury determination.

5.9 Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. This intermediate standard is used to prepare calibration standards, matrix spikes, CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard A must be prepared fresh monthly ,and disposed of appropriately after use. (Note: the concentrations of all stock standards must be certified by the vendors as traceable to NIST reference materials).

5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. The source of the stock standard used to prepare Intermediate Mercury Standard B must be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard B must be prepared fresh monthly, and disposed of appropriately after use.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Aqueous samples to be analyzed for mercury should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	28 days
Aqueous (dissolved)	P, G	250 mL	HNO_3 to pH < 2	28 days

[1] P = polyethylene or , G = glass

7.0 PROCEDURES

BOTTLE PREPARATION

7.1 Mercury digestions are performed in two different types of vessels. Calibration standards, the Initial Calibration Verification (ICV) standard, and the Initial/Continuing Calibration Blank (ICB/CCB) are prepared in 250 mL Pyrex media bottles. Large bottles are used to provide sufficient volumes of these standards to allow for multiple reanalyses when required. Field samples, Method Blanks, and Laboratory Control Samples are digested in 40 mL VOA vials. These smaller vials provide enough digestate to allow one or two reanalyses when required, but reduce the amounts of samples consumed and waste generated.

VOA vials are reused if the samples they have contained have no measurable mercury above the PQL. After the previous contents of the vials have been discarded, these vials are segregated according to whether the measured mercury concentrations of the previous contents were above the PQL (contaminated vials) or below the PQL (uncontaminated vials). Labels are removed from the vials by wiping with a paper towel saturated with toluene. Uncontaminated vials are rinsed with laboratory grade reagent water. Contaminated vials are discarded.

The Pyrex media bottles in which standards are prepared are emptied, rinsed, and reused. Each of these bottles is permanently marked with the concentration of the standard it contains.

PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

7.2 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS Metals database and print out a copy of the sample prep bench sheet. All necessary details of sample preparation (standards preparation information, digestion times, initial and final volumes, pertinent observations, etc.) must be recorded on this spreadsheet, which will be bound in the Mercury Preparation Logbook. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.

7.3 Using a silver paint marker, label clean VOA vials with the appropriate sample numbers and standard identifications for each sample and standard to be digested.

7.4 Use a bottle-top dispenser to add 100 mL of laboratory grade reagent water to 6 standards digestion bottles (250 mL media bottles). Using calibrated adjustable pipettes, prepare calibration standards by adding 0 uL, 20 uL, 50 uL, 100 uL, 500 uL, and 1000 uL of Intermediate Mercury Standard A to separate appropriately-labeled media bottles containing 100 mL of laboratory grade reagent water. The mercury concentrations of these calibration standards are, respectively, 0 ug/L (calibration blank), 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L, and 10.0 ug/L. The 0.2 ug/L and 0.5 ug/L standards are analyzed after calibration as the PQL standard and the CCV (refer to Section 8.0), respectively, as well as being used in the creation of the calibration curve.

7.5 Add 100 mL of laboratory grade reagent water to the media bottle labeled "ICV". Using a calibrated adjustable pipette, prepare the Initial Calibration Verification standard (refer to Section 8) by adding 600 uL of Intermediate Mercury Standard B to the water in this bottle, and record the bottle number in the Mercury Preparation Logbook. The mercury concentration of the ICV is 6.0 ug/L.

7.6 Prepare an appropriate number of preparation blanks (PBW) by adding 25 mL of laboratory grade reagent water to labeled vials.

7.7 Prepare an appropriate number of laboratory control samples (LCSW) by adding 125 uL of Intermediate Mercury Standard A to labeled digestion vials containing 25 mL of laboratory grade reagent water. The mercury concentration of each LCSW is 5.0 ug/L.

7.8 Matrix spikes are prepared by adding 25 uL of Intermediate Mercury Std A to 25 mL aliquots of samples. The concentration of mercury added to each matrix spike is 1.0 ug/L.

7.9 All QC samples and blanks are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, sections 7.10 through 7.13 of this SOP. The volumes of reagents added to the standards prepared in the media bottles are four times those listed in sections 7.10 through 7.13.

SAMPLE PREPARATION AND DIGESTION

7.10 Using a graduated disposable dosecup, transfer 25 mL of sample, or an aliquot diluted to 25 mL, to a digestion vial. Add 1.25 mL of concentrated sulfuric acid and 0.625 mL of concentrated nitric acid, swirling to mix after each addition. Add 3.75 mL of potassium permanganate solution, swirl to mix, and allow to stand for at least 15 minutes. Samples that contain large amounts of organic substances may require additional 3.75 mL aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 3.75 mL aliquots to samples as necessary until the purple color persists for 15 minutes. If any of the samples require these additional aliquots of potassium permanganate solution, record the additional volume used for each sample on the mercury preparation benchsheet.

7.11 Add 2 mL of potassium persulfate solution to each sample. Cap the vials and place them in a preheated water bath. Monitor the temperature of the bath with a spirit thermometer throughout the digestion. The temperature of the water bath will fall below 95° C upon addition of the digestion vials. After the temperature of the bath has risen back to 95° C, continue heating the samples at 95° C for two hours.

7.12 Remove bottles from the water bath and allow to cool to room temperature. If the purple permanganate color has failed to persist after digestion in any of the samples, add additional 3.75 mL aliquots of potassium permanganate solution as required to the samples, and record these additions in the mercury preparation benchsheet. Heat the samples that required additional permanganate in the water bath at 95° C for an additional two hours. Remove the bottles from the water bath and allow to cool to room temperature. If the purple color fails to persist after the

second heating step, consult the Department Manager for advice on how to proceed.

7.13 Add 1.5 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion vial and swirl to mix. This will reduce the excess permanganate, and the sample will change from purple to colorless. Wait at least 30 seconds before proceeding with analysis.

INSTRUMENTAL ANALYSIS

7.14 Digested mercury samples are analyzed using the CETAC M-6100 Automated Mercury Analyzer. Analysis is automated and is controlled by the QuickTrace Mercury Analyzer software running on a dedicated PC. Detailed instructions for setting up the instrument and analyzing samples are given Katahdin SOP CA-629, "Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer".

METHOD OF STANDARD ADDITIONS

7.15 The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

7.15.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration C_x is calculated:

$$C_X = \frac{S_B V_S C_S}{(S_A - S_B) V_X}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

7.15.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 3. A linear regression program may be used to obtain the intercept concentration.

7.15.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

1) 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.

2) 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.

3) The determination must be free of spectral interference and corrected for nonspecific background interference.

DATA REDUCTION AND REPORTING

7.16 Results are obtained in concentration units (ug/L) from the instrument. Electronic instrument data files are imported into the Metals ACCESS database for data reduction. Sample preparation information (initial sample volumes and final digestate volumes) are entered directly into the Metals ACCESS database to allow calculation of final results for reporting. Results are calculated as follows:

$$\text{Mercury concentration (ug/L)} = \frac{MC \times DF \times IV}{FV}$$

Where: MC = Measured mercury concentration (ug/L)
DF = Dilution factor at instrument
IV = Initial sample volume (mL)
FV = Final digestate volume (mL)

7.17 Results that exceed the calibration range of the instrument may not be reported - the sample must be appropriately diluted and reanalyzed. Results for diluted samples should be multiplied by the dilution factor prior to reporting. If additional aliquots of potassium permanganate were added during digestion, the resulting dilution must be corrected for before reporting.

7.18 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported to the PQL and flagged with a "U" qualifier.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 7470 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.4 through 7.8 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

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

TITLE: **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

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.

ANALYTICAL RUN QC

8.3 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.

8.4 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared from a standard source different than that of the calibration standard and at a concentration within the working range of the instrument. The result of the ICV must fall within 90% to 110% of the expected value. If the ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.

8.5 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.

8.6 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 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.

8.7 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 50% to 150% of the expected values. No corrective action has been established at this time.

PREPARATION BATCH QC SAMPLES

8.8 A preparation blank (PBW), consisting of laboratory grade 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). If a preparation blank fails, results may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. Associated sample results that are below the PQL may be reported without notation.

8.9 A laboratory control sample (LCSW), consisting of spiked reagent carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless laboratory-generated statistical limits are available. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested.

SAMPLE MATRIX QC SAMPLES

8.10 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.

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

$$\text{RPD (\%)} = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where:

D_1 = Spike sample result
D_2= Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) 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 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 USEPA Method 245.1 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, and III 1996, Office of Solid Waste and Emergency Response, U.S. EPA, Method 7470

TITLE: **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

QuickTrace M-6100 Mercury Analyzer – Operator Manual, Version 1.0.1, CETAC Technologies, 2003.

QuickTrace Mercury Analyzer – Software Manual, Version 1.0, Cetac Technologies, June 2002

List of Tables and Figures

Table 1 QC Requirements
Table 2 Method Modifications
Figure 1 Example Mercury Preparation Logbook Page
Figure 2 Standard Additions Plot

TITLE: **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA 7470	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ≥ 0.995.	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL.	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within ± 50% of true value.	No corrective action required at this time.
	Continuing Calibration Verification (CCV)	At beginning or run, after every 10 samples, and at end of the run	Recovery within ± 20% of true value	Repeat calibration and reanalyze all samples analyzed since the last successful CCV.
	Continuing Calibration Blank (CCB)	At beginning or run, after every 10 samples, and at end of the run	Less than PQL.	Repeat calibration and reanalyze all samples analyzed since the last successful CCB.
	Preparation Blank (PBW)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and < 10x the blank concentration.
	Laboratory Control Sample (LCSW)	One per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value.	Redigest all affected samples.

TITLE: **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

TABLE 1, cont'd

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA 245.1	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ±25% of true value, if sample > 4x spike value.	Flag results.
	Matrix Spike Duplicate Sample (P)	One per digestion batch of 20 or fewer samples.	1) Recovery ± 25% of true value, if sample < 4x spike added. 2) RPD 20% for duplicate spikes.	Flag results
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < PQL	1) Repeat IDL study. 2) Raise PQL.
	Method Detection Limit (MDL) Study	Annually.	Ideally, PQL = at least 3 X the MDL.	1) Repeat MDL study. 2) Raise PQL.

TITLE: **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-615-01	USEPA METHOD 7470
Reagents	Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	1)Sampling and gas stream switching performed automatically by mercury analyzer. 2)Working Mercury standard prepared monthly in 2% nitric; calibration standards prepared fresh daily.	1)Sampling and gas stream switching performed manually by analyst. 2)Working Mercury standard prepared fresh daily and acidity maintained at 0.15% nitric.
QC – Calibration Verification	1) Known reference sample (ICV) analyzed daily. 2) Calibration verified after every 10 samples with CCV.	1) Known reference sample analyzed quarterly. 2) Calibration verified after every 20 samples.
QC - Calibration Blanks	Acceptance criteria employed for 245.1: ± PQL	Acceptance criteria stated in 245.1: ± MDL

TITLE: **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK

Katahdin Analytical Services, Inc.

Metals Preparation Benchsheet

Reagent Information:

JT Baker HNO3: Y42044 JT Baker HCL: N/A JT Baker H2SO4: Y19092

JT Baker KMNO4: MR164 JT Baker K2S2O8: MR156 JT Baker NH2OH-HCl: MR167

Method: 7470

REVIEWED

MJF 1.14.04
KATAHDIN ANALYTICAL METALS SECTION

Standards Information:

1ppm A = MW7971
1ppm B = MW7972

TCLPMS(M) = 200uL of 1ppm N/A to 100mL

S0.5 = 50uL of 1ppm B to 100 mL
S1.0 = 100uL of 1ppm B to 100 mL
S5.0 = 500uL of 1ppm B to 100 mL
S10.0 = 1000uL of 1ppm B to 100 mL

LCSW = 125uL of 1ppm B to 25mL
Spike(S/P) = 25uL of 1ppm B to 25mL

ICV = 600uL of 1ppm A to 100 mL
S0.2 = 20uL of 1ppm B to 100 mL

Digestion Start Time (@ 95 °C): 13:30 Digestion End Time: 15:30

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LCSWUA13HGW0	UA13HGW0	0.025	L	0.025	L	AQ	HG	EAM	01/13/2004	N/A	N/A	N/A	N/A		
PBWUA13HGW0	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004	N/A	N/A	N/A	N/A		
WU0041-001	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-001P	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-001S	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-002	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-003	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-004	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-005	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-006	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-007	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-001	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-002	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-003	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-004	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-005	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-006	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-007	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-008	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-009	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-010	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						

MJF 1.14.04

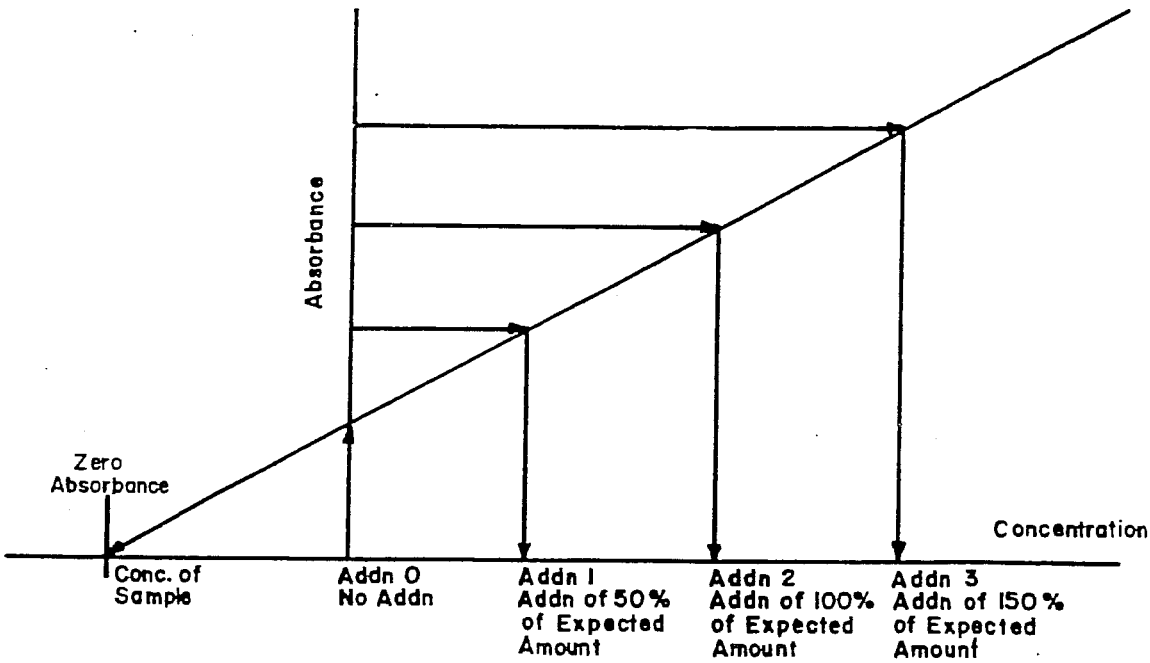
Digestion performed by: Sam On: 01-13-04

Page: UA023 Revision: 00

TITLE: **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

FIGURE 2

STANDARD ADDITIONS PLOT



KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-627
Revision History
Cover Page
Page 1

TITLE: TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020

Prepared By: George Brewer Date: 03/01

Approved By:

Group Supervisor: George Brewer Date: 04/02/01

Operations Manager: Joel C. Burton Date: 3/29/01

QA Officer: Deborah J. Nadeau Date: 03·27·01

General Manager: Dennis P. Hufnagle Date: 04/03/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Changed acid solution conc. changed Run ID naming convention added data reduction and reporting procedures updated standards tables (4-8) updated Table 10 in include ISIS configuration	LAD	02-16-05	02-16-05
02	Sect. 4.2 - changed tubing size Sect. 5 - changed acid conc.'s Sect. 7 - major changes to reflect current practises including reporting data in the metals database. Sect. 8 - major changes updating acceptance criteria. Updated Tables 1, 3 → 8, 10 & 11	LAD	04/06	04/06
03	Updated Tables 4, 5 and 6 with current standards. Updated Table 1 with serial dilution, Post Digestion Matrix spike, MSA, ICS-A, ICS-AB and IDL minimum frequency or criteria. Updated Sect. 8 regarding client specific requirements.	LAD	07/07	07/07

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

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

I acknowledge receipt of copy ___ of document **SOP CA-627-02**, titled **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**
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STANDARD OPERATING PROCEDURE

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Recipient: _____Date:_____

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of sub-ppb (ug/L) concentrations of a large number of elements in water samples and in waste extracts or digests. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are required.

ICP-MS has been applied to the determination of over 60 elements in various matrices. Analytes for which EPA has demonstrated the acceptability Method 6020 in a multi-laboratory study on solid wastes are listed as "analytes" in Table 3. Instrument detection limits, sensitivities, and linear ranges will vary with the matrices, and operating conditions.

If Method 6020 is used to determine any analyte not listed in Table 3, it is the responsibility of the analyst to demonstrate the accuracy and precision of the method in the waste to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality.

An appropriate internal standard is required for each analyte determined by ICP-MS. Recommended internal standards are ^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho,and ^{209}Bi. The lithium internal standard should have an enriched abundance of ^{6}Li, so that interference from lithium native to the sample is minimized. Other elements may need to be used as internal standards when samples contain significant amounts of the recommended internal standards.

1.1 Definitions:

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-MS - Inductively Coupled Plasma Mass Spectrometry.

ICS - Interference Check Samples - Two standards (ICS-A and ICS-AB) used to verify the effectiveness of interference correction equations. Solution ICS-A contains only interferents (Al, Ca, Fe, Mg, Na, K, P, S, Mo, Ti, C, Cl) at high

concentrations; solution ICS-AB contains interferents at the same concentrations as well as analytes at low (20 ug/L) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

Post-Digestion Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP-MS analysis by USEPA Method 6020 who are knowledgeable in the recognition and in the correction of spectral, chemical, and physical interferences in ICP-MS. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in ICP-MS analysis by USEPA Method 6020 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also

be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Chemical Hygiene 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 and use of all safety equipment.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Spilled samples and reagents should be cleaned up from instrument and laboratory surfaces immediately.

Liquid argon represents a potential cryogenic and suffocation hazard and safe handling procedures should be employed at all times when handling liquid argon tanks and fittings. Safety glasses and cryogenic-resistant gloves should be worn when changing or adjusting argon tanks.

The Agilent 7500 ICP-MS spectrometer is safety-interlocked to prevent user exposure to harmful electrical voltages, radio frequency emissions, ultraviolet radiation, high temperatures, and other hazards. At no time should the operator attempt to disable these interlocks or operate the instrument if any safety interlock is suspected to be disabled

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention and waste minimization techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in ICP-MS spectrometry may contain high concentrations of acids and toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested samples and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled "Waste Stream A") that is located in the Metals Instrument lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Chemical Hygiene Plan and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples that require total ("acid-leachable") values must be digested using appropriate sample preparation methods (such as USEPA Methods 3005 - 3051).

2.2 USEPA Method 6020 describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled argon plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of a vacuum interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

3.0 INTERFERENCES

Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). The Agilent 7500 ChemStation data system is used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences which could affect ICP-MS determinations have been identified. Examples include $ArCl^+$ ions on the As signal and MoO^+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotopic abundances from the literature, the most precise coefficients for an instrument must be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting statistics. Because the ^{35}Cl natural abundance of 75.77 percent is 3.13 times the ^{37}Cl abundance of 24.23 percent, the chloride correction for

arsenic can be calculated (approximately) as follows (where the $^{38}Ar^{37}Cl^{+}$ contribution at m/z 75 is a negligible 0.06 percent of the $^{40}Ar\ ^{35}Cl^{+}$ signal):

Corrected ^{75}As signal (using natural isotopic abundances for coefficient approximations) =

(m/z 75 signal) - (3.13) (m/z 77 signal) + (2.73) (m/z 82 signal),

where the final term adjusts for any selenium contribution at 77 m/z.

NOTE: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}Se^{+}$, (e.g. , $^{81}BrH^{+}$ from bromine wastes or ^{82}Kr from krypton contamination in the Ar).
Similarly:

Corrected ^{114}Cd signal (using natural isotopic abundances for coefficient approximations) =

(m/z 114 signal) - (0.027)(m/z 118 signal) - (1.63)(m/z 108 signal),

where last 2 terms adjust for any tin or MoO^{+} contributions at m/z 114.

NOTE: Cadmium values will be biased low by this type of equation when $^{92}ZrO^{+}$ ions contribute at m/z 108. Also, use of m/z 111 for Cd is even subject to direct ($^{92}ZrOH^{+}$) ions and indirect ($^{90}ZrO^{+}$) additive interferences when Zr is present.

NOTE: As for the arsenic equation above, the coefficients in the Cd equation are only illustrative. The most appropriate coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<l percent) counting precision.

The interference correction equations that are used by this laboratory in performing USEPA Method 6020 are listed in Table 3. The accuracy of these types of equations is based upon the constancy of the observed isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the "parent" ion have not been found to be reliable, e.g., oxide levels can vary. If a correction for an oxide ion is based upon the ratio of parent-to-oxide ion intensities, the correction must be adjusted for the degree of oxide formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferent. This type of correction has been reported for oxide-ion corrections using ThO^{+}/Th^{+} for the determination of rare earth elements. The use of aerosol desolvation and/or mixed plasmas have been shown to greatly reduce molecular interferences (the Agilent 7500 ICP-MS spectrometer employs spray chamber cooling to effect aerosol desolvation). These techniques can be used provided that method detection limit, accuracy, and precision requirements for analysis of the samples can be met.

3.1 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) are recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes. When the intensity level of an internal standard is less than 30 percent or greater than 120 percent of the intensity of the first standard used during calibration, the sample must be reanalyzed after a fivefold (1+4) or greater dilution has been performed.

3.2 Memory interferences can occur when there are large concentration differences between samples or standards that are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory interferences that are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

4.0 APPARATUS AND MATERIALS

4.1 Agilent 7500 ICP-MS system, consisting of the Agilent 7500 ICP-mass spectrometer and its controlling computer data station. The spectrometer is capable of providing resolution better than or equal to unit resolution at 10% peak height. The Agilent 7500 mass range of 2-260 amu exceeds the method requirement of 2- 240 amu. The Agilent 7500 ChemStation software allows automatic corrections for isobaric interferences and correction for internal standard responses as required by the method. All critical argon flows including nebulizer argon are under mass flow controller control and a peristaltic pump is used for sample introduction. Peripheral equipment include a Cetac ASX-500 random-access autosampler, and Agilent ISIS dilution and prep station, and an Agilent LaserJet printer.

4.2 Peristaltic pump tubing – 3-stop Tygon white-white (1.02 mm ID), orange-blue (0.25 mm ID), and 2-stop red-red (1.14 mm ID). 3-stop Pharmed blue-yellow (1.52 mm ID).

4.3 15 ml 17x100 mm polypropylene or polystyrene disposable test tubes for samples and 50 ml polypropylene centrifuge tubes for standards.

4.4 Automatic adjustable-volume pipetters of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Trace metal grade pipette tips.

4.6 Volumetric glassware or plasticware of suitable precision and accuracy.

4.7 Talc free vinyl gloves.

4.8 Argon gas supply (high purity grade gas or liquid, 99.99%).

4.9 For the determination of trace levels of elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory environment from dust etc. A clean laboratory work area, designed for trace element sample handling must be used. Standards, samples and blanks should be exposed to the laboratory environment as little as possible. The use of preparation blanks and spikes should be used to verify the absence of sources of contamination and loss. If necessary, polypropylene sample tubes should be rinsed and stored in dilute acid prior to use.

NOTE: Chromic acid must not be used for cleaning glassware for trace metals analysis.

5.0 REAGENTS

5.1 Acids used in the preparation of standards and for sample processing must be of high purity. Redistilled acids are recommended because of the high sensitivity of ICP-MS. Mallincrodt/Baker "Instra-Analyzed" trace-metals grade acids are appropriate. It is important to match the acid concentration in standards and samples. Concentrations of antimony and silver between 50-500 ug/L require 1% (v/v) HCl for stability; for concentrations above 500 ug/L additional HCl will be needed. For this reason, it is recommended that antimony and silver concentrations in samples and standards be maintained below 500 ppb wherever possible. Acids are received in poly-coated glass bottles, and are stored under the hood in the Metals sample preparation laboratory at room temperature until use. All acids are considered to have a shelf life of three years from date of receipt unless otherwise indicated by the vendor. Refer to the current revision of Katahdin SOP CA-105, Reagent and Solvent Handling, for further details.

5.2 Reagent water, trace metals free, equivalent to ASTM Type 1 (ASTM D 1193), >18 Megohm/centimeter resistivity. Polyethylene carboys are filled with reagent water in the glassware prep room and are transported to the laboratory for use. Reagent water is replaced at least weekly.

5.3 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals, and supplied by the vendors with certificates of purity and analysis. Refer to Tables 4 and 5 for a listing of stock standards required, and to Table 8 for element concentrations in stock standards. Purchased stock standards are received in polyethylene containers and are stored in their original containers at room temperature in the Metals Standards Preparation Laboratory. All purchased stock standards are given an expiration date as indicated by the manufacturer. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.4 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 5 for a listing of intermediate standards required and for preparation instructions. Refer to Table 7 for element concentrations in intermediate standards. Intermediate standards are stored at room temperature in acid-washed polyethylene containers in the Metals Standards Preparation Laboratory. Intermediate standards are assigned an expiration date of three months from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.5 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument, to provide internal standardization through on-line addition, and to perform all necessary QC checks. Refer to Table 4 for a listing of working standards and for preparation instructions. Refer to Table 6 for element concentrations in working standards. Working standards are stored at room temperature in acid-washed polyethylene containers in the Metals Standards Preparation Laboratory. All working standards except the ICSA and ICSAB solutions are assigned an expiration date of three months from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. The ICSA and ICSAB solutions are assigned an expiration date of one week from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.6 Blanks: Three types of blanks are required for the analysis. The calibration blank is used in establishing the calibration curve. The preparation blank is used to monitor for possible contamination resulting from the sample preparation procedure. The rinse blank is used to flush the system between all samples and standards.

5.6.1 The calibration blank consists of the same concentrations of the same acid(s) used to prepare the final dilution of the analyte calibration solutions (currently 1% HNO_3 and 0.5% HCl, v/v, in laboratory reagent grade water). Use of HCl for antimony and silver is cited in Section 5.1.

5.6.2 The preparation blank must be carried through the complete preparation procedure and contain the same volumes of reagents as the associated digested sample solutions.

5.6.3 The rinse blank consists of 4% HNO_3 and 0.5% HCl,v/v, in reagent water. Prepare a sufficient quantity to flush the system between standards and samples.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP-MS should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months

[1] P = polyethylene or , G = glass

7.0 PROCEDURES

7.1 Instrument control and data acquisition are completely automated through the use of the Agilent Chemstation software, running in Windows NT. The main Chemstation screen is accessed by double-clicking the "ICP-MS Top" icon on the Windows desktop. Autosampler tables are edited by double clicking the "Edit Sample Log Table" icon on the Windows desktop. In the following discussion, software menu items that are to be selected are printed in boldface. The instrument operating conditions, acquisition parameters, acquisition masses, and internal standards for analysis USEPA Method 6020 are detailed in Table 10.

7.2 Turn on the argon supply at the tank and set the pressure to >700 kPa.

7.3 Turn on the water chiller/recirculator.

7.4 Verify that the exhaust hood is in operation.

7.5 Ensure that the internal standard solution bottle is adequately full. Consumption is approximately 2.5 mL/hour.

7.6 Verify that the rinse station reservoir has an adequate supply of reagent water.

7.7 Verify that the drain reservoir has adequate room to accept the day's drain waste. Empty the reservoir as necessary into an appropriate waste container (Waste Stream A) located in the Hazardous Waste Storage Area.

7.8 Inspect the peristaltic pump tubes for signs of flattening and wear, and replace them as necessary. Clamp the peristaltic pump tubes into the peristaltic pump.

7.9 Open the Chemstation software by double-clicking the "ICP-MS Top" icon. Initiate the plasma by selecting **Instrument>>Instrument Control>>Plasma>>Plasma On** and allow the instrument to aspirate calibration blank solution for at least 15 minutes. During this warm-up, select **Tune>>Sensitivity>>Start** to start the instrument scanning the mass range. Verify that the flow of sample and internal standard solutions through the uptake lines and into the nebulizer is free from pulsations by introducing an air bubble into each line and observing its progress. Adjust the pump clamp tension on each line to obtain a constant, pulse-free flow.

7.10 After the 15 minute warm-up, check the responses of masses 82 and 83 to insure minimal krypton intereference with selenium. Mass 83 response should be < 2000 counts per second. Then aspirate the Instrument Tune Solution (10 ppb Li, Y, Ce, Tl) and check the responses and RSDs at masses 7, 89, and 205.

7.11 Generate a tune report by selecting **Tune>>File>>Generate Report**. Evaluate the tune report against the tune specifications listed in Table 11. If the tune passes all specifications, proceed to step 7.14.

7.12 If the tune report indicates unacceptable instrument performance for any parameter, initiate an autotune by selecting **Tune>>Autotune>>Run**. The Chemstation software will attempt to tune the instrument to meet the tune specifications, and will generate a new tune report after autotuning. Evaluate the new tune report against the tune specifications listed in Table 11.

7.13 Repeat step 7.12 until all tune specifications have been met. File the final tune report.

7.14 Aspirate a 100 ppb solution of all analytes and run a P/A autotune by selecting **Tune>>Tune>>P/A Factor>>Run**. This will calibrate the pulse and analog modes of the detector. File the P/A report with the Tune report.

7.15 Load the instrument analytical method and calibrations table for USEPA Method 6020 into memory by selecting **Methods>>Load>>K6020.M>>K6020.C**.

7.16 Edit the sequence template "K6020.S" to create an analytical sequence table listing all of the samples to be analyzed. To do this, double click the "Edit Sample Log Table" icon on the Windows desktop. Select **Open>>K6020.S**. Click the drop-down menu at the top left of the screen, and select the **Sample** block. Fill in the sample table with sample IDs, vial numbers, analytical method (K6020.M for all samples), dilution factors, and failure actions. When the sample table is complete, click the drop-down menu at the top left of the screen, and select **Whole List** to view the entire analytical sequence, including all calibration standards and QC samples. Select **Print** to print this table. Save the sample log table under a new name by selecting **Save** and then typing the name. Close the "Edit Sample Log Table" window.

7.17 Load the autosampler racks according to the analytical sequence printout.

7.18 Select **Sequence>>Load and Run Sequence**, and select the appropriate autosampler sequence table from the displayed list. Enter the analyst's initials in the Operator box. Change data file name to appropriate designation. The protocol for naming data files is as follows: the 1st character is a letter that identifies the instrument ("F" for the Agilent 75000 ICP-MS), the 2nd character is a letter that identifies the year of the run ("S" for 2002, "T" for 2003, etc.), the 3rd character is a letter that identifies the month of the run ("A" for January, "B" for February, etc.), the 4th and 5th characters are numbers that identify the date of the run ("01" for the first day of the month, etc.), and the 6th character is a letter that sequentially identifies the run ("A" for the first run of the day on that instrument, "B" for the second run, etc.). For example, the run identified as "FSA16A" is the first run of the day that was performed on January 16, 2002, using the Agilent 7500 ICP-MS. Select **Run**. The instrument will analyze all samples in the order listed in the table. Analysis must proceed in the sequence described in Table 9 to ensure that all necessary quality control samples are analyzed at the appropriate frequencies. A minimum of three replicate scans is required for all standards and samples. Analysis always begins with the analysis of a calibration blank followed by at least three multielement calibration standards to calibrate the instrument. The system is flushed with rinse blank between each sample and standard, and each sample and standard is aspirated for at least one minute prior to the beginning of mass scanning.

7.19 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.20 A practical quantitation limit standard (PQL) is analyzed at the beginning of the run to verify calibration accuracy at the reporting limit. Refer to Section 8 and Table 1 for additional information.

7.21 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples, and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.22 Interference check standard solutions ICS-A and ICS-AB must be analyzed at the beginning of each run and every 12 hours to verify the adequacy of interference corrections. Refer to Section 8 and Table 1 for additional information.

7.23 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a calibration verification sample (ICV, ICB, CCV, or CCB) for that element must not be reported, except as noted in Sections 8.5, 8.6, and 8.9. The sample must be reanalyzed for the element in question.

7.24 All samples that exceed the linear dynamic range must be diluted and reanalyzed.

7.25 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is accomplished automatically by entering the dilution factor in the sample log table prior to initiation of analysis.

7.26 If an element has more than one monitored isotope, examination of the concentration calculated for each isotope, or the isotope ratios, will provide useful information for the analyst in detecting a possible spectral interference. Consideration should therefore be given to both primary and secondary isotopes in the evaluation of the element concentration. In some cases, secondary isotopes may be less sensitive or more prone to interferences than the primary recommended isotopes, therefore differences between the results do not necessarily indicate a problem with data calculated for the primary isotopes. In the case of Pb, quantitation is based on the sum of isotopes 206, 207 and 208 to compensate for any variation in naturally occurring isotope ratios. This is accomplished through the use of the interference correction equation for lead.

7.27 Calculations, aqueous samples: Final element concentrations for aqueous samples are reported in units of micrograms per liter (ug/L). The reported concentrations are calculated from measured digestate concentrations as follows:

$$\text{Concentration (ug/L)} = \frac{MC \times DF \times FV}{IV}$$

where:

MC = Measured digestate concentration (ug/L)
DF = Instrument dilution factor

FV = Final digestate volume (L)
IV = Digested sample volume (L)

7.28 Calculations, solid samples: Final element concentrations for solid samples are reported in units of milligrams per kilogram (mg/kg) on a dry weight basis. The reported concentrations are calculated from measured digestate concentrations as follows:

$$\text{Concentration (mg/kg dry weight)} = \frac{MC \times DF \times FV \times 100}{W \times S}$$

where:

MC = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Final digestate volume (L)
W = Weight of digested wet sample (g)
S = Percent solids

DATA REDUCTION AND REPORTING

7.29 When the analytical run is completed, the analyst should print a run summary and create a data import file.

7.30 Follow these steps to print the run summary: Open the FileViewNT program using the "FIVIEWNT" icon on the Windows Desktop. Select the data file of interest and move the required samples into the "Process List". Make sure the "Corrected Data" box is checked. Click the "Process" button. The data will be displayed in a spreadsheet format.

7.31 Select "Configure" from the top menu and "Sublists" from the displayed options. Select "Load Sublist" and then select "K6020" from this list of options. Make sure the "Enable Sublist" box is checked. Click the "OK" button. This will display only the analyte masses in the spreadsheet.

7.32 Select "Quant Info" from the top menu and "Quant Results" from the displayed options. This will display the data in concentration units. Click on the cell in the top left corner of the spreadsheet to highlight all data. Select "Tools" from the top menu and "Import Data into Spreadsheet Application" from the displayed options. Click the "Save" button. The data is transferred to a Microsoft Excel spreadsheet. Minimize this window and return to the FileView spreadsheet.

7.33 Select "Configure" from the top menu and "Sublists" from the displayed options. Select "Load Sublist" and then select "INTSTDS" from this list of options. Make sure the "Enable Sublist" box is checked. Click the "OK" button. This will display only the internal standard masses in the spreadsheet.

7.34 Select "Count Info" from the top menu and "Counts/sec" from this list of options. This will display the data in counts per second units. Click on the cell in the top left

corner of the spreadsheet to highlight all data. Select "Tools" from the top menu and "Import Data into Spreadsheet Application" from the displayed options. Click the "Save" button. The data is transferred to a Microsoft Excel spreadsheet. Copy the internal standard names and results and paste into the analyte results Excel spreadsheet.

7.35 In a separate cell of the Excel spreadsheet, input a formula to calculate the percent recovery of the internal standard relative to that of the calibration blank for each internal standard measurement of each sample.

7.36 Input a formula into the sample results cells to correct the data for dilution.

7.37 Format, print and save (as FileName.XLS, e.g. FSA28A.XLS) this run summary.

7.38 Follow these steps to create the data import file: Open the FileViewNT program using the "FIVIEWNT" icon on the Windows Desktop. Select the data file of interest and move the required samples into the "Process List". Make sure the "Corrected Data" box is checked. Click the "Process" button. The data will be displayed in a spreadsheet format.

7.39 Select "Configure" from the top menu and "Sublists" from the displayed options. Select "Load Sublist" and then select "K6020" from this list of options. Make sure the "Enable Sublist" box is checked. Click the "OK" button. This will display only the analyte masses in the spreadsheet.

7.40 Select "Quant Info" from the top menu and "Quant Results" from the displayed options. This will display the data in concentration units.

7.41 Select the "Transpose" from the menu.

7.42 Select "Tools" from the top menu and "Copy Selected Data to CSV File" from this list of options. Set the name the file as "FileName.CSV", e.g., "FSA28A.CSV". Click the "Save" button.

To import data into the Metals Database:

7.43 Select the "ICPMS Import" icon from the Windows Desktop, the ICPMS Import window will appear. Enter the datafile name without extension, (e.g., "FSA28A") and click the "Import" button.

7.44 When the "Import finished" message appears, close the ICPMS Import window and select the "KIMS_METALS" icon from the Windows Desktop. The Metals Database Main Menu will appear.

7.45 Select the "Reporting Menu" button. From the Reporting Menu screen select the Batch QC Menu button and then the "Calculate Batch QC" button.

7.46 From the resulting list of QC results, deselect any items that fail run QC or do not need ICP-MS analysis. Click on the "Accept Selected Batch QC" button.

7.47 From the Metals Main Menu, select the "Additional Data Handling" button. The Data Menu will appear. Select the "Report Added Compounds" button.

7.48 From the resulting list of sample results, deselect any items that fail run QC or do not need ICP-MS analysis. Click on the "Accept Data" button.

7.49 Once all associated data from an analysis run have been processed, go to the Generate Coverage portion of the Export Menu and print the Run Log, Valid Data Report and Logbook Page for the analysis run.

7.50 Combine these reports with the raw data printout and the Run Info Sheet and scan into a PDF format. Save in the "ICPMS DATA" section of the "METPDF" directory on the IMAGESERVER.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6020 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

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

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of a reagent blank, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed by each method on each instrument. This determination requires at least seven replicate digestions and analyses of 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.

ANALYTICAL RUN QC SAMPLES

8.3 Initial instrument calibration: The instrument is calibrated by running a calibration blank and at least three multielement calibration standards. For each element, calibration is performed fitting a single order equation to the calibration data, as follows:

$$Y=aX + [Blank]$$

where:
Y = Concentration (ug/L)
X = Measured signal intensity (counts per second)
a = Slope of the calibration line
[Blank] = Measured signal intensity of the calibration blank

Fitting the calibration equation through the measured intensity of the calibration blank, rather than through the y-intercept of the line, provides the best calibration accuracy at the low end of the calibration range. When this equation is used, however, the Agilent software does not calculate a calibration coefficient. For this reason, calibration accuracy at the high end of the calibration range is checked by reanalyzing the highest calibration standard as a sample immediately after instrument calibration. Recoveries for all elements must be within 90% to 110% of the true value in the high calibration standard. If the high calibration standard fails, result for the failing elements may not be reported from the run until the problem is corrected and a passing high calibration standard has been analyzed. Calibration accuracy in the middle of the calibration range is verified by analysis of the CCV solution (see Section 8.6 below). Calibration accuracy at the reporting limit is verified by analysis of the PQL Check Standard (see Section 8.7 below).

8.4 The intensities of all internal standards must be monitored for every analysis. When the intensity of any internal standard fails to fall between 30 and 120 percent

of the intensity of that internal standard in the initial calibration standard, the following procedure is followed. The sample must be diluted fivefold (1+4) and reanalyzed with the addition of appropriate amounts of internal standards. This procedure must be repeated until the internal-standard intensities fall within the prescribed window. The intensity levels of the internal standards for the calibration blanks (ICB and CCBs) and calibration verification standards (ICV and CCVs) must agree within ± 20 percent of the intensity level of the internal standard of the original calibration solution. If they do not agree, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples.

8.5 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared by combining compatible elements from standard sources different than those of the calibration standards and at concentrations within the linear working range of the instrument. The results of the ICV must fall within 90% to 110% of the expected values. If the ICV fails, result for the failing elements may not be reported from the run, unless the ICV recovery is greater than 110% and the sample result is less than the PQL.

8.6 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.

8.7 A Practical Quantitation Limit (PQL) Check Standard is analyzed at the beginning of each run, after the ICV and ICB samples. Element concentrations in this solution are one-fifth the laboratory's practical quantitation limit (assuming a 5-fold dilution of all digestates during analysis). Element recoveries for the PQL Check Standard must fall within 80% to 120% of the expected values. If the PQL Check Standard fails, results for the failing elements may not be reported from the run, unless the PQL Check Standard recovery is greater than 120% and the sample results is less than the PQL.

8.8 A calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the applicable reporting limit (or PQL) for each element. The reporting limit should be determined on a project specific basis, taking into account the data quality objectives for the project. This information must be communicated through a project QAPP and through the Katahdin project manager. When no project specific reporting limit is specified, the laboratory PQL shall be used. Some project specific limits may require reporting down to the MDL or IDL and taking corrective action based on these levels. Results that fall between the PQL and the IDL or MDL must always be flagged as "estimated" with a "J".

8.9 If an ICB or a CCB fails the acceptance criteria of less than the reporting limit, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for an ICB or CCB is greater than the PQL (or reporting limit), sample results that are less than the PQL (or reporting limit) or that are greater than or equal to ten times the measured ICB or CCB concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

8.10 To obtain analyte data of known quality, it is necessary to measure more than the analytes of interest in order to apply corrections or to determine whether interference corrections are necessary. If the concentrations of interference sources (such as C, Cl, Mo, Zr, W) are such that, at the correction factor, the analyte is less than the limit of quantitation and the concentration of interferents are insignificant, then the data may go uncorrected. Note that monitoring the interference sources does not necessarily require monitoring the interferent itself, but that a molecular species may be monitored to indicate the presence of the interferent. When correction equations are used, all QC criteria must also be met. Extensive QC for interference corrections are required at all times. The monitored masses must include those elements whose hydrogen, oxygen, hydroxyl, chlorine, nitrogen, carbon and sulfur molecular ions could impact the analytes of interest. Interference check solutions ICS-A and ICS-AB are analyzed at the beginning of each run and at least every 12 hours during the run to verify the effectiveness of interference corrections. Solution ICS-A contains high concentrations of interferents (Al, Ca, Fe, Mg, Na, P, K, S, C, Cl, Mo, and Ti) only. The measured apparent concentrations of other elements in this solution should be very low, indicating that interfering mass correction equations are adequate. Solution ICS-AB contains interferents at the same high concentrations, and several analytes (As, Cd, Cr, Co, Cu, Mn, Ni, Ag, and Zn) at concentrations of 20 ug/L. Measured recoveries for these analytes should approach 100%. No acceptance criteria for analyses of solutions ICS-A and ICS-AB have yet been established.

PREPARATION BATCH QC SAMPLES

Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spiked sample, or matrix spiked sample duplicate.

8.11 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) (or project specific reporting limit, if applicable) for each element. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is

greater than the PQL or reporting limit, associated sample results that are less than the PQL or reporting limit or that are greater than or equal to ten times the measured preparation blank concentration may be reported.

8.12 A laboratory control sample (LCSW, LCSO, or LCSS), consisting of spiked reagent water or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the recovery of a laboratory control sample is greater than 120%, associated sample results that are less than the PQL or reporting limit may be reported.

SAMPLE MATRIX QC SAMPLES

8.13 The relative percent difference (RPD) between matrix duplicate, matrix spike duplicate , or laboratory control duplicate sample results is calculated as follows:

$$\text{RPD (\%)} = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where:

D_1 = First sample or LCS result
D_2= Second (duplicate) sample or LCS result

A control limit of 20% RPD is applied to duplicate analysis, if the result is greater than 100 times the instrument detection limit. If the matrix duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.14 The recovery for each element in a spiked sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If a recovery fails, the associated sample result must be flagged on the report of analysis. The spike recovery should be calculated as follows:

$$\text{Recovery (\%)} = \frac{S-U}{SA} * 100\%$$

where:

S = Measured concentration of spiked aliquot
U = Measured concentration of unspiked aliquot
SA = Amount of spike added

8.15 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL), the measured concentration of a five-fold dilution (1:5 dilution) of the sample should

agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$\text{Difference (\%)} = \frac{|L-S|}{S} * 100\%$$

where:

L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

8.16 An analyte spike that is added to an aliquot of a prepared sample, or its dilution, should be recovered within 75% to 125% of the known value if the result for the unspiked aliquot is less than four times the amount of spike added. If the post-digestion spike is not recovered within these limits, the sample should be diluted and reanalyzed to compensate for the matrix interference.

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 Metals 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 current revisions of USEPA Method 6020 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846 Update II, September 1994, Method 6020.

Agilent 7500 ICP-MS ChemStation Operator's Manual, Agilent Technologies, Inc., 2000.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

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TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6020	Initial Calibration, minimum 3 points plus a calibration blank.	Daily prior to sample analysis.		
	High Calibration Standard	Analyzed as a sample after calibration.	Recovery within ± 10% of true value.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	1) Do not use sample results for failing elements, unless ICV >110% and sample result < PQL/reporting limit.2) Investigate and correct problem.
	Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL or project specific reporting limit.	1) Do not use sample results if ≥ PQL/reporting limit and < 10x ICB level. 1) Investigate and correct problem.
	PQL Standard	At beginning of run	80%-120% of true value	Do not use results for failing elements
	Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within ± 10% of true value.	1) Do not use sample results for failing elements, unless CCV >110% and sample result < PQL/reporting limit. 2) Investigate and correct problem.
	Continuing Calibration Blank (CCB)	After every 10 samples and at end of the run.	Absolute value of CCB < PQL or project specific reporting limit.	2) Do not use sample results if ≥ PQL/reporting limit and < 10x CCB level. 3) Investigate and correct problem.
	Interference Check Solution A (ICS-A)	Before beginning a sample run, and every 12 hours during a run.	± PQL unless otherwise specified by client	None established.
	Interference Check Solution AB (ICS-AB)	Before beginning a sample run, and every 12 hours during a run.	± PQL unless otherwise specified by client	1) None established.

TABLE 1 (continued)

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6020	Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL (standard practice), or based on the project specific guidelines.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and <10x the blank conc.
	Laboratory Control Sample (LCSW/LCSS/LCSO)	At least one per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples, unless LCS >120% and sample result < PQL .
	Matrix Spike Sample (S)	See section 8.11	Recovery ± 25% of true value, if sample < 4x spike added.	Flag results.
	Duplicate Sample (D), Matrix Spike Duplicate (P), or LCS Duplicate (LC2W/LC2S/LC2O)	See section 8.11	1) RPD ≤ 20%, if sample > 100x IDL.	Flag results
	Post-Digestion Matrix Spike (A)	When serial dilution fails or analyte concentration for all samples < 100 x MDL.	Recovery ± 25% of true value, if sample < 4x spike added.	Flag results.
	Serial Dilution (L)	1 per digestion batch	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag result or dilute and reanalyze sample to eliminate interference.

TABLE 1 (continued)

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6020	Internal Standard (IS) Appropriate IS required for all analytes in all samples. Mass of IS must be <50 amu different from that of analyte.	1) For each sample, IS intensity within 30%-120% of that of intial calib. standard. 2) For ICV, ICB, CCV, and CCB, IS intensity within 80%-120% of that in initial calib. standard.	1) Repeat IDL study.	2) Raise PQL.
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < MDL PQL at least 2-3x IDL	1) Repeat IDL study. 2) Raise PQL.
	Method Detection Limit (MDL) Study	Annually.	PQL at least 2-3x MDL	1) Repeat MDL study. 2) Raise PQL.
	Method of Standard Additions	When matrix interference is suspected	NA	NA

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-627-03	METHOD 6020, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6020: ± PQL	Acceptance criteria stated in 6020: <3xIDL

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 3

ISOTOPES MONITORED AND CORRECTION EQUATIONS USED FOR USEPA METHOD 6020

Element Class	Element	Sym-bol	Isotopes Monitored	Correction Equations [See note 1]
Analytes	Aluminum	Al	27	
	Arsenic	As	75	^{75}As = (75)*1 - (77)*2.95 + (82)*2.548 – (83)*2.571 [See note 2]
	Barium	Ba	135, 137	
	Beryllium	Be	9	
	Cadmium	Cd	106, 108, 111, 114	^{111}Cd = (111)*1 – (108)*1.073 + (106)*0.764 [See note 3] ^{114}Cd = (114)*1 – (118)*0.0268 [See note 4]
	Chromium	Cr	52, 53	
	Cobalt	Co	59	
	Copper	Cu	63, 65	
	Lead	Pb	206, 207, 208	^{208}Pb = (208)*1 + (206)*1 + (207)*1 [See note 5]
	Manganese	Mn	55	
	Nickel	Ni	60, 61	
	Silver	Ag	107, 109	
	Thallium	Tl	203, 205	
	Zinc	Zn	66, 67, 68	
Internal Stan-dards.	Bismuth	Bi	209	
	Germanium	Ge	72	
	Indium	In	115	^{115}In = (115)*1 – (118)*0.016 [See note 6]
	Lithium	Li	6	
	Scandium	Sc	45	
	Terbium	Tb	159	
	Yttrium	Y	89	
Inform-ation Only	Antimony	Sb	121, 123	
	Boron	B	11	
	Calcium	Ca	44	^{44}Ca = (44)*1 – (88)*0.0169 [See note 7]
	Iron	Fe	54, 56, 57	^{54}Fe = (54)*1 – (52)*0.0282 [See note 8] ^{57}Fe = (57)*1 – (43)*0.03 [See note 9]
	Magnesium	Mg	25	
	Molybdenum	Mo	98	^{98}Mo = (98)*1 – (99)*0.146 [See note 10]
	Potassium	K	39	
	Selenium	Se	82	^{82}Se = (82)*1 – (83)*1.009 [See note 11]
	Sodium	Na	23	
	Strontium	Sr	88	
	Thorium	Th	232	

TABLE 3 (continued)

ISOTOPES MONITORED AND CORRECTION EQUATIONS USED FOR USEPA METHOD 6020

Element Class	Element	Sym -bol	Isotopes Monitored	Correction Equations [See note 1]
Inform-ation Only	Tin	Sn	118, 120	
	Titanium	Ti	47, 49	
	Tungsten	W	182	
	Uranium	U	238	
	Vanadium	V	51	$^{51}V = (51)*1 - (53)*2.95 + (52)*0.333$ [See note 12]

Notes:
1) Numbers in parentheses, e.g "(51)", indicate measured counts at the indicated mass.
2) Corrects for ArCl interference, taking into account secondary interferences from Se and Kr
3) Corrects for MoO interference, taking into account secondary interference from ^{108}Cd
4) Corrects for Sn interference
5) Corrects for variations in isotopic composition of lead
6) Corrects for Sn interference
7) Corrects for interference from $^{88}Sr^{2+}$
8) Corrects for Cr interference
9) Corrects for Ca interference
10) Corrects for Ru interference
11) Corrects for Kr interference
12) Corrects for ClO, taking into account secondary interference from Cr

TABLE 4

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Calibration Standard 1 (S1) (1.0% HNO_3 / 0.5% HCl)	MS-MIX-Z	Lab Prepared	0.010
	Instrument Calibration Solution 1	Spex Industries	0.0025
	Instrument Calibration Solution 2	Spex Industries	0.005
Calibration Standard 2 (S2) (1.0% HNO_3 / 0.5% HCl)	MS-MIX-Z	Lab Prepared	0.10
	Instrument Calibration Solution 1	Spex Industries	0.025
	Instrument Calibration Solution 2	Spex Industries	0.050
Calibration Standard 3 (S3) / CCV (1.0% HNO_3 / 0.5% HCl)	MS-MIX-Z	Lab Prepared	0.50
	Instrument Calibration Solution 1	Spex Industries	0.125
	Instrument Calibration Solution 2	Spex Industries	0.25
Calibration Standard 4 (S4) / High Standard (1.0% HNO_3 / 0.5% HCl)	MS-MIX-Z	Lab Prepared	1.0
	Instrument Calibration Solution 1	Spex Industries	0.25
	Instrument Calibration Solution 2	Spex Industries	0.50
Initial Calibration Verification (ICV) (1.0% HNO_3 / 0.5% HCl)	CL-ICS-1,CL-ICS-4, CL-ICS-5	Spex Industries	0.20 of each
	CL-ICS-3	Spex Industries	2.0
	1000 mg/L Si	Inorganic Ventures	0.040
	1000 mg/L Al	Inorganic Ventures	0.038
	1000 mg/L B, W	Inorganic Ventures	0.002 of each

TABLE 4 (Cont'd)

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)
Practical Quantitation Limit Solution (PQL) (1.0% HNO_3 / 0.5% HCl)	ICP-MS PQL Intermediate 1	Lab Prepared	1.0
	ICP-MS PQL Intermediate 2	Lab Prepared	0.1
Interference Check Solution A (ICS-A) (1.0% HNO_3 / 0.5% HCl)	6020ICS-0A	Inorganic Ventures	10.0
Interference Check Solution AB (ICS-AB) (1.0% HNO_3 / 0.5% HCl)	6020ICS-0A	Inorganic Ventures	10.0
	6020ICS-0B	Inorganic Ventures	1.0
	ICP-MS ICSAB Intermediate	Lab Prepared	1.0
P/A Tuning Solution (1.0% HNO_3 / 0.5% HCl)	CL-CAL-1	Spex Industries	1.0 of each
Instrument Tuning Solution (1.0% HNO_3 / 0.5% HCl)	ICP-MS-TS-2	High Purity Standards	0.10
Internal Standard Solution (5.0% HNO_3 / 0.5% HCl)	Internal Standard Mix	Spex Industries	10
Method Tuning Solution (1.0% HNO_3 / 0.5% HCl)	ICP-MS Method Tune Intermediate	Lab Prepared	1.0

TABLE 5

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
ICP-MS PQL Intermediate 1 (1.0% HNO_3 / 0.5% HCl)	1000 mg/L As, Se	High Purity Standards	0.01 of each
	1000 mg/L Zn 10,000 mg/L Ca, Fe, Mg	High Purity Standards	0.02 of each
	10,000 mg/L Si	High Purity Standards	0.04
	10,000 mg/L Al	High Purity Standards	0.06
	1000 mg/L B	High Purity Standards	0.08
	10,000 mg/L K, Na	High Purity Standards	0.20 of each
	CL-ICS-4	Spex Industries	2.0
ICP-MS PQL Intermediate 2 (1.0% HNO_3 / 0.5% HCl)	1000 mg/L Sb, Ba, Be, Cd, Co, Cu, Pb, Mn, Ni, Ag, Sr, Sn	High Purity Standards or Inorganic Ventures	0.02 of each
	1000 mg/L Tl	High Purity Standards	0.04
	1000 mg/L Cr	High Purity Standards	0.06
	1000 mg/L Mo	High Purity Standards	0.08
	1000 mg/L V, W	High Purity Standards or Inorganic Ventures	0.1 of each
ICP-MS Method Tune Intermediate (1.0% HNO_3 / 0.5% HCl)	1000 mg/L Be, Co, In, Pb, Li, Tl 10,000 mg/L Mg	High Purity Standards or Inorganic Ventures	1.0 of each
MS-MIX-Z (1.0% HNO_3 / 0.5% HCl)	10,000 mg/L Ca, Fe, Mg, K, Na	High Purity Standards or Inorganic Ventures	5.0 of each
	10,000 mg/L Si	High Purity Standards	1.0
	10,000 mg/L Al	High Purity Standards	0.95
	1000 mg/L B, Sr, Sn, Ti, W	High Purity Standards	0.50 of each
ICP-MS ICSAB Intermediate (1.0% HNO_3 / 0.5% HCl)	CL-ICS-4	Spex	20.0
	10,000 mg/L Si	High Purity	0.50
	1,000 mg/L B, Ba, Be, Pb, Sb, Se, Sn, Sr, Tl, W, V	High Purity or Inorganic Ventures	0.20 each

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 6

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L						
	S1	S2	S3/CCV	S4	ICV	PQL	P/A Tune Soln.
Aluminum	10.0	100.0	500.0	1000.0	400.0	60.0	200
Antimony	0.5	5.0	25.0	50.0	20.0	0.2	200
Arsenic	0.5	5.0	25.0	50.0	20.0	1.0	200
Barium	0.5	5.0	25.0	50.0	20.0	0.2	200
Beryllium	0.5	5.0	25.0	50.0	20.0	0.2	200
Boron	0.5	5.0	25.0	50.0	20.0	8.0	
Cadmium	0.5	5.0	25.0	50.0	20.0	0.2	200
Calcium	100.0	1000.0	5000.0	10000.0	4000.0	20.0	
Chromium	0.5	5.0	25.0	50.0	20.0	0.6	200
Cobalt	0.5	5.0	25.0	50.0	20.0	0.2	200
Copper	0.5	5.0	25.0	50.0	20.0	0.2	200
Iron	100.0	1000.0	5000.0	10000.0	4000.0	20.0	
Lead	0.5	5.0	25.0	50.0	20.0	0.2	200
Magnesium	100.0	1000.0	5000.0	10000.0	4000.0	20.0	
Manganese	0.5	5.0	25.0	50.0	20.0	0.2	200
Molybdenum	0.5	5.0	25.0	50.0	40.0	1.0	200
Nickel	0.5	5.0	25.0	50.0	20.0	0.2	200
Potassium	100.0	1000.0	5000.0	10000.0	4000.0	20.0	
Selenium	0.5	5.0	25.0	50.0	20.0	1.0	200
Silicon	10.0	100.0	500.0	1000.0	400.0	40.0	
Silver	0.5	5.0	25.0	50.0	20.0	0.2	200
Sodium	100.0	1000.0	5000.0	10000.0	4000.0	20.0	
Strontium	0.5	5.0	25.0	50.0	20.0	0.2	
Thallium	0.5	5.0	25.0	50.0	20.0	0.4	200
Thorium	0.5	5.0	25.0	50.0	20.0	0.2	200
Tin	0.5	5.0	25.0	50.0	20.0	0.2	
Tungsten	0.5	5.0	25.0	50.0	20.0	1.0	
Uranium	0.5	5.0	25.0	50.0	20.0	0.2	200
Vanadium	0.5	5.0	25.0	50.0	20.0	1.0	200
Zinc	0.5	5.0	25.0	50.0	20.0	2.0	200

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 6 (continued)

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L			
	ICSA[1]	ICSAB[1]	Internal Std Solution	Instrument Tuning Solution
Aluminum	100000	100000		
Antimony		20		
Arsenic		20		
Beryllium		20		
Cadmium		20		
Calcium	100000	100000		
Chromium		20		
Cobalt		20		
Copper		20		
Iron	100000	100000		
Lead		20		
Magnesium	100000	100000		
Manganese		20		
Molybdenum	2000	2000		
Nickel		20		
Potassium	100000	100000		
Selenium		20		
Silver		20		
Sodium	100000	100000		
Thallium		20		10.0
Vanadium		20		
Zinc		20		
Uranium		20		
Barium		20		
Strontium		20		
Tin		20		
Tungsten		20		
Boron		20		
Thorium		20		
Bismuth			1000.0	
Germanium			1000.0	
Indium			1000.0	
Lithium (^{6}Li)			1000.0	
Scandium			1000.0	
Terbium			1000.0	

TABLE 6 (continued)

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Yttrium			1000.0	10.0
Cerium				10.0
Lithium(^{7}Li)				10.0

1) Solution also contains 1000 mg/L Chloride, 200 mg/L Carbon, and 100 mg/L Phosphorus and Sulfur, and 2mg/L Titanium.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 7

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

ELEMENT	CONCENTRATION IN SOLUTION, mg/L			
	MS-MIX-Z	ICP-MS PQL Intermediate 1	ICP-MS PQL Intermediate 2	ICP-MS Method Tune Intermediate
Aluminum	95.0	6.0		
Antimony			0.20	
Arsenic		0.10		
Barium			0.20	
Beryllium			0.20	10.0
Boron	5.0	0.8		
Cadmium			0.20	
Calcium	500	2.0		
Chromium			0.60	
Cobalt			0.20	10.0
Copper			0.20	
Indium				10.0
Iron	500	2.0		
Lead			0.20	10.0
Lithium				10.0
Magnesium	500	2.0		10.0
Manganese			0.20	
Molybdenum		0.02	0.80	
Nickel			0.20	
Potassium	500	20.0		
Selenium		0.10		
Silicon	100	4.0		
Silver			0.20	
Sodium	500	20.0		
Strontium	5.0		0.20	
Thallium			0.40	10.0
Thorium		0.020		
Tin	5.0		0.20	
Tungsten	5.0		1.0	
Uranium		0.020		
Vanadium			1.0	
Zinc		0.20		

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 8

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L					
	Instrument Calibration Standard 1 (Spex)	Instrument Calibration Standard 2 (Spex)	CL-ICS-1 (Spex)	CL-ICS-3 (Spex)	CL-ICS-4 (Spex)	CL-ICS-5 (Spex)
Aluminum	20.0		10.0			
Antimony	20.0		10.0			
Arsenic	20.0		10.0			
Barium	20.0		10.0			
Beryllium	20.0		10.0			
Boron						
Cadmium	20.0		10.0			
Calcium		1000		200.0		
Chromium	20.0		10.0			
Cobalt	20.0		10.0			
Copper	20.0		10.0			
Iron		1000		200.0		
Lead	20.0		10.0			
Magnesium		1000		200.0		
Manganese	20.0		10.0			
Molybdenum	20.0				10.0	10.0
Nickel	20.0		10.0			
Potassium		1000		200.0		
Selenium	20.0		10.0			
Silver	20.0		10.0			
Sodium		1000		200.0		
Strontium						10.0
Thallium	20.0		10.0			
Thorium	20.0				10.0	
Tin						10.0
Tungsten						
Uranium	20.0				10.0	
Vanadium	20.0		10.0			
Zinc	20.0		10.0			

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 8 (continued)

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L		
	6020ICS-0A[1] (Inorganic Ventures)	**6020ICS-0B (Inorganic Ventures)**	**ICP-MS-TS-2 (High Purity)**
Aluminum	1000		
Arsenic		2.0	
Cadmium			
Calcium	1000		
Chromium		2.0	
Cobalt		2.0	
Copper		2.0	
Iron	1000		
Magnesium	1000		
Manganese		2.0	
Molybdenum	20.0		
Nickel		2.0	
Potassium	1000		
Silver		2.0	
Sodium	1000		
Zinc		2.0	
Cerium			10000
Lithium			10000
Thallium			10000
Yttrium			10000

1) Solution also contains 10000 mg/L Chloride, 2000 mg/L Carbon, and 1000 mg/L Phosphorus and Sulfur, and 20 mg/L Titanium.

TABLE 9

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Method Tuning Solution	Verify mass calibration and resolution
2	S0 (Calibration Blank)	Initial calibration
3	S1 (Calibration Standard)	Initial calibration
4	S2 (Calibration Standard)	Initial calibration
5	S3 (Calibration Standard)	Initial calibration
6	S4 (Calibration Standard)	Initial calibration
7	ICV (Initial Calibration Verification)	Check calibration accuracy
8	ICB (Initial Calibration Blank)	Check calibration accuracy
9	PQL (Practical Quantitation Limit)	Check calibration accuracy at low concentration
10	High Standard (Reanalysis of S4)	Check calibration accuracy at upper end of curve
11	ICS-A (Interference Check Solution A)	Verify accuracy of mass correction equations
12	ICS-AB (Interference Check Solution AB)	Verify accuracy of mass correction equations
13	Rinse Blank	Clear system of high levels of interferents
14	CCV (Continuing Calibration Verification)	Check calibration stability
15	CCB (Continuing Calibration Blank)	Check calibration stability
16-25	Analyze up to 10 samples	
26	CCV (Continuing Calibration Verification)	Check calibration stability
27	CCB (Continuing Calibration Blank)	Check calibration stability
...	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	
...	After last analytical sample, analyze ICS-A and ICS-AB, followed by a CCV and a CCB	

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 10

INSTRUMENT OPERATING CONDITIONS

Data Acquisition Program		Acquisition Mode	Spectrum
		Points per Mass	3
		Number of Replicates	3
		Detector Mode	Auto for all elements
		Integration Time per Point (for listed masses and their correction masses)	0.10 sec for Li, B, ^{29}Si, Sc, V, Cr, Mn, Ni, Cu, Zn, Y, Mo, Ag, In, Sn, Sb, Ba, Tb, W, Tl, Pb, Bi, Th, U
			0.30 sec for Be, As, Cd, Ge
			0.010 sec for Na, Al, K, ^{28}Si
			0.030 for Ca, Fe, Sr
			1.00 sec for Se
			0.050 sec for Mg, Co
		Spray Chamber Temperature	2° C
		Total Acquisition Time	111 sec for 3 replicates
Peristaltic Pump Program		Analysis Speed	0.12 rps
ISIS Pump Program	Before Acquisition	Uptake Speed	1.00 rps
		Uptake Time	50 sec
		Stabilization Time	65 sec
	After Acquisition (Probe Rinse)	Rinse Speed	1.00 rps
		Rinse Time (sample)	5 sec
		Rinse Time (standard)	5 sec
	After Acquisition (Rinse)	Rinse Vial	1
		Uptake Speed	1
		Uptake Time	60 sec
		Stabilization Time	55 sec
Calibration Curvefit		All quantitation masses	Y=ax+(blank)
		All internal standard masses	(Excluded)
		All interference correction masses	(Excluded)
Reporting Parameters		QC Reports	On-Printer
		All Other Reports	Off

TABLE 11

INSTRUMENT TUNE SPECIFICATIONS

Sensitivity	Li >5000 cts/0.1 sec/10 ppb
	Y >10,000 cts/0.1 sec/10 ppb
	Tl >5000 cts/0.1 sec/10 ppb
Precision	Li <15% RSD (0.1 sec integration time)
	Y <15% RSD (0.1 sec integration time)
	Tl <15% RSD (0.1 sec integration time)
Oxides	<1.0%
Doubly Charged (Ce^{++}/Ce^{+})	<5.0%
Background	Li <30 cps
	Y <15 cps
	Tl <15 cps
Mass Resolution	Width at 10% peak height: 0.7-0.8 amu
Mass Axis	Li ±0.1 amu of nominal mass
	Y ±0.1 amu of nominal mass
	Tl ±0.1 amu of nominal mass

TABLE 12

METHOD TUNE SPECIFICATIONS

Precision	≤5% RSD of 4 replicates
Mass Resolution	Width at 10% peak height: <0.9 amu
Mass Calibration	±0.1 amu of nominal mass

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-742
Revision History
Cover Page
Page 1

TITLE: **ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056**

Prepared By: Deborah McGrath Date: 05/00

Approved By:

Group Supervisor: Keith Tangley Date: 05/29/01

Operations Manager: [illegible] Date: 5/23/01

QA Officer: Deborah J. Nadeau Date: 5.22.01

General Manager: Duncan F. Lufkin Date: 5/24/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
02	Format changes, added pollution prevention, added SW846 method 9056 reference and requirements. Updated logbook attachments.	DN	5.22.01	
03	Added reference to KIMS data entry (Sect. 7.14). Replaced fig.s 2 & 5. Added wording to sect. 8. minor changes throughout to reflect current practices	LAD	031805	031805
04	Added information on Retention time Windows. Added reference for SOP CA 107 to Section 1.4	LAD	04/06	04/06
05	Sect. 5.0 - edited to reflect current Std. prep procedures. Sect. 7.0 - changed base range, added syringe pursing procedure, added info for setting up calibration in active sequence, added need for new sequence at beginning of every month	LAD	06/08	06/08
06	Minor revisions to sections 1.1 and 4 for clarity. Revisions to Section 8, 10, Table 2 and 3 to update from SW846 method 9056 to 9056A. Added LLQC criteria to Section 8.	LAD	02/09	02/09

TITLE: ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056

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

I acknowledge receipt of copy _____ of document **SOP CA-742-06,** titled **ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **SOP CA-742-06,** titled **ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056.**

Recipient: _____ Date: _____

1.0 SCOPE AND APPLICATION

This SOP describes the procedures used by Katahdin Analytical Services, Inc. technical personnel to determine the concentration of the following inorganic anions using ion chromatography (IC) by EPA Method 300.0 and SW-846 9056, current version: Sulfate, Bromide, Phosphate-P, Chloride, Nitrate-N, and Nitrite-N. This method may be used for the analysis of the following matrices: Drinking water, Surface water, Mixed Domestic and Industrial Waste waters, Ground water, Reagent waters, solids (after aqueous extraction), and Leachates (when no acetic acid is used).

1.1 Definitions

Analytical Batch – A group of 20 or fewer samples that are analyzed together on the same day.

Calibration Blank (CB) - A volume of laboratory reagent grade water fortified with the same matrix as the calibration standards but without the analytes. In most cases the CB will consist of laboratory reagent grade water.

Continuing Calibration Blank (CCB) – An aliquot of reagent water that is analyzed after each CCV to ensure continuing calibration accuracy.

Continuing Calibration Verification (CCV) – A midrange standard containing all method analytes that is run at the beginning of each run, after every 10 samples, and at the end of each run to ensure continuing calibration accuracy. The CCV is prepared from the same source as the calibration standards. The CCV is sometimes called the Instrument Performance Check Solution (IPC).

Laboratory Control Sample (LCS) / Initial Calibration Verification (ICV) – An aliquot of reagent water to which known amounts of the method analytes are added, and that is processed through the entire analytical procedure in the same manner as a sample. One LCS is processed and analyzed with each batch of 20 or fewer samples. The LCS/ICV is prepared from a different standard source than the calibration standards. LCSs provide a sample of known concentration to assess the accuracy of the analytical system, and when analyzed in duplicate may be used to a measure of precision for the analytical system. The LCS/ICV is sometimes called the Laboratory Fortified Blank (LFB).

Laboratory Duplicate – A duplicate is a second aliquot of a sample that is analyzed to to assess the precision of the analysis.

Matrix Spike (MS) – An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a samples, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample

matrix must be determined in a separate aliquot and the measured values in the MS corrected for the background concentrations. The MS is sometimes called the Laboratory Fortified Matrix (LFM).

Method Blank (MB) – An aliquot of reagent water that is carried through the entire analytical procedure in the same manner as a sample. One method blank is processed and analyzed with each batch of 20 or fewer samples. For the purposes of this analysis the MB water is filtered through a 0.45 μm filter.

Method Detection Limit (MDL) – The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of anions by IC using EPA Method 300.0 and SW-846 9056. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of anions by IC using EPA Method 300.0 or SW-846 9056 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab logbook. Any deviations from the test or irregularities with the samples should also be recorded in the lab logbook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate

personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

The outflow from the chromatograph, WISP needle wash, WISP waste and regenerate wash is collected in a single container and disposed of in a Satellite Waste "A" Acid for proper disposal in main waste area "A". Other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual and SOPs SD-903, "Sample Disposal" and CA-107, "The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents and Standards," current revisions. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

A small volume of sample is introduced into an ion chromatograph. The anions of interest are separated and measured using a system comprised of a guard column, analytical column, suppressor device and conductivity detector. An aqueous extraction procedure must be performed in order to utilize this method for solid matrices.

3.0 INTERFERENCES

3.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or spiking can be used to solve most interference problems. The most common source of interference is high chloride concentrations.

3.2 The water dip or negative peak that elutes near and can interfere with the chloride peak can usually be eliminated by the adding the equivalent of 1mL of eluent concentrate to 100 mL of each sample.

3.3 The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

3.4 Method interferences may be caused by contaminants in the water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in the ion chromatograph.

3.5 Samples that contain particles larger than 0.45 μ and reagent solutions that contain particles larger than 0.2 μm require filtration to prevent damage to instrument columns and flow systems.

3.6 Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other small organic anions. At concentrations of 1.5 mg/L fluoride this interference may not be significant.

4.0 APPARATUS AND MATERIALS

4.1 Ion chromatograph capable of delivering 1 to 5 mL of eluent per minute at a pressure of 1000 to 4000 psi. The chromatograph must be equipped with an injection valve, a 25- to 100-uL sample loop, and set up with the following components.

4.1.1 Autosampler – Waters Model 717plus or equivalent

4.1.2 Ion chromatography pump - Spectra System Model P4000 or equivalent

4.1.3 Conductivity detector – Waters Model 431 or equivalent

4.1.4 Precolumn – A guard column placed before the separator column to protect the separator column from fouling by particulates or organic constituents. Dionex Guard Cartridge AG4 or equivalent.

4.1.5 Separator column – A column packed with an anion exchange resin, suitable for resolving the anions of interest. Dionex Ionpac AS4A or equivalent.

4.1.6 Conductivity suppressor – An ion exchange-based device that is capable of converting the eluent and separated anions into their respective acid forms. Dionex Micromembrane Suppressor Model AMMS 300 or equivalent.

4.2 2 Liter regenerant reservoir

4.3 Analytical balance capable of accurately weighing to the nearest 0.0001g.

4.4 5 mL disposable syringes

4.5 Syringe filters - 45 μm Whatman Puradisc or equivalent

4.6 Autosampler rack with 1 mL sample vials and caps

4.7 Class "A" pipets, assorted volumes, Auto pipets or equivalent

4.8 Class "A" volumetric flasks, assorted volumes

4.9 Turbochrom Navigator software

4.10 Masterflex C/L regenerant pump or equivalent

5.0 REAGENTS

5.1 Regenerant, 0.025N H_2SO_4: Dilute 2.8 mL conc. H_2SO_4 to the 4 L with laboratory reagent grade water. Regenerant is made fresh on the day of the analysis.

5.2 Eluent, 1.7mM sodium bicarbonate, 1.8mM sodium carbonate: Dissolve 0.2856g sodium bicarbonate ($NaHCO_3$) and 0.3816g sodium carbonate (Na_2CO_3) in reagent water and dilute to 2 L. Vacuum filter through a 0.20 u or 0.45 u filter before use. Prepare fresh daily.

5.3 Laboratory reagent water

5.4 Needle wash solution: Reagent water containing 10% methanol by volume to inhibit bacterial growth.

5.5 Stock Standard Solutions: Stock standard solutions may be purchased as certified solutions or prepared from ACS reagent grade material. All purchased standards prepared from high purity salts and supplied by the vendors with certificates of purity and analysis. All purchased stock standards are given an expiration date as indicated by the manufacturer.

5.5.1 Bromide (Br^-), 1000 mg/L, purchased.

5.5.2 Chloride (Cl^-), 1000 mg/L, purchased

5.5.3 Nitrate (NO_3^--N), 225.8 mg/L as N, purchased

5.5.4 Nitrite (NO_2^--N), 304.4 mg/L as N, purchased

5.5.5 Phosphate (PO_4^{3-}-P), 326.3 mg/L as P, purchased

5.5.6 Sulfate ($SO_4^{=}$), 1000 mg/L, purchased

5.6 Primary Mixed Calibration Standard - Combine the following and dilute to 200 mL with laboratory reagent grade water:

Analyte	Amount of Stock Std. Added (mL)	Concen-tration in Stock Std. (mg/L)	Final Volume of Primary Mixed Cal. Std. (mL)	Final Conc. In Primary Mixed Cal. Std. (mg/L)
Cl^-	2.0	1000	200	10.0
NO_2^--N	2.63	304.4		4.0
NO_3^--N	3.54	225.8		4.0
Br^-	4.0	1000		20.0
$SO_4^{=}$	4.0	1000		20.0
$PO_4^{=}$-P	3.06	326.3		5.0

NOTE: At any time a stock may be prepared with an abbreviated list of analytes and should be clearly labeled with the list of analytes contained.

5.6 Primary Working Calibration Standards (WCS) – Dilute Primary Mixed Calibration Standard as follows to prepare the five-point level working calibration standards:

Work-ing Std. ID	Amount of Primary Mixed Cal. Std. Added (mL)	Final Volume of Working Cal. Std. (mL)	Analyte Conc. In Working Cal. Std. (mg/L)					
			Cl^-	NO_2^- as N	NO_3^- as N	Br^-	$SO_4^{=}$	$PO_4^{=}$ as P
IC1	1	1	10	4	4	20	20	5
IC2	0.5	1	5	2	2	10	10	2.5
IC3	0.25	1	2.5	1	1	5	5	1.25
IC4	0.1	1	1	0.4	0.4	2	2	0.5
IC5	0.01	1	.1	0.04	0.04	0.2	0.2	0.05

5.7 Primary Continuing Calibration Verification Standard (CCV1) – The CCV will be prepared using the same stock as the calibration standards and will be prepared at the same concentration as calibration standard IC2. Volumetrically pipet 25 mL Primary Mixed Calibration Stock Standard into 50 mL volumetric flask and bring to volume with reagent water.

5.8 Primary LCS Stock Standard – LCS Stock must be comprised of independent sources for all analytes relative to the calibration standard stock. Combine the following using purchased standards from second source and dilute to 200 mL with laboratory reagent grade water: 1.5 mL of standard solution "A" and 1.5 mL of standard solution "B".

Final Concentrations (mg/L):

Cl	NO2(as N)	NO3 (as N)	Br	SO4	PO4 (as P)
7.50	2.28	1.69	7.50	7.50	2.45

5.8.1 Standard Solution "A"- Multi-element standard which contains analytes in the following concentrations:

- Nitrate (as N) – 225.8 mg/L
- Bromide- 1000 mg/L
- Orthophosphate (as P)- 326.3 mg/L
- Chloride – 1000 mg/L
- Fluoride – 1000 mg/L
- Sulfate- 1000 mg/L

5.8.2 Standard Solution "B" – Contains Nitrite (as N) only in the concentration of 304.4 mg/L

5.9 Primary Matrix Spike Mixed Standard - The Primary Matrix Spike Mixed Standard is prepared from the same anion stock standards as the Primary LCS stock standard. The mixed standard is prepared as follows: 7.5 mL of standard solution "A" and 7.5 mL of standard solution "B" diluted to 100 mL with DI water. Adding 0.05 mL of the mixed standard to 1.0 mL of sample yields the following concentrations(mg/L):

Cl	NO2 (as N)	NO3 (as N)	Br	SO4	PO4 (as P)
3.75	1.14	.845	3.75	3.75	1.22

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples should be collected in clean glass or polyethylene bottles. Sample preservation and holding times for the anions that can be determined by this method are as follow.

ANALYTE	PRESERVATION	HOLDING TIME
Bromide	None required	28 days
Chloride	None required	28 days
Nitrate-N	Cool to 4°C	48 hrs
Nitrite-N	Cool to 4°C	48 hrs
Ortho-Phosphate-P	Cool to 4°C	48 hrs
Sulfate	Cool to 4°C	28 days

NOTE: Due to the potential of and persistence of chloride contamination and the associated short hold times for nitrite, nitrate and ortho-phosphate, it is recommended that these ions are run concurrently by alternate methods.

7.0 PROCEDURES

7.1 SAMPLE PREPARATION

7.1.1 Solid Samples Extraction – Add an amount of laboratory reagent grade water equal to ten times the weight of the dry sample. This slurry is mixed for ten minutes using a magnetic stirrer. The resulting slurry is allowed to stand or may be centrifuged prior to filtration through 0.45 µm filter. Hold times for analytes of interest should be considered to commence upon the generation of the extract.

7.1.2 All samples are filtered through 0.45 µm filter by means of a syringe with attached filter disk just prior to loading the auto sampler tray.

7.2 TURNING ON INSTRUMENT

7.2.1 The instrument is configured as illustrated in Figure 1.

7.2.2 Prepare sufficient volumes of fresh eluent and regenerant solutions.

NOTE: Both Eluent and Regenerant should be prepared DAILY.

7.2.3 Using the P4000 pump, press the run button on the front. Make sure the flow rate is set at 2.0 mL/min.

7.2.4 Using the peristaltic pump, crimp the tubing around the rollers by snapping the clamp up. Turn the pump on by pressing the black button down.

NOTE: The peristaltic tubing easily crimps shuts. Make sure regenerant is flowing, especially if the pump has been off for more than a few hours. You may have to replace the section of the tubing that runs through the pump head. Fittings are provided to easily allow for changing the crimped section of the tubing.

7.2.5 Monitor and record the backpressure on the HPLC pump. The pressure should not exceed 3500 psi. If this occurs it would indicate either you need to replace or clean the column, see column care and maintenance in the column manual.

7.3 TURNING ON THE CONDUCTIVITY DETECTOR

7.3.1 The Waters 431 conductivity detector is used for all of the anion determinations. If this detector does not pass QC criteria the system is out of control and maintenance must be initiated. Corrective action may include cleaning the injector with multiple purge cycles, blank injections, recalibration or more extensive column cleanup, replacement, or instrument maintenance.

7.3.1.1 The "*BASE RANGE* "should be set To 50 µS and the "*SENSITIVITY* " should be set to 0.05. The scale should be set to "*% FULL SCALE*" and the reading should be about 20 when the system has equilibrated. The reading can be adjusted using the up and down arrows on the keypad.

NOTE: It is important that this setting is positive. If the setting is at zero the baseline will be at 0V. Chromatography should be examined for indications of "*electrical zero*".

7.4 PURGING THE SYRINGE

7.4.1 The syringe should be purged daily. Make sure there is enough methanol in the bottle and that the green line is submerged in the methanol. Also make sure that the clear line and the yellow line are in the waste beaker.

7.4.2 To purge the syringe, select purge page from the main page. Click start purge. The syringe will proceed to purge for however long the time is set for.

7.5 CALIBRATION

7.5.1 A new calibration curve must be prepared at least one time every 6 months (check the runlog for the date of the last calibration) or if one of the following occurs:

7.5.1.1 The daily calibration verification is outside of the method criteria (Refer to section 8.0).

7.5.1.2 Major maintenance has been performed on the instrument (Refer to maintenance log, Figure 2).

NOTE: If one of the above is true, a new calibration curve must be prepared even if it has been less than 6 months since the last calibration.

7.5.2 If a calibration curve is required, prepare the standards as described in section 5.0.

7.5.3 Record the standards in the runlog (Figure 3) and load onto the autosampler. When entering the calibration points into the sample sequence, change the sample type to "cal: replace" and type the calibrator names in the level column. Make sure you go into the method first to save a new calibration file. This is done by opening an old file with the same method and saving over it with a new file name. In the active sequence, change the instrument, processing, and calibration methods to match the new curve file name.

7.5.4 The acceptance criteria for the calibration is a 0.995 correlation coefficient for the full curve. It is important to compare curves and responses vs. standard concentration with those previously generated to insure quality of data. Printouts of calibrations are filed in the Lab for approximately 3 months. Prior calibrations are archived. All are available in the database. Significant changes should be investigated. Switching to fresh standards or fresh reagents may change response slightly. Check historical data. Operator discretion in approving or rejecting calibrations is encouraged. The decision and rationale should be recorded in the logbook.

7.5.5 Retention time windows should be established when a new column is installed, if there is a change in instrument conditions or at least annually if there are no changes. Three injections of the CCV must be analyzed over the course of a 72 hour period. The standard deviation of the three retention times is calculated for each anion. Plus or minus three times the standard deviation of the retention times is used to define the retention time window. The retention time window should not extend beyond 5% of the retention time for each anion. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

7.6 LOADING SAMPLES

7.6.1 Select autosampler rack for 96 samples.

7.6.2 Write sequence in IC Run Log; follow page format and proper sample coding.

7.6.3 Filter all samples through 0.45 μm filters into the appropriate sample vials.

7.6.4 If high sample concentrations are suspected, steps should be taken to minimize reruns and protect the system from contamination and/or carryover. In general samples from potable sources, drinking water samples, may be

analyzed at an as received concentration. Monitoring wells, leachates, and estuarine samples may have extremely high concentrations of chloride and/or sulfate. To avoid contaminating the system, analyze these samples at an initial dilution. In the event that the analyzed aliquot does have higher concentrations, inject water samples after the sample to clean out the system. For highly concentrated samples it may take as many as 5 or more water injections to clean out the injector and remove the carryover.

7.6.5 Make sure that the autosampler and the TurboChrom software are set up and ready to acquire data. It is recommended that the tray is initially set up to run the opening QC before loading samples. Opening QC consists of a CCV, CCB, and LCS. Evaluate the opening QC to ensure adequate separation, good chromatography, acceptable recovery as it relates to the calibration, and clean blanks. If the opening QC is within criteria and the chromatographic system is in control proceed to load samples. If QC criteria are not met or chromatography is not acceptable initiate appropriate corrective action.

7.6.6 To automatically run tray:

- Place sample carriage in carriage drive.
- Press "*START AUTO BUTTON*".
- Enter your starting and ending vial locations.
- Make sure the injection volume is 20 and the run time is ten minutes.
- Press "START BUTTON" after performing section 7.7.

7.7 PREPARING THE TURBOCHROM NAVIGATOR

7.7.1 Click on "*MODIFY*" under "*ANALYSIS VIEW*"

7.7.2 Click on "*ACTIVE SEQUENCE*". This will take you to the last sequence used or the sequence currently running.

7.7.2.1 NOTE: At the start of each month, a new sequence must be started. This is done by clicking the sequence box, opening the last sequence file, and renaming the sequence to reflect the current month. Make sure all of the columns are changed to the correct information. Also rename the files by selecting change data file, rename file, and then clicking the folder icon. Select the W:\data\IC01 file and type in the basefile name to match the new sequence. Select Channel B and click all three boxes. Click save and exit out. Before running any samples for the new month, open the setup box and the sequence to the new sequence name.

7.7.3 Place cursor on the first empty cell.

7.7.4 Type in sample sequence of run under name, if samples are being diluted enter your dilution factor in the dilution column.

7.7.5 Go to "*FILE*" and select "*EXIT*".

7.7.6 At "*RESUME SEQUENCE- STARTING ROW*" enter the row number of the first row of the new sequence. "*RESUME SEQUENCE- ENDING ROW*" enter the last row of the new sequence.

7.7.7 Click "*OK*". At this point the status should say "*READY*" and the LINC box light should be green.

7.8 SHUT DOWN PROCEDURE – **It is CRITICAL to explicitly follow long term shutdown storage for columns to prevent damage.**

7.8.1 Turn off both the eluent and regenerant pumps

7.8.2 **If the system will not be used for more than a week it is critical to fill the columns with the appropriate storage solution and cap them to prevent evaporation. If a column dries out it is most likely useless.**

7.8.3 Flush the lines in the WISP and in the detectors with DI for storage.

7.8.4 **It is critical to fill the suppressor with fresh regenerant and cap it. Again if this dries out it is most likely useless.**

DATA ANALYSIS, CALCULATIONS & REPORTING

7.9 If a sample is run and the analyte of interest concentration is above the calibration range the sample must be diluted and reanalyzed. Multiple dilutions may be required to obtain results for all analytes of interest in the calibration range. For samples run at multiple dilutions, the analysis of greatest concentration, qualified retention time, good peak shape, and satisfactory resolution should be quantitated and reported. However, all dilutions should be assessed comparing the consistency of the determinations and possible matrix effects. In certain instances, the more diluted analysis may be the more appropriate reported result.

7.10 If a sample is run at dilution and the concentration of the analyte of interest is below the PQL the sample should be reanalyzed at a greater concentration. Where there is coelution of higher relative concentration analytes to adjacent analytes, e.g. chloride to nitrite, a sufficient dilution of the sample should be analyzed to maximize the

resolution of the two analytes and report the affected analyte concentration at an elevated PQL narrated to that effect.

7.11 If the chromatogram fails to produce adequate resolution, or if the identification of specific anions is questionable, the sample may be spiked with an appropriate amount of standard and reanalyzed. In some instances dilution of the sample may provide sufficient resolution for identification and quantitation.
NOTE: Retention time is inversely proportional to concentration. Nitrate and sulfate exhibit the greatest amount of change, although all anions are affected to some degree. In some cases this peak migration may produce poor resolution or identification.

7.12 Calculations are performed by TurboChrom using responses measured during analysis of the calibration standards for the operating curve that has been calculated based upon a linear regression formula. Individual calibration curves are calculated for each detector. The analyst must assure that the method file is calculating against the appropriate curve. Aqueous and soil sample analyte concentrations are calculated using the following equation:

$$A = (mR + B) * DF$$

where:
A = analyte concentration mg/L
m = slope
R = response in peak area
B = y intercept
DF = dilution factor prior to analysis

NOTE: In order for the dilution factor to be applied during calculation by TurboChrom it must be entered at the time the sequence is entered and/or edited.

7.13 In the event that the software interpretation of integration is not appropriate manual integration may be performed. It is expected that the same sound technical judgments and assessments will be equally applied to both samples and standards in the review of integrations and the decisions to perform or not perform manual integrations. In accordance with Section 7.7 of the Katahdin Analytical Services Quality Assurance Manual, any manual integration must be initialed and dated by both the analyst performing the integration and by the reviewer. **Under no circumstances is the original software generated result file to be overwritten with a manually edited file. Both the original software generated integration and the manual integration must be preserved with the raw data.** The analyst should rarely be required to manually integrate any QC elements. This is usually indicative of poor system performance and corrective action should be taken through proper maintenance.

7.14 The final raw data report will include information on initial and continuing calibrations and results of all quality control data. The instrument printout Includes Result File Name, Calibration File Name, Sample #, Analyst, Date Analyzed, Ion Initial Volume, Dilution Factor, Observed Concentration, Reported Concentration, % Recovery. (See Figure 4.)

7.15 Sample preparation information is entered manually into the Katahdin Information Management System (KIMS). Instrument data files are then imported electronically into KIMS for calculation and reporting of sample results and quality control data. Refer to the current revision of SOP CA-762 ("W et Chemistry Data Entry and Review Using Katahdin Information Management System") for further information.

7.16 A batch sheet is generated (Figure 5). Raw data and batch sheets are reviewed for completeness and accuracy by the Department Manager or other qualified designee.

7.17 Printouts of instrument calibrations and sample data are filed in the lab for approximately 3 months for reference by analysts. Prior calibrations are archived and all are available in the TurboChrom database.

TROUBLESHOOTING

7.18 High biased blanks, CCVs, and LCSs for Cl^- may indicate a contaminated system. The most common cause of this type of contamination is the intrinsically high chloride concentration of many samples. Chloride contamination can contribute to the failure of QC criteria for both chloride and nitrite. A number of techniques can be used to return the system to normal operation.

7.18.1 The WISP, auto-sampler, is equipped with a purge function that serves to wash out the injection assembly. Running 4-5 purges can remove much contamination.

7.18.2 The injection of water blanks after injecting high Cl^- concentrations samples (e.g. estuarine water) may be effective. However, in some cases it can take 10-15 DI injections to return the system to normal operation.

7.18.3 Consult source and/or history of samples to make an educated judgment relative to dilutions that will place the samples within the linear calibration range.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 2 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 2 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 2, 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 2 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, remaining sample volume and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.
In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Initial Instrument Calibration – Instrument calibration, which is generated using a blank the five standards listed in Section 5.6, is performed at least once every six months or whenever there is a significant change in instrument operating conditions or hardware. One of the calibration standards must be at or below the Practical Quantitation Limit for each analyte. The curve fit is accomplished using least squares linear regression, and the correlation coefficient for the curve must be at least 0.995. Sample results that exceed the calibration range of the instrument may not be reported; the sample must be diluted and reanalyzed until the measured concentration of the analyte is within the calibration range. Because calibration linearity is established each time the instrument is calibrated (at least every six months) and because sample results that fall outside the calibration range may not be reported, a separate linear calibration range study is not performed.

8.2 Lower Limit of Quantitation - The laboratory should establish the LLOQ for each analyte as the lowest reliable laboratory reporting concentration or in most cases the lowest point in the calibration curve which is less than or equal to the desired regulatory action levels, based on the stated project requirements. Analysis of a standard prepared at the LLOQ concentration levels or use of the LLOQs as the lowest point calibration standard provides confirmation of the established sensitivity of the method. The LLOQ recoveries must be within 50% of the true values to verify the data reporting limit.

8.3 Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB) – Ongoing calibration accuracy is verified by analyzing a CCV standard (a mid-range check standard) and a CCB at the beginning of each run, after every ten samples, and at the end of each run. The recovery of each CCV must be within 90% - 110% of

the true value for each analyte. The measured concentration of each analyte in the CCB must be below the Practical Quantitation Limit for the analyte. If a CCV or CCB fails, the analysis must be stopped, the problem corrected, and the previous ten samples must be reanalyzed, with the following exception. If one or both CCVs bracketing a sample result are biased high and the sample concentration is <PQL, the sample result may be reported. CCVs or CCBs that are biased high may be indicative of carryover or contamination in the system by high concentration samples.

NOTE: High bias for chloride may be indicative of chloride contamination in the injector. If the run is attended and chloride is an analyte of interest, halt the run and take corrective action if bias is observed.

8.4 Laboratory Control Sample (LCS) / Initial Calibration Verification (ICV) – One LCS/ICV, prepared from a separate standard source from the Initial Instrument Calibration, must be analyzed with each batch of 20 or fewer samples. LCS/ICV recovery acceptance limits are 90% - 110% for EPA Method 300.0 and 80% - 120% for SW846 Method 9056.

8.5 Method Blank (MB) – A method blank consisting of reagent water that filtered in the same fashion as the associated samples must be analyted with each batch of 20 or fewer samples. The measured concentreation of each analyte in the MB must be less than the PQL. In the instance where there is a value in excess of the CCB, the filter source should be suspected as contributing contamination. Repeat the analysis with additional cartridges.

8.6 Matrix Spike (MS) Sample – Matrix spike samples must be prepared and analyzed at a frequency of 10% (one sample in 10) for EPA Method 300.0 or 5% (one sample in 20) for SW846 Method 9056A. Matrix spikes is prepared by adding 0.05 mL of Primary Matrix Spike Mixed Standard (Section 5.9) to 1.0 mL of the filtered sample prior to loading. If the concentration of the spike is less than 25% of the native sample concentration the matrix spike recovery should not be calculated. The matrix spike recovery acceptance limits are 90% - 110% for EPA Method 300.0 and 80% - 120% for SW846 Method 9056A/ If the recovery of any analyte falls outside the criteria range and the LCS and CCVs are within criteria, the poor recovery should be attributed to sample matrix.

8.7 Laboratory Duplicate (Dup) or Matrix Spike Duplicate (MSD) - One duplicate or matrix spike duplicate sample must be analyzed with each batch of 20 or fewer samples. Analytes with measured values $\geq$ 5 times the PQL should achieve duplicate/MSD sample precision of $\leq$ 20% RPD.

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 revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.
Refer to the current revisions of the applicable methods for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Methods for the Determination of Inorganic Substances in Environmental Samples", EPA - 600/R - 93 - 100, August 1993.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, Third Edition, Final Update IV, February 2007, Method 9056.

WISP717plus Autosampler Operator Manual

Waters 510 HPLC Pump Manual

Dionex 200i Manual

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TABLE 1

EXAMPLE ANALYTICAL SEQUENCE WITH ACCEPTANCE CRITERIA

Sample Number	Instrument Runlog	Acceptance Limit
1	CCV	90%-110%
2	CCB	< PQL
3	LCS	90 -110% (300.0) 80%-120% (9056A)
4	Sample 1	
5	Sample 2	
6	Sample 3	
7	Sample 4	
8	Sample 5	
9	Sample 6	
10	Sample 7	
11	Sample 8	
12	Sample 9 -Duplicate	20% RPD
13	Sample 10 - Matrix Spike	90 -110% (300.0) 80%-120% (9056A)
14	CCV	90%-110%
15	CCB	< PQL
16	Sample 11	
17	Sample 12	
18	Sample 13	
19	Sample 14	
20	Sample 15	
21	Sample 16	
22	Sample 17	
23	Sample 18	
24	Sample 19	
25	Sample 20 - Matrix Spike	90 -110% (300.0) 80%-120% (9056A)
26	CCV	90%-110%
27	CCB	< PQL

TABLE 2

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Anions by Ion Chromato-graphy EPA Method 300.0 and SW846 Method 9056A	Initial Instrument Calibration (Blank + 5 standards, lowest standard at or below PQL)	Every 6 months or with each change in instrument operating conditions or equipment	1) Correlation coefficient ≥ 0.995 2) Recovery of lowest standard within 50%-150%	Correct problem and recalibrate
	Method blank	One per prep/analysis batch of 20 or fewer samples	No analyte detected ≥PQL	(1) Investigate source of contamination (2) Report all sample results <PQL. (3) Report sample results >10X the blank result and flag results with a "B". (4) Reanalyze all other samples associated with the failing blank.
	LCS/ICV	One per prep/analysis batch of 20 or fewer samples, prepared from a separate source than calibration standards	90%-110% Recovery	(1) If the LCS fails high, report samples that are <PQL. (2) Recalibrate and/or reanalyze other samples.
	CCV	At beginning of run, after every 10 samples, and at end of run	90%-110% Recovery	(1) If the CCV fails high, report samples that are <PQL. (2) Recalibrate and/or reanalyze samples back to last acceptable CCV recovery
	CCB	Immediately following each CCV	No analyte detected ≥PQL	1) Investigate source of contamination (2) Report all sample results <PQL. (3) Report sample results >10X the blank result and flag results with a "B". (4) Reanalyze all other samples associated with the failing CCB.

TABLE 2

QC REQUIREMENTS (Continued)

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Anions by Ion Chromato-graphy EPA Method 300.0 and SW846 Method 9056A	Matrix Spike	One for every set of 10 samples (EPA 300.0) or one for every set of 20 samples (SW846 9056A)	90%-110% Recovery (EPA 300.0) 80%-120% Recovery (SW846 9056A)	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS are unacceptable reprep and reanalyze the samples and QC. (3) Notate sample result in raw data if matrix interference suspected.
	Sample Duplicate or Matrix Spike Duplicate	One per 20 samples	RPD $\leq$20%	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD still out, report original result with notation or narration.
	Demonstration of analyst proficiency; accuracy and precision	One time initially by each analyst performing the method and annually thereafter.	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personnel training file
	MDL study	Once every 6 months or with significant change in analysis conditions	Ideally, PQL = at least 3 X the MDL.	(1) Perform any required maintenance (2) Repeat MDL

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-742-06	Method 300.0/9056, current revisions
Calibration	1) Calibration consists of blank + 5 standards 2) Linearity verified by performing calibration at least every 6 months, and diluting all samples exceeding calibration range	1) Calibration consists of blank + at least 3 standards 2) Linear range verification required every 6 months (Method 300.0)
QC – Method Blank	Acceptance limit < PQL	Acceptance limit <MDL (Method 300.0) Acceptance limit <10% of LLOQ or regulatory limit or lowest sample (Method 9056A)
QC – LCS/ICV	Combined LCS and ICV with tighter acceptance limits (90%-110% recovery)	LCS acceptance limits 80%-120%, ICV acceptance limits 90%-110% (Method 9056A)
QC – Duplicate / Matrix Spike Duplicate	RPD acceptance limit ≤20%.	RPD acceptance limit ≤15% for samples at or above midrange, ≤50% for samples near LLOQ (Method 9056A)

TITLE: ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056

FIGURE 1

IC SYSTEM CONFIGURATION

ION CHROMATOGRAPH CONFIGURATION

January 2000

HPLC Pump # 510-143438
Dionex Conductivity Detector # CDM-1 852108
AS4A # 67384
Anion Suppressor # AMMS-II 14930

Gradient Controller # 680-006872
Waters Conductivity Detector # 000168
AG4A # 29841
Peristaltic Pump 77120-70

WISP # 712-00-8346
In-Line Filter # Dionex 044105
NG1 # 15058

NOTE: It is very important to assure that the flow of the eluent is consistent with the "flow direction" arrow printed on the columns

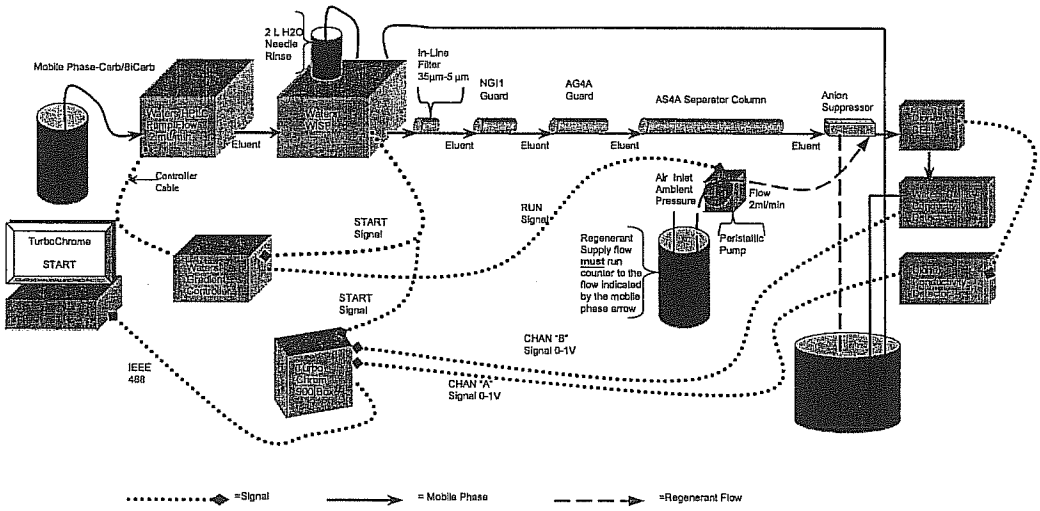


FIGURE 2

EXAMPLE IC MAINTENANCE LOG PAGE

KATAHDIN ANALYTICAL SERVICES, INC.
WATERS IC (ION CHROMATOGRAPHY) - MAINTENANCE LOGBOOK
SERIAL NO. 001457

DAILY INSTRUMENT PERFORMANCE

GRADIENT CONTROLLER/HPLC PUMP

ANIONS	SET AT 2 ML/MIN	☐	LINE PRESSURE	
PERCHLORATE	SET AT 1.0ML/MIN	☐		

WATERS DETECTOR - CHANNEL B

WATERS DETECTOR

DESCRIPTION	SETTING
BASE RANGE	
SENSITIVITY	

WATERS WISP

DESCRIPTION	SETTING
INJECTION VOLUME	
RUN TIME	

MAINTENANCE (AS NEEDED)

TASK	DATE	ANALYST	COMMENTS
Clean surfaces of system unit as needed.			
Check regenerate pump tubing & replace as needed.			
Clean or regenerate column as needed.			
Change analytical column as needed.			
Change guard column as needed.			
Change suppressor as needed.			

NON ROUTINE MAINTENANCE

Initials:_____Date:_____

Reviewed By:_____Date:_____

TITLE: ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056

FIGURE 3

EXAMPLE OF ANIONS BY IC RUNLOG PAGE

KATAHDIN ANALYTICAL SERVICES, INC. - ION CHROMATOGRAPHY RUN LOG

METHODS – EPA 300.0 (E) SW846 9056 (S)						DATE OF LAST INITIAL CALIBRATION : 2/14/08			INSTR. ID.: IC01	
ANALYTES OF INTEREST						STANDARD	CONCENTRATION	PREPARATION	STANDARD CODE	
						IC CAL MIX	SEE FRONT COVER	SEE FRONT COVER	From Standards Log	W 7173
CL	NO2	BR	NO3	PO4	SO4	IC LCS MIX	SEE FRONT COVER	SEE FRONT COVER	From Standards Log	W 7174
✓	✓	✓	✓	✓	✓	ICV/CCV STD	CAL STD #2	SEE FRONT COVER	ICV plus Date	~~ICV~~ MS W7175

Date	Init	Result File	Sample #	Method	Dilution	Accept ? Check Analytes (R = Rerun) CL	NO2	BR	NO3	PO4	SO4	Manual Integration? List Analytes	Comments
2/14/08	CP	EYB2048	IC1	E	1	✓	✓	✓	✓	✓	✓		
		49	IC2									Cl, NO2	
		50	IC3									Cl, NO2	
		51	IC4									All anions	
		52	IC5									Cl, NO3, NO2, Br	
		53	IC6			✓	✓	✓	✓	✓	✓	All anions	
		54	Blank			✓	✓	✓	✓	✓	✓	All anions	~~Rerun Br low lod~~ CP
		55	LCS		1	✓	✓	✓	✓	✓	✓	All anions	~~Rerun~~ CP
		56	CP ~~LCS~~ SB0570-8		1	~~R~~	~~R~~	~~R~~ CP	R	✓	R	PO4	dilute
		57	CP ~~BLCA~~ LCS SB0570-9		1		✓					NO2	
		58	SB0474-7		1					✓		PO4	
		59	SB0570-8DQ		1					✓		PO4	
		60	-8MS		1					✓			
		61	SB0570-8DQ		5	✓			✓		✓		
		62	-8MS CP		5	✓			✓		✓		
		63	SB0570-8DQMS		5	✓			✓		✓		
✓	✓	64	SB0570-8	F	10								-dilution not needed

REVIEWED BY Amb ON 3/11/08

W648859
R77295

QAWL465

0000044

TITLE: ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056

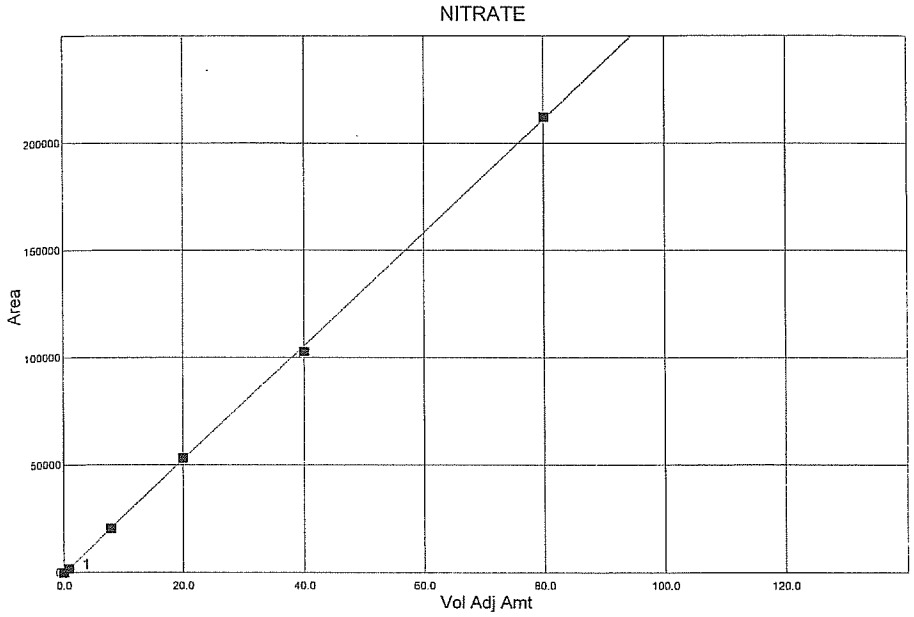
FIGURE 4

IC CALIBRATION CURVE

Fit Analysis Output For Method File: "W:\DATA\IC01\METHODS\EICC088.MTH"
Component Name: "NITRATE"
Date: 02/14/2008 Time: 05:11 PM

Curve Parameters:

Curve #1 : 1st Order - Incl Origin
Weighting Factor = 1.0 (No Weighting) r^2 = 0.999758
Calibration Curve = (-377.872938) + (2644.867657)X



Level Name	Observed X-Value	Calculated X-Value	Delta	%Diff.	Observed Y-Value	Calculated Y-Value	Delta	%Diff.
IC6	0.002000	0.142870	-0.140870	-98.600	0.000	-372.583	372.583	-100.00
IC5	0.800000	0.859919	-0.059919	-6.968	1896.500	1738.021	158.479	9.118
IC4	8.000000	7.920575	0.079425	1.003	20571.000	20781.068	-210.068	-1.011
IC3	20.000000	20.293468	-0.293468	-1.446	53295.663	52519.480	776.183	1.478
IC2	40.000000	39.016649	0.983351	2.520	102816.000	105416.833	-2600.833	-2.467
IC1	80.000000	80.425648	-0.425648	-0.529	212337.323	211211.540	1125.784	0.533

CP
2/14/08

TITLE: ANIONS BY ION CHROMATOGRAPHY USING EPA METHOD 300.0 AND SW-846 9056

FIGURE 5

IC BATCH SHEET

WET CHEMISTRY BATCH REPORT
Apr 16 2008, 09:01 am
Batch: WG50311

Parameter: Nitrate as N

Date Analyzed: 15-APR-08

Analyst Initials: AMB

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
SB1835-1	SAMP	SW846 9056	1.0000mL	1.0000mL	5	19.9079	19.3 mg/L	NA	.05	.058	.25		
WG50311-1	MBLANK	SW846 9056	1.0000mL	1.0000mL	1	.0147	U0.050 mg/L	NA	.05	.012	.05		
WG50311-2	LCS	SW846 9056	1.0000mL	1.0000mL	1	1.672	1.7 mg/L	NA	.05	.012	.05		99
WG50311-3	DUP	SW846 9056	1.0000mL	1.0000mL	5	18.8796	18.9 mg/L	NA	.05	.058	.25	5	
WG50311-4	MS	SW846 9056	1.0000mL	1.0000mL	10	25.885	25.9 mg/L	NA	.05	.12	.5		78

Comments:

WG50311-1	SB1835-1
WG50311-2	SB1835-1
WG50311-3	SB1835-1
WG50311-4	SB1835-1

Entered by: AMB Date: 4/16/08 Accepted by: [illegible] Date: 04/16/08

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

Prepared By: Chad Delaney Date: 07.14.04

Approved By:

Department Manager: George Brewer Date: 07/14/04

Operations Manager: Deborah J. Nadeau Date: 7.14.04

QA Officer: Maria Crouch Date: 07.14.04

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Revised Title and text to incorporate analysis for Total Inorganic Carbon (TIC) minor changes throughout Updated Figures	LAD	04/06	04/06
02	Changed SM5310B to SM5310C throughout. Added reference to SW846 9060 throughout. Updated Figure 6. Updated Table 2 to include that we do not subtract the blank from the sample.	LAD	03/07	03/07

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

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

I acknowledge receipt of copy ___ of document **SOP CA-763-02**, titled **Analysis of TOC, DOC, and TIC in Aqueous Samples using the Shimadzu Carbon Analyzer: EPA Method 415.1, SW846 9060 and SM5310B**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-763-02**, titled **Analysis of TOC, DOC, and TIC in Aqueous Samples using the Shimadzu Carbon Analyzer: EPA Method 415.1, SW846 9060 and SM5310B**.

Recipient: _____ Date: _____

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services, Inc. technical personnel for analyzing total organic carbon (TOC), dissolved organic carbon (DOC), and total inorganic carbon (TIC) in aqueous samples using the Shimadzu Carbon Analyzer in accordance with EPA method 415.1, SW846 9060 and SM 5310B.

This procedure applies to drinking and surface waters, domestic and industrial wastewater. The Practical Quantitation Limit (PQL) is 1 mg/L.

1.1 Definitions

Total Organic Carbon (TOC) – Carbon that is bound with hydrogen or oxygen in organic compounds, analyzed from an unfiltered sample.

Dissolved Organic Carbon (DOC) - Carbon that is bound with hydrogen or oxygen in organic compounds, analyzed from a filtered sample.

Total Inorganic Carbon (TIC) – Carbon contained in carbonates, hydrogen carbonates, or dissolved carbon dioxide.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the preparation and analysis of samples for TOC, DOC, and TIC using EPA Method 415.1, SW846 9060 and SM5310 B. Each analyst must demonstrate his/her ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability."

It is the responsibility of all Katahdin technical personnel involved in the determination of TOC and DOC to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method have not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with 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 Program for further details on pollution prevention techniques.

Waste generated during the preparation and analysis of samples must be discarded appropriately. Place all analyzed samples, standards, and rinsings in a Satellite Waste "A" Acid for proper disposal in main waste area "A".

Other wastes generated during the preparation of samples must be disposed of in adherence with the Katahdin 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

Organic carbon analysis – Sample is drawn into a syringe in the instrument, and hydrochloric acid is then drawn into the syringe to acidify the sample to pH 2 to 3. Oxygen is then bubbled through the acidified sample in the syringe to drive off the inorganic carbon component. The sparged sample is then introduced into the total carbon combustion tube,

where it is heated to 680° C in the presence of an oxidation catalyst. The organic carbon remaining in the sample after sparging is converted to carbon dioxide during combustion. Carrier gas sweeps the sample combustion gases through an electronic dehumidifier, where they are cooled and dehydrated, and then through a halogen scrubber to remove chlorine and other halogens. Finally, the carrier gas delivers the combustion gases to the cell of a non-dispersive infrared (NDIR) gas analyzer, where carbon dioxide is detected and quantitated. The amount of carbon dioxide in a sample is directly proportional to the concentration of organic carbon (TOC or DOC) in the sample. Strictly speaking, this method measures non-purgeable organic carbon (NPOC), because purgeable (volatile) organic compounds are lost during sparging of the sample. However, the amount of purgeable organic compounds in natural waters is small, and NPOC is nearly equivalent to total organic carbon. When necessary, total organic carbon can be indirectly determined by measuring total carbon on an unsparged sample, measuring inorganic carbon on a separate aliquot of the sample, and calculating TOC by difference.

Inorganic carbon analysis – Inorganic carbon (IC) consists of the carbon contained in carbonates and in carbon dioxide dissolved in water. IC is measured by injecting an aliquot of unpreserved sample into the instrument's IC reaction vessel, where it is acidified with hydrochloric acid and inorganic carbon is converted to carbon dioxide. The carbon dioxide is then sparged from solution with oxygen and carried to the NDIR detector, where it is detected and quantitated.

3.0 INTERFERENCES

High salt samples can affect the oxidation rate, leading to low recoveries. Interference begins at ~0.1% total dissolved solids and becomes severe at > 0.35%.

4.0 APPARATUS AND MATERIALS

4.1 Shimadzu Carbon Analyzer Model TOC-V

4.2 Shimadzu carousel autosampler Model ASI-V

4.3 Glass 40 mL VOA Vials

4.4 Class A volumetric flasks (100 mL and 200 mL)

4.5 Adjustable pipets (0.1, 1.0 and 5.0 mL)

4.6 Oxygen gas

5.0 REAGENTS

NOTE: All standards and reagents should be prepared daily except where noted. Record all standard and reagent preparation in the appropriate logbook (Figures 1 and 2).

5.1 Phosphoric acid (H_3PO_4), concentrated, reagent grade.

5.2 Phosphoric acid (H_3PO_4), 1:1 – Slowly add 100 mL concentrated H_3PO_4 to 100 mL and mix. Prepare fresh every six months.

5.3 Hydrochloric acid (HCl), concentrated, reagent grade.

5.4 Hydrochloric acid (HCl), 2M – Slowly add 42.5 mL concentrated HCl to 200 mL of reagent water and bring to a final volume of 250 mL. Prepare fresh every six months.

5.5 Potassium hydrogen phthalate ($HOCOC_6H_4COOK$), primary standard grade. Two different lots of potassium hydrogen phthalate are required – one is used to prepare organic carbon calibration standards, and the other is used to prepare a laboratory control sample (second source).

5.6 Sodium carbonate (Na_2CO_3), 1 N – Purchased certified standard solution, inorganic carbon true value = 6000 mg/L. This solution is used to prepare the inorganic carbon stock standard and the alkalinity check standard.

5.7 Sodium carbonate (Na_2CO_3), anhydrous – ACS reagent grade. Store in a desiccator. This reagent is used to prepare the inorganic carbon laboratory control sample.

Organic Carbon (OC) Standards

5.8 OC Stock Standard (used in preparing OC calibration standards and CCV): Prepare a 2000 mg/L standard by dissolving 0.4250g of potassium hydrogen phthalate in 100 mL of laboratory reagent grade water. Add 0.10 mL of concentrated phosphoric acid. Stored in an amber bottle in the refrigerator, this solution may be used for three months.

5.9 OC Calibration Standards – Prepare a series of five calibration standards by diluting the OC Stock Standard with reagent water in accordance with the following table:

Calibration Std. Conc. (mg/L OC)	mL of OC Stock Std. Added	Final Volume (mL)
1.0	0.10	200
5.0	0.50	200
20.0	2.0	200
50.0	5.0	200
200.0	20.0	200

5.10 OC CCV Standard - Prepare a 100 mg/L standard for the CCV by diluting 25 mL of the OC Stock Standard to 500 mL using laboratory reagent grade water. Stored in an amber bottle in the refrigerator, this solution may be used for three months.

5.11 OC Laboratory Control Sample (LCS) – Prepare a 50 mg/L standard from a different source from the calibration standards, as follows. Fill a 1 L volumetric flask half full with laboratory reagent grade water and add one mL of concentrated phosphoric acid as a preservative. Add 0.1062 g of potassium hydrogen phthalate and bring to a final volume of 1 L with laboratory reagent grade water. Stored in an amber bottle in the refrigerator, this solution may be used for three months.

5.12 OC Matrix Spike (MS) sample – OC matrix spike aliquots are prepared by adding 2.0 mL of OC Stock Standard to 38 mL of sample in a VOA vial. The added matrix spike concentration is 100 mg/L.

5.13 Alkalinity Check Sample - Prepare by diluting 2.5 mL of 1.0 N Na_2CO_3 to one liter with laboratory reagent grade water. The inorganic carbon content of this solution is 15 mg/L. This standard is used to monitor for false positives during OC analysis from incomplete removal of inorganic carbon.

Inorganic Carbon (IC) Standards

5.14 IC Stock Standard (used in preparing IC calibration standards and CCV): Prepare a 2000 mg/L standard by diluting 100 mL of 1 N $Na_2CO_3$to a final volume of 300 mL in a graduated cylinder. Stored in a glass bottle in the refrigerator, this solution may be used for three months.

5.15 IC Calibration Standards – Prepare a series of five calibration standards by diluting the OC Stock Standard with reagent water in accordance with the following table:

Calibration Std. Conc. (mg/L IC)	mL of IC Stock Std. Added	Final Volume (mL)
1.0	0.10	200
5.0	0.50	200
20.0	2.0	200
50.0	5.0	200
200.0	20.0	200

5.16 IC CCV Standard - Prepare a 100 mg/L standard for the CCV by diluting 25 mL of the OC Stock Standard to 500 mL using laboratory reagent grade water. Stored in a glass bottle in the refrigerator, this solution may be used for three months.

5.17 IC Laboratory Control Sample (LCS) – Prepare a 50 mg/L standard from a different source from the calibration standards, as follows. Fill a 1 L volumetric flask half full with laboratory reagent grade water and add 0.4417 g of anhydrous sodium carbonate. Mix the solution until the sodium carbonate has dissolved, and bring to a final volume of 1 L with laboratory reagent grade water. Stored in a glass bottle in the refrigerator, this solution may be used for three months.

5.18 IC Matrix Spike (MS) sample – IC matrix spike aliquots are prepared by adding 2.0 mL of IC Stock Standard to 38 mL of sample in a VOA vial. The added matrix spike concentration is 100 mg/L.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Collect samples in glass jars or VOA vials. For organic carbon analysis, preserve with H_2SO_4 at time of collection and store at 4 (±2) °C prior to analysis. For inorganic carbon analysis, do not add acid to the sample, but store at4 (±2) °C prior to analysis. The holding time for TOC, DOC, and IC analysis is 28 days from the time of collection.

If dissolved organic carbon (DOC) is to be determined, the sample must be filtered through a 0.45 micron glass fiber filter prior to preservation with H_2SO_4. If the sample has not been filtered in the field, sample log-in must inform the Inorganic Department Manager that samples need to be filtered upon receipt by the laboratory. Please refer to the current revision of Katahdin SOP SD-902, Sample Receipt and Internal Control, section 7.7.5, for further filtration procedures.

7.0 PROCEDURES

ANALYZER START-UP PROCEDURE

7.1 Turn on the TOC-V module, computer, and oxygen tank.

7.2 Fill rinse water reservoir with laboratory reagent grade water. Verify that there is sufficient room in the satellite waste container to contain the day's waste. Verify that the instrument's humidifier water level is between the "High" and "Low" lines, and add laboratory reagent grade water if needed. Also verify that the diution water, hydrochloric acid, and phosphoric acid reservoirs are full.

7.3 Carrier gas flow rate should be set at 150 mL/min. The oxygen pressure should read 200 kPa on the gauge inside the instrument.

7.4 From the main screen, double-click on the **TOC-Control V** icon.

7.5 Double-click the **Sample Table Editor** icon. Enter the User ID and Password.

7.6 At the menu bar, click on File , then click New. Double-click on the **Sample Run** icon.

7.7 Make certain that the System setting is set for "TOC-Vcph/ASI-V", then click OK.

7.8 Click on the **Connect (yellow lightning bolt)** icon to connect the computer to the instrument. Click "Use Settings on PC". This turns on the furnace inside the instrument, and turns on the gas flows. The furnace must be allowed to heat up for one half hour before beginning analysis.

SAMPLE ANALYSIS

7.9 At the menu bar, click on Insert, then click Auto Generate. Double-click on the appropriate method ("npoc AQ.met" for TOC/DOC, "INORG CARB AQ.met" for inorganic carbon). Then click Next.

7.10 Enter the number of samples as "60" and the start vial as "5", then click Next.

7.11 At the Calibration Curve screen (Screen 3), click Next. You must verify the calibration curve is current (within three months of analysis) by going into the NPOC Method Menu. If the curve is more than three months old, a new curve must be run.

7.12 Click to put a checkmark in all three boxes (beside "At The Beginning", "After Every Ten Samples", and "At The End of Sample Group") under Quality Control.

7.13 Select (1) by clicking on the number (it will highlight the (1) in blue). Click Add. Then click "CCV". This inserts a CCV in autosampler position (1).

7.14 Repeat 7.13 for autosampler position (2), clicking "Blank", and then for autosampler position (3), clicking "LCS".

7.15 Click Finish.

7.16 You may bypass the vial selection screen at this time by clicking OK.

7.17 An autosampler table now appears on the screen. Fill in sample numbers in the "Sample Name" column. If a manual dilution has been performed on the sample in the VOA vial, enter the dilution factor in the "Dilution" column for each diluted sample.

7.18 Unneeded Quality Control and Sample rows may be deleted by selecting each row and then clicking the **Cut (scissors)** icon.

7.19 Print the autosampler table (Figure 3) by clicking File on the menu bar, then clicking Print, then clicking Table. Some of the autosampler positions listed will be incorrect, because the computer does not account for vials that are sampled twice (vials may be sampled twice for duplicate or matrix spike duplicate analysis). Write autosampler table corrections on the autosampler table printout. Using the corrected autosampler table for reference, place each sample vial in its appropriate position on the autosampler carousel. Matrix spikes are prepared by adding 2 mL of the OC or IC Stock Standard to a VOA vial and adding 40 mL of sample.

7.20 Place the autosampler carousel on the ASI auto sampler and replace the ASI cover.

7.21 Click the **Start (traffic light)** icon, the enter a data file name and click "Save". Data files are named according to the analyte (TOC or TIC), the matrix (AQ), and the date (MMDDYY format), e.g. "TOC_AQ_031505" for an aqueous TOC run performed on 03/15/05.

7.22 Click "Standby".

7.23 An image of the carousel will appear on the screen, along with a table listing the sample numbers and corresponding autosampler positions. Carefully enter the corrected autosampler positions in accordance with the previously printed list that was used to set up the autosampler carousel.

7.24 Click "OK".

7.25 The instrument will then run samples until it has finished with the programmed autosampler sequence.

ANALYTICAL QUALITY CONTROL

7.27 An analytical batch consists of 20 or fewer field samples (not counting method blanks, laboratory control samples, duplicates, and matrix spikes).

7.28 The operating range of OC and IC analysis for a 150 uL sample injection is 0-200 mg/L. Samples with measured concentrations greater than 200 mg/L must be diluted and reanalyzed.

7.29 Each analytical sequence must start with analysis of a CCV, followed by analysis of a method blank consisting of laboratory reagent grade water. Analyze an second-source laboratory control sample (LCS) with each analytical batch.

7.30 Each TOC or DOC sample is sparged with oxygen to remove residual inorganic carbon. To check for sparging efficiency, analyze an alkalinity check standard near the beginning of each run.

7.30 One matrix spike (MS) should be analyzed for every ten (or fewer) samples.

7.31 One sample in every batch of 20 or fewer samples must be analyzed in duplicate.

DATA REVIEW AND REPORTING

7.32 After completion of analysis, instrument data files are imported electronically into the Katahdin Information Management System (KIMS) for calculation and reporting of sample results and quality control samples. After data processing by KIMS, a batch sheet (Figure 5) listing Katahdin Sample Numbers with associated reported results and associated quality control data is printed out of KIMS for each analytical batch. Refer to the current revision of SOP CA-762 ("Wet Chemistry Data Entry and Review Using Katahdin Information Management System") for further information.

7.33 Analytical data are stored in the instrument's computer during analysis, and data reports are printed after each analytical run has been completed. The instrument data printout shows the signal versus run time plot that was obtained for each sample, as well as the peak area and measured concentration, the dilution factor, and the analysis date and time. A run data summary table (Figure 4) is also printed out from the instrument, containing all of this information except peak plots in analysis order. Copies of the run data summary tables are bound in the Aqueous Carbon Analysis Run Logbook.

7.34 A Carbon Analysis Run Information Sheet (Figure 6) is completed by the analyst for each analytical run. This form lists the analysis methods, dates, and times, calibration information, and the standard IDs for each standard used in the run.

7.35 The raw data package for each analytical run is assembled by the analyst, and consists of the following information (in the order listed):

- KIMS batch sheets (Figure 5) for each batch contained in the run
- The Carbon Analysis Run Information Sheet (Figure 6)
- The run data summary table (Figure 4)
- The raw data printouts which include peak plots
- Associated calibration data

7.36 All raw data packages are stored on site for at least three months, and are subsequently stored off site by the laboratory.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

See below and refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) 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 Method detection limits (MDL) are determined annually for each analyte analyzed by each method on each instrument. This determination requires at least seven replicate digestions and analyses of reagent water spiked at 3-5 times the

anticipated MDL for each analyte. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

ANALYTICAL RUN QC SAMPLES

8.2 A Continuing Calibration Verification (CCV) standard is analyzed for each analyte after every ten samples and at the end of the analytical run. The CCV solution is as described in Section 5. Results of the CCVs must fall within 80% to 120% of the expected value. If a CCV fails, sample results that are associated with (bracketed by) the failing CCV my not be reported from the run. The samples must be reanalyzed after the problem is corrected and a passing CCV has been analyzed.

8.3 A Continuing Calibration Blank (CCB) consisting of reagent water is analyzed after each CCV. The absolute values of results of CCBs must be less than the Practical Quantitation Level (PQL) for each analyte. If a CCB fails, sample results that are associated with (bracketed by) the failing CCB may not be reported from the run, with the following exception. If the absolute value of a result for a CCB is greater than the PQL, sample results that are less than the PQL or greater than or equal to ten times the measured CCB concentration may be reported. All other samples must be reanalyzed after the problem is corrected and a passing CCB has been analyzed.

8.4 An Alkalinity Check Sample, prepared as described in Section 5.13, is analyzed in each TOC and DOC run. This sample is used to monitor for false positives during OC analysis from incomplete removal of inorganic carbon. The absolute value of results of the Alkalinity Check Sample must be less than the Practical Quantitation Level (PQL) for each analyte. If the Alkalinity Check Sample fails, associated sample results may not be reported from the run. The samples must be reanalyzed after the problem is corrected and a passing Alkalinity Check has been analyzed.

BATCH QC SAMPLES

8.5 A Method Blank, consisting of reagent water, is analyzed with each batch of twenty or fewer samples. The results of method blanks must be less than the Practical Quantitation Level (PQL) for each analyte. If a method blank fails, results for associated samples may not be reported from the batch, with the following exception. If the result for a method blank is greater than the PQL, associated sample results that are greater than or equal to ten times the measured preparation blank concentration may be reported with "B" notation. Associated sample results that are less than the PQL may be reported with no additional notation.

8.6 A laboratory control sample (LCS), prepared as described in Section 5, is analyzed with each batch of twenty or fewer samples. The LCS recovery must be 80-120% for USEPA Method 415.1 and SW846 9060. The LCS recovery must be 90-110% for SM5310B. If a laboratory control sample fails, results for the associated samples may not be reported from the batch, and the samples must be reanalyzed, withthe following exception: if the LCS recovery is greater than the upper acceptance limit, samples with measured concentrations below the PQL may be reported with appropriate narration.

MATRIX QC SAMPLES

8.7 Duplicate samples are analyzed at a minimum frequency of one per batch of 20 or fewer samples. The relative percent difference (RPD) between duplicate sample results is calculated as follows:

8.8

$$\text{RPD (\%)} = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where:

D_1 = First sample result
D_2= Second (duplicate) sample result

A control limit of 20% RPD is applied to duplicate analysis. If the matrix duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.8 Matrix spiked samples are prepared at a minimum frequency of one per 10 samples. The recovery for each element in a spiked sample must fall within 75%-125% for USEPA Method 415.1 and SW846 9060 or 80%-120% for SM 5310B if the result for the unspiked sample is less than four times the amount of spike added. If a recovery fails, the associated sample result must be flagged on the report of analysis. The spike recovery should be calculated as follows:

$$\text{Recovery (\%)} = \frac{S-U}{SA} *100\%$$

where:

S = Measured concentration of spiked aliquot
U = Measured concentration of unspiked aliquot
SA = Amount of spike added

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 Inorganic 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 current revision of USEPA Method 415.1, SW846 9060 and SM5310B for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

EPA Method 415.1 "Methods for Chemical Analysis of Water and Wastes", EPA 600/4-79-020, Revised March, 1983.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update III, December 1996, Method 9060.

"Standard Methods for the Examination of Water and Wastewater", 18th edition, 1992, APHA-AWWA-WPCF, Method 5310 B.

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TABLE 1

QC REQUIREMENTS

Analytical Method/ Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
EPA 415.1 SW846 9060 & SM 5310B TOC/TIC/ DOC	Method blank	One per analytical batch of 20 or fewer samples	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
	Laboratory control sample (LCS)	One LCS per analytical batch of 20 or fewer samples	80%-120% for EPA 415.1 and SW846 9060 90%-110% for SM5310B	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep the LCS and reanalyze the remaining samples.
	Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	80%-120% of true value	Reanalyze all associated samples.
	Continuing Calibration Blank (CCB)	After every CCV	Absolute value <PQL	1) If sample result < PQL, or >10x measured CCB value, report result 2) Else, reanalyze
	Matrix spike	One MS per ten samples	75%-125% for EPA 415.1 and SW846 9060 80%-120% for SM5310B	If LCS in criteria and matrix interference suspected, flag data
	Sample Duplicate	One sample duplicate per twenty samples	RPD $\leq$ 20	If lab QC in criteria and matrix interference suspected, flag data
	Initial demonstration of performance (4 replicate analyses of LCS)	Once per analyst per year	Precision and accuracy within method acceptance limits	Correct problem and repeat initial demonstration of performance
	MDL study	Once per year	Ideally, PQL = at least 3 X the MDL	Repeat MDL study

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TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-763-02	METHODS 415.1, SW846 9060 and SM5310B, current revisions
Apparatus/Materials	None	
Reagents	None	
Sample preservation/ handling	None	
Procedures	None	
QC – Blanks	Blank Subtraction not performed	Method SM5310B requires subtraction of method blank concentration from results for associated samples.
QC - Spikes	None	
QC - LCS	None	
QC - Accuracy/Precision	None	
QC - MDL	None	

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

FIGURE 1

EXAMPLE OF STANDARD PREPARATION LOGBOOK PAGE

Katahdin Analytical Services, Inc. - Wet Chemistry Lab

INTERMEDIATE AND WORKING STANDARD PREPARATION LOGBOOK

Standard ID	Standard Name	Preparation: Date	Preparation: Inits.	Expiration Date	Source Standard or Reagent Used: Vol.(mL) or Wt.(g)	Source Standard or Reagent Used: I.D. or Lot Number	Final Vol. (mL)
W4952	NO2/NO3 Std	7.6.04	PAG	7.20.04	2.0	PAG 7.6.04 W4727 4901	100 ml
					1.0		
					0.5		
					0.25		
					0.05		
W4953	NO3 CCV	7.6.04	PAG	7.20.04	0.5	W 4724	100 ml
W4954	ALKALINITY 1000/ml	7-7-04	[illegible]	7-21-04	1.0609	Std 964	1000 mL
W4955	ALKALINITY LOW LEVEL CCV	7-7-04	[illegible]	7-21-04	2.5 mL	Std 1605	1000 mL

WCStdPrep.xls

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TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

FIGURE 2

EXAMPLE OF REAGENT PREPARATION LOGBOOK PAGE

Katahdin Analytical Services, Inc. - Wet Chemistry Lab
REAGENT PREP LOGBOOK

Katahdin Reagent ID	Reagent Name	Prep Date	Exp. Date	Prep Initials	Component	ID or Lot Number	Amount Added	Prep Notes
W4959	IODINE FOR Se	7-12-04	7-12-05	[illegible]	IODINE	SWL1557	6.4g	
↓	↓	↓	↓	↓	KI	SWL1567	492g	
W4960	Sodium Nitroprusside	7.12.04	1.12.05	PAG	Sodium Nitroprusside	SWL1430	3.5g	Dissolve in 1L DI
W4961	NH3 Buffer	7.12.04	7.19.04	PAG	EDTA	SWL1001	25g	Dissolve in 500mL
↓	↓	↓	↓	↓	NaOH	SWL1576	4.5g	DI
W4962	NH3 Carrier	7.12.04	7.12.05	PAG	H2SO4	SWL1511	4ml	Mix in 2L DI
W4963	Sodium Phenolate	7.12.04	7.14.04	PAG	phenol	SWL1187	22ml	Dissolve in 250ml DI
↓	↓	↓	↓	↓	NaOH	SWL1576	8g	—
W4964	HIGH LEVEL ACID (QCU)	7.12.04	7.26.04	[illegible]	SOD. CARB	SWL1605	12.5mL	Dissolve in 500mL DI
W4965	Phosphate Buffer	7.14.04	8.14.04	PAG	NaH2PO4·H2O	SWL1556	35.5g	Dissolve in 250ml DI
W4966	NO3 Buffer	7.14.04	8.14.04	PAG	HCl	A16039	210ml	Dissolve in 2L DI
					NH4OH	6665 KD KE	190ml	—
↓	↓	↓	↓	↓	EDTA	SWL1001	2g	—

WCReagPrep.xls

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TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

FIGURE 3

EXAMPLE OF AUTOSAMPLER TABLE

	Type	Analysis	Sample Name	Dilution	Comment	Vial
1	Control	NPOC	blank	1.000		1
2	Control	NPOC	ccv	1.000		2
3	Control	NPOC	lcs	1.000		3
4	Unknown	NPOC	alkalinity check	1.000		4
5	Unknown	NPOC	ALKALINITY	1.000		4
6	Unknown	NPOC	WW1798-1	1.000		5
7	Unknown	NPOC	WW1798-1DUP	1.000		5
8	Unknown	NPOC	WW1798-1MS	1.000		6
9	Unknown	NPOC	WW1590-1	1.000		7
10	Unknown	NPOC	WW1501-12	1.000		8
11	Unknown	NPOC	WW1719-5	1.000		9
12	Unknown	NPOC	WW1719-6	10.00		10
13	Unknown	NPOC	WW1719-7	1.000		11
14	Unknown	NPOC	WW1719-8	1.000		12
15	Unknown	NPOC	WW1725-1	1.000		13
16	Control	NPOC	blank	1.000		1
17	Control	NPOC	ccv	1.000		2
18	Unknown	NPOC	WW1725-2	1.000		14
19	Unknown	NPOC	WW1725-3	1.000		15
20	Unknown	NPOC	WW1725-4	1.000		16
21	Unknown	NPOC	WW1725-5	1.000		17
22	Unknown	NPOC	WW1725-6	1.000		18
23	Unknown	NPOC	WW1725-7	1.000		19
24	Unknown	NPOC	WW1725-8	1.000		20
25	Unknown	NPOC	WW1725-9	1.000		21
26	Unknown	NPOC	WW1725-10	1.000		22
27	Unknown	NPOC	WW1725-11	1.000		23
28	Control	NPOC	blank	1.000		1
29	Control	NPOC	ccv	1.000		2
30	Unknown	NPOC	WW1725-12	1.000		24
31	Unknown	NPOC	WW1798-2	1.000		25
32	Unknown	NPOC	WW1798-2MS	1.000		26
33	Control	NPOC	blank	1.000		27
34	Control	NPOC	ccv	1.000		28

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

FIGURE 4

EXAMPLE OF RUN DATA SUMMARY TABLE

	Type	Analysis	Sample Name	Dilution	Result	Comment	Date / Time
1	Control	NPOC	blank	1.000	NPOC:0.4635 mg/L		04/21/06 03:59:04 PM
2	Control	NPOC	ccv	1.000	NPOC:89.50 mg/L		04/21/06 04:10:08 PM
3	Control	NPOC	lcs	1.000	NPOC:46.01 mg/L		04/21/06 04:20:57 PM
4	Unknown	NPOC	alkalinity check	1.000	NPOC:0.000 mg/L		
5	Unknown	NPOC	ALKALINITY	1.000	NPOC:0.3675 mg/L		04/21/06 04:42:04 PM
6	Unknown	NPOC	WW1798-1	1.000	NPOC:0.7568 mg/L		04/21/06 05:25:44 PM
7	Unknown	NPOC	WW1798-1DUP	1.000	NPOC:0.5154 mg/L		04/21/06 05:33:08 PM
8	Unknown	NPOC	WW1798-1MS	1.000	NPOC:99.71 mg/L		04/21/06 05:44:00 PM
9	Unknown	NPOC	WW1590-1	1.000	NPOC:2.994 mg/L		04/21/06 05:53:37 PM
10	Unknown	NPOC	WW1501-12	1.000	NPOC:0.3184 mg/L		04/21/06 06:02:50 PM
11	Unknown	NPOC	WW1719-5	1.000	NPOC:3.710 mg/L		04/21/06 06:12:28 PM
12	Unknown	NPOC	WW1719-6	10.00	NPOC:675.9 mg/L		04/21/06 06:23:01 PM
13	Unknown	NPOC	WW1719-7	1.000	NPOC:315.6 mg/L		04/21/06 06:34:05 PM
14	Unknown	NPOC	WW1719-8	1.000	NPOC:37.11 mg/L		04/21/06 06:44:07 PM
15	Unknown	NPOC	WW1725-1	1.000	NPOC:5.132 mg/L		04/21/06 06:55:56 PM
16	Control	NPOC	blank	1.000	NPOC:0.2845 mg/L		04/21/06 07:05:19 PM
17	Control	NPOC	ccv	1.000	NPOC:90.08 mg/L		04/21/06 07:16:48 PM
18	Unknown	NPOC	WW1725-2	1.000	NPOC:1.626 mg/L		04/21/06 07:26:33 PM
19	Unknown	NPOC	WW1725-3	1.000	NPOC:1.493 mg/L		04/21/06 07:36:03 PM
20	Unknown	NPOC	WW1725-4	1.000	NPOC:4.154 mg/L		04/21/06 07:45:49 PM
21	Unknown	NPOC	WW1725-5	1.000	NPOC:1.624 mg/L		04/21/06 07:55:23 PM
22	Unknown	NPOC	WW1725-6	1.000	NPOC:1.538 mg/L		04/21/06 08:04:53 PM
23	Unknown	NPOC	WW1725-7	1.000	NPOC:2.217 mg/L		04/21/06 08:14:36 PM
24	Unknown	NPOC	WW1725-8	1.000	NPOC:4.974 mg/L		04/21/06 08:24:28 PM
25	Unknown	NPOC	WW1725-9	1.000	NPOC:0.5274 mg/L		04/21/06 08:33:52 PM
26	Unknown	NPOC	WW1725-10	1.000	NPOC:0.7416 mg/L		04/21/06 08:43:19 PM
27	Unknown	NPOC	WW1725-11	1.000	NPOC:0.3847 mg/L		04/21/06 08:52:41 PM
28	Control	NPOC	blank	1.000	NPOC:0.1905 mg/L		04/21/06 09:01:57 PM
29	Control	NPOC	ccv	1.000	NPOC:89.85 mg/L		04/21/06 09:13:07 PM
30	Unknown	NPOC	WW1725-12	1.000	NPOC:3.528 mg/L		04/21/06 09:23:07 PM
31	Unknown	NPOC	WW1798-2	1.000	NPOC:0.6877 mg/L		04/21/06 09:32:32 PM
32	Unknown	NPOC	WW1798-2MS	1.000	NPOC:99.48 mg/L		04/21/06 09:43:39 PM
33	Control	NPOC	blank	1.000	NPOC:0.2665 mg/L		04/21/06 09:53:07 PM
34	Control	NPOC	ccv	1.000	NPOC:87.25 mg/L		04/21/06 10:04:19 PM

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

FIGURE 5

EXAMPLE OF TOC BATCH SHEET (FROM KIMS)

WET CHEMISTRY BATCH REPORT
Apr 27 2006, 08:20 am
Batch: WG27364

Parameter: Total Organic Carbon

Prep Date: N/A

Date Analyzed: 21-APR-06

Prep Method: N/A

Analyst Initials: CP

Prep Chemist: N/A

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
WG27364-1	MBLANK	EPA 415.1	20.000mL	20.000mL	1	.4635	U1.0 mg/L	NA	1	.1023	1		
WG27364-2	LCS	EPA 415.1	20.000mL	20.000mL	1	46.01	46. mg/L	NA	1	.1023	1		92
WG27364-3	DUP	EPA 415.1	20.000mL	20.000mL	1	.5154	U1.0 mg/L	NA	1	.1023	1	NC	
WG27364-4	MS	EPA 415.1	20.000mL	20.000mL	1	99.71	100 mg/L	NA	1	.1023	1		99
WG27364-5	MS	EPA 415.1	20.000mL	20.000mL	1	99.48	99. mg/L	NA	1	.1023	1		99
WW1501-12	SAMP	EPA 415.1	20.000mL	20.000mL	1	.3104	U1.0 mg/L	NA	1	.1023	1		
WW1719-5	SAMP	EPA 415.1	20.000mL	20.000mL	1	3.71	3.7 mg/L	NA	1	.1023	1		
WW1719-6	SAMP	EPA 415.1	20.000mL	20.000mL	10	675.9	680 mg/L	NA	1	1	10		
WW1719-8	SAMP	EPA 415.1	20.000mL	20.000mL	1	37.11	37. mg/L	NA	1	.1023	1		
WW1725-1	SAMP	EPA 415.1	20.000mL	20.000mL	1	5.132	5.1 mg/L	NA	1	.1023	1		
WW1725-10	SAMP	EPA 415.1	20.000mL	20.000mL	1	.7416	U1.0 mg/L	NA	1	.1023	1		
WW1725-11	SAMP	EPA 415.1	20.000mL	20.000mL	1	.3847	U1.0 mg/L	NA	1	.1023	1		
WW1725-12	SAMP	EPA 415.1	20.000mL	20.000mL	1	3.528	3.5 mg/L	NA	1	.1023	1		
WW1725-2	SAMP	EPA 415.1	20.000mL	20.000mL	1	1.626	1.6 mg/L	NA	1	.1023	1		
WW1725-3	SAMP	EPA 415.1	20.000mL	20.000mL	1	1.493	1.5 mg/L	NA	1	.1023	1		
WW1725-4	SAMP	EPA 415.1	20.000mL	20.000mL	1	4.154	4.2 mg/L	NA	1	.1023	1		
WW1725-5	SAMP	EPA 415.1	20.000mL	20.000mL	1	1.624	1.6 mg/L	NA	1	.1023	1		
WW1725-6	SAMP	EPA 415.1	20.000mL	20.000mL	1	1.538	1.5 mg/L	NA	1	.1023	1		
WW1725-7	SAMP	EPA 415.1	20.000mL	20.000mL	1	2.217	2.2 mg/L	NA	1	.1023	1		
WW1725-8	SAMP	EPA 415.1	20.000mL	20.000mL	1	4.974	5.0 mg/L	NA	1	.1023	1		
WW1725-9	SAMP	EPA 415.1	20.000mL	20.000mL	1	.3274	U1.0 mg/L	NA	1	.1023	1		
WW1798-1	SAMP	EPA 415.1	20.000mL	20.000mL	1	.7568	U1.0 mg/L	NA	1	.1023	1		
WW1798-2	SAMP	EPA 415.1	20.000mL	20.000mL	1	.6877	U1.0 mg/L	NA	1	.1023	1		

Comments:

WG27364-1	WW1798-1
WG27364-2	WW1798-1
WG27364-3	WW1798-1
WG27364-4	WW1798-1
WG27364-5	WW1798-2
WW1590-1	Limited Volume
WW1725-1	only one vial for analysis
WW1725-10	only one vial for analysis
WW1725-11	only one vial for analysis
WW1725-12	only one vial for analysis
WW1725-2	only one vial for analysis
WW1725-3	only one vial for analysis
WW1725-4	only one vial for analysis
WW1725-5	only one vial for analysis
WW1725-6	only one vial for analysis
WW1725-7	only one vial for analysis
WW1725-8	only one vial for analysis

Entered by: _____ Date: 04/27/06 Accepted by: _____ Date: 04/27/06

TITLE: **ANALYSIS OF TOC, DOC, AND TIC IN AQUEOUS SAMPLES USING THE SHIMADZU CARBON ANALYZER: EPA METHOD 415.1, SW846 9060 AND SM 5310B**

FIGURE 6

EXAMPLE OF CARBON ANALYSIS RUN INFORMATION SHEET

KATAHDIN ANALYTICAL SERVICES, INC.
CARBON ANALYSIS RUN INFORMATION SHEET

INSTR. ID: WC2 (Shimadzu TOC-V_{CPH}) **ANALYST:** AMB **DATE:** 01/30/07

FILE NAME: TOC013007 **METHOD(S):**

TOC	DOC	TIC
EPA 415.1	EPA 415.1	EPA 415.1
SM5310B	SM5310B	SM5310B
_____	_____	_____

CALIBRATION DATE: 12/15/06 **CALIBRATION ANALYST:** CP

Note: Calibration must be performed quarterly or whenever a change in analysis conditions warrant. A copy of the associated calibration data is attached to this run.

STANDARDS USED:

Standard Name	Standard ID	Prep Date	Expiration Date	Standard Conc.
CCV	W6Z74	12/14/06	03/14/07	100 mg C/L
LCS	W6Z17	10/30/06	01/30/07	50 mg C/L
MS Stock Std. *	W6Z69	12/14/06	03/14/07	2000 mg C/L
Alkalinity Check	SWL 1991	11/13/06	11/13/07	15 mg/L (Inorg. C)

* Matrix spikes are prepared by adding 2.0 mL of MS Stock Std. to 38 mL of sample.

Additional Comments and Notes:

01/30/07

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: SD-902
Revision History
Cover Page
Page 1

TITLE: **SAMPLE RECEIPT AND INTERNAL CONTROL**

Prepared By: Andrea Colby Date: 6/2002

Approved By:

Group Supervisor: Andrea Coy Date: 6/6/02

Lab Operations Mgr: Joel C. Burton Date: 6/5/02

QA Officer: Deborah J. Nadeau Date: 6.6.02

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
04	Changed cover sheet, minor changes to sections 7.1, 7.6 7.7.4, 7.10 + 7.20. Complete rewrite of sections 7.11 + 7.12 to comply with new KIMS	DN	6.6.02	6.6.02
05	Added verbal date entry to KIMS. Added reference to immediate internal COC book. Added Department Manager reference. Added section 7.7.3. Updated new incoming	DN	05.04	05.04
06	Added procedure + Logbook page for checking turbidity of drinking water samples. Changed wet chem shorts board to a book (included example page). Added custody procedures for food/micro. Added VOA soil freezer storage.	DN	01.26.04	01.26.04
07	Added instructions to create lettered labels. Changed sample locations to reflect new building. Removed figures 8 and 10. Updated Table and figures w/current ones. Added wording to Sect. 7.7.5 to clarify how pH measurements are taken.	LAD	02/07	02/07

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-902-07**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-902-07**, 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 isdetected or suspected, initiating corrective action when needed, documenting any actions taken, and notifying the appropriate individual (e.g., Department Manager, Operations Manager, QAO). The primary responsibility for implementing real-time corrective actions and maintaining an effective QA self-inspection system resides with Katahdin staff. When problems are identified, Katahdin personnel are expected to attempt to resolve situations within the scope of their technical knowledge, and to seek assistance from peers and the Department Manager as necessary.

It is the responsibility of Department Managers to oversee the adherence to Katahdin QC practices and internal documentation of laboratory activities within their area, to take corrective actions where needed and communicate problems to the Operations Manager, QAO or Vice President/President when warranted.

It is the responsibility of the Operations Manager to oversee adherence to Katahdin QA/QC practices by all laboratory groups under his/her authority, to help identify problems and assure resolution, 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 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 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 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

Not applicable.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

4.1 Radiation monitor (when required)

4.2 Thermometer – Digital probe style capable of reading 0.1°C.

4.3 Capillary tubes – 75 mm Hematocrit Tubes, disposable

4.4 Wide range pH test strips, pH 0 to 14pH, EMD ColorpHast or equivalent.

4.5 Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.

4.6 Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.

5.0 REAGENTS

Preservatives - refer to Table 1, Sampling and Preservation Requirements, for specifics.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Refer to Table 1, Sampling and Preservation Requirements, for specifics.

7.0 PROCEDURES

REQUIREMENTS FOR USE OF RADIATION MONITOR

7.1 Samples originating from USACOE sites and previously determined radioactive sites need to be screened for radioactivity upon receipt at the lab as a requirement of the program governing the site.

7.2 One radiation scanning device is available for screening samples for radiation. The meter is calibrated with a standard of known radioactivity on an annual frequency. A back-up meter is stored by the QAO.

7.3 A "hot" sample is defined as any sample that causes the radiation scanning device to read a level above the control limit for background radiation. The control limit for background radiation in the sample receiving area should be statistically determined annually by taking a series of measurements with both meters. Because some subjectivity is involved in reading the meters, at least two individuals should participate in the study.

7.3.1 Measure and record a minimum of seven background readings.

7.3.2 Calculate the mean and standard deviation of the background readings.

7.3.3 Calculate the control limit for background radiation as X + 3s, where X is the mean and s is the standard deviation of the background readings.

7.3.4 Maintain documentation that includes the calculated background control limit and the date the background radiation study was performed.

PROCEDURES FOR SAMPLE CUSTODIAN

7.4 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 or delivery service shall sign that they have relinquished custody to the laboratory. The laboratory shall sign and record the date and time that custody is accepted. (Refer to Figure 1 for Katahdin COC)

7.5 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 2). Initial each entry (line) to maintain a record of the individual who assigned each group of samples a discreet lab work order/login number.

7.6 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 3). The project manager is notified of any discrepancies or problems with sample receipt. The PM contacts the client as necessary. If breakage of a potentially hazardous sample is discovered, the packing container with all the samples inside is

closed, sealed, and moved 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 is 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 Chemical Hygiene Plan and Safety Manual and the Katahdin Hazardous Waste Management Program. 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.

7.7 If there is no breakage of a potentially hazardous sample:

7.7.1 If applicable, check samples for radiation emission by passing a calibrated radiation monitor over the samples; the meter should be moved slowly and deliberately across the sample containers, maintaining a distance of about 3-5 inches from the sample containers. Contact the safety officer or the Operations Manager immediately for further instructions if the meter displays a reading above background level as defined in Procedure Section 7.3 above. Otherwise, proceed with Section 7.7.2.

7.7.2 Check cooler temperatures using the thermometer assigned to the sample receipt area. If a cooler temperature blank is present, insert the thermometer in the temperature blank; otherwise place the thermometer in the cooler as close to a sample as possible. Once the thermometer reading has stabilized, record the temperature on the Sample Receipt Condition Report. Cooler temperatures should be 4° C ± 2° C. Any temperature falling outside of this range must be noted on the SRCR and reported to the appropriate Katahdin project manager.

7.7.3 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, 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.

7.7.4 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.

7.7.5 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. For metals and wet chemistry parameters, additional preservative is added 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, date and time added, final pH and custodian's initials must be placed on the sample container.

Note: Preservatives are obtained from the larger containers in the bottle preparation room. Fill smaller 100 mL or 250 mL containers and record the lot number on the bottle. These containers should be kept in the hood in the sample receipt area.

7.7.6 For samples requiring filtration as pretreatment (i.e. for dissolved metals), the work order/login numbers are recorded in the filtration logbook (see Figure 4). The samples are filtered by the Metals Group.

7.7.6.1 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.

7.7.6.2 A vacuum pump is attached.

7.7.6.3 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.

7.7.6.4 A sufficient sample aliquot is filtered and preserved with concentrated nitric acid.

7.7.6.5 The bottles are labeled with the work order/login number and other sample information and stored at 4° C until the time of digestion.

7.7.7 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.

7.7.8 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 5).

7.7.9 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.

7.8 Review any 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. (examples included as Figures 6 - 8).

7.9 Resolve any questions or concerns raised by steps 7.1-7.8 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).

7.10 Notify appropriate section managers of any "RUSH" analyses or quick hold times. List any samples for analyses that have short hold times in the "Wet Chemistry Shorts and Rushes Logbook" (Figure 9) in the wet chemistry laboratory. Be sure to list the client, number of samples and date and time of the earliest sample. GC or GC/MS personnel must be informed when ENCORES are received so that they may be scheduled for extrusion. Microbiology personnel should also be informed of any microbiology samples that arrive. Parameters that routinely require short analytical hold times are:

Coliforms	Color	pH
Nitrate/Nitrite	Dissolved Oxygen	Turbidity
Ferrous iron	Orthophosphate	Hex. Chromium
MBAS	BOD	Free CO_2
Sulfite	ENCORE soil samples	

7.11 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.

7.12 The following information is documented via the Katahdin Information Management System (KIMS) and a work order/login COC report (Figure 10) is generated for the samples received:

7.12.1 Log onto KIMS by entering employee ID under "Username", employee specific password under "Password" and KIMS under "Database".

7.12.2 Once logged onto KIMS select "Sample Management" and then "Login".

7.12.3 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.

7.12.4 In the Sample Definition Screen, enter the following information.

Client ID -	Enter client sample description.
ReceiveDate -	Enter in date samples were received in the lab in the format YY-Month-DD.
CollectDate -	Enter in date samples were collected in the format YY-Month-DDTIME.
TAT -	Enter TAT for hardcopy report.
DueDate -	Due date will automatically be calculated based on calendar days.
VerbalDate -	Manually type in verbal due date.

QuoteRef -	Enter quote number if applicable.
Project -	Enter project number if applicable.
Account -	Enter client specific account number.
Account Name -	Account name will automatically be entered.
Collected By -	Enter name/initials of sampler listed on COC. If unknown, enter "Client".
Locator -	May be used for client ID information when requested by the project manager.
Site -	Enter project site name.
Description -	May be used for long client Ids when requested by the project manager.
Discount -	No entry-not currently used.
Priority -	No entry-not currently used.
Fact. -	No entry-not currently used.
Expected -	No entry-not currently used.
Comments -	Enter MS/MSD, verbal due date and any sample irregularities if applicable.
OrderDate -	Current date is automatically entered.
Matrix -	Enter sample matrix code where

AQ = Aqueous	SLD = Food Solid
SL = Solid, Soil, Sludge	AR = Air
FP = Free Product	SWAB = Swab
WP = Wipe	SAL = Saline
NOAQ = NonAqueous	TIS = Tissue
DW = Drinking Water	

Product Code -	Enter analysis code per test requested on COC.

Type -	Product code type will automatically be entered where S = Stand alone P = Parent C = Children
Fact. -	No entry-default is 1.
Price -	This is left as is by sample log-in. During project management review of the work order, the prices are entered based on quotes or standard prices.
Cost -	No entry needed.
Lev -	No entry needed.
Type -	Container type will automatically be entered.
Bot -	Enter number of containers per test for printing of labels.
Login Info -	Parameter Data Screen will open. Enter following information

KAS Proj. Manager-	Initials of Katahdin person overseeing the project.
Client PO#-	Client purchase order.
Project-	Project name.
Cooler Temperature-	Temperature blanks or cooler temps.
Delivery Services-	Method of delivery to the lab.
QC Level-	QC Level of report and regulatory agency (ie., IV NFESC).
SDG ID-	Sample Delivery Group ID if applicable.
SDG Status-	Begin, Continue or End.
Analysis Instructions-	PM will enter special instructions regarding project.
Report Instructions-	PM will enter special instructions regarding project.
Regulatory List-	Used for federal programs.
EDD Format-	Specific KAS EDD format.

Select "SAVE" and then "CANCEL".

Addresses - Select "Addresses" and the Address Links screen will open. The billing address is the default address of the account. Enter the client account code under "Project/Account" and select the report to contact under "Address Type". Select the appropriate boxes for report, report CC and invoice CC. Select "SAVE" and then "CLOSE".

Create Containers - Select "Create Containers". Letters will be assigned to each sample number. Select "OK" until letters have been assigned to each sample number. To manually assign letters, Select "Enter Container IDs" and "OK". Enter sample numbers including letters and select "OK", "Close", "Yes" to save changes, "Cancel" and "Cancel".

7.12.5 To print the login report, select "Reports", "Login" and "Login COC". Enter login number under "Login Number". Select "OK", "Run Report" and then "Print".

7.13 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".

7.14 Affix permanent sample number labels to sample containers, assuring that sample IDs on labels correspond to sample bottle IDs. Mark any known hazardous samples with appropriate warning labels. Do not obscure client ID on the bottles.

7.15 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 11). Place form in the appropriate binder in the log in area. Non-environmental food samples do not get an internal COC and are taken immediately to the food/microbiology lab for storage.

Storage location of the samples is determined by type of sample and/or type of analysis, as outlined below. Most samples are stored in the walk-in cooler, which is organized by test type and work order/login number.

Specific storage locations are described below. NOTE: Samples originating in the State of North Carolina must be stored in locations maintaining a storage temperature of 1 - 4.4 °C.

1. Aqueous samples for wet chemistry (except hardness, see Step 4 below) - left aisle as you enter walk-in cooler. TOC vials are to be stored in the trays designated for TOC samples (orange row).

2. Aqueous samples for organic extractions – right aisle as you enter walk-in cooler.

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 the "white" refrigerator located Volatiles Laboratory.

   Aqueous samples for metals and/or hardness analyses – right and left aisles towards the front as you enter walk-in cooler.

4. 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 the "blue" refrigerator in the Volatiles Laboratory. VOA soils in jars or ENCORE samplers are stored in the "white" refrigerator 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.

5. Soil samples for volatile organics analyses (VOA) that are unpreserved or preserved with Laboratory Reagent Grade Water are stored in the freezer across from the wet chemistry laboratory.

7.16 Sample Receipt gives the Work order/login COC report and confirmation of the job, as logged-in, to the appropriate Katahdin project manager. All chain-of-custody and other receipt documentation must accompany the job. The project manager reviews the job for accuracy and completeness. Any unresolved issues should be resolved at this time. 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.

7.17 Project or Program-specific log-in forms are completed as needed and submitted to the Project Manager with the original Work order/login package. These forms may include any or all of the following (Figures 3 and 8).

Sample Receipt Condition Report
non-EPA CLP DC-1 Form

7.18 Complete the sample log-in notebook for "Time Logged In".

7.19 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 12).

PROCEDURES FOR CHEMISTS

7.20 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.

7.21 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 13) 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.

7.22 If 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.

7.23 If analysis consumes the entire sample, indicate this on the laboratory internal chain-of-custody record.

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 yearly 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., Chemical Hygiene Plan and Safety Manual, current revision.

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TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES					
Acidity	305.1	100 mL	P,G	1,2	14 days
Alkalinity-Manual Titrimetric	310.1	100 mL	P,G	1,2	14 days
Ammonia-Nitrogen with distill-Auto. Phenate	350.1	1 L	P,G	1,3	28 days
Ammonia-Nitrogen-Automated Phenate	350.1, 350.2	250 mL	P,G	1,3	28 days
Anions (Cl, Br, SO4, NO2, NO3)	300.0	250 mL	P, G	1	48hr/28days
Bicarbonate, Carbonate (see pH & alkalinity)	calc.				
Biochemical Oxygen Demand-Carbonaceous	405.1	1 L	P,G	1	48 hours
Biochemical Oxygen Demand-Total	405.1	1 L	P,G	1	48 hours
Bromide	320.1	500 mL	P,G	1	28 days
Chemical Oxygen Demand-Manual Colorimetric	410.4	100 mL	P,G	1,3	28 days
Chloride-Automated Ferricyanide	325.2	100 mL	P,G	1	28 days
Chlorine, Residual	SM4500-Cl G	100 mL	P,G	1,9	ASAP
Chromium, Hexavalent	SM3500Cr D / SW7196	200 mL	P,G	1,9	24 hours
Color, Apparent	110.2	100 mL	P,G	1,2	48 hours
Cyanide, Amenable-Spectrophotometric	335.1	250 mL	P,G	1,5	14 days
Cyanide, Total-Spectrophotometric	SM4500CN C 335.3, 335.4	250 mL	P,G	1,5	14 days
Dissolved Oxygen(Lab)-Membrane Electrode	360.1	500 mL	G	1	ASAP
Ferrous Iron - Colorimetric	SM3500-Fe D	250mL	P	1	24 hrs
Fluoride with distillation, Potentiometric ISE	SM4500F C/340.2	500 mL	P only	1	28 days
Fluoride, Potentiometric ISE	340.2	200 mL	P only	1	28 days
Free CO_2	SM4500-CO_2 C	250mL	P	1	24 hrs.
Hardness, Total-Manual Titrimetric	130.2,SM2340C	250 mL	P,G	4	6 months
MBAS, Extraction-Colorimetric	SM5540C	1 L	P,G	1	48 hours
Nitrate+Nitrite-Automated Cadmium Reduction	353.2	100 mL	P,G	1,3	28 days
Nitrate-Automated Cadmium Red./Diazotization	353.2	100 mL	P,G	1	48 hours
Nitrite-Automated Diazotization	353.2	100 mL	P,G	1	48 hours
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	1664	(2) 1 L	glass only	1,11	28 days
pH (Laboratory)	150.1	100 mL	P,G	1,2	24 hours
Phenolics, Total Recoverable-Manual 4AAP	420.1	1000 mL	glass only	1,3	28 days
Phosphate, Ortho- Ascorbic Acid	365.2	100 mL	P,G	1	48 hours
Phosphate,Total	365.4	100 mL	P,G	1,3	28 days
Solids-Filterable Residue (TDS),Gravimetric180	160.1	250 mL	P,G	1	7 days
Solids-Nonfilterable Residue (TSS)	160.2	500 mL	P,G	1	7 days
Solids-Settleable Solids (SS)	160.5	1 L	P,G	1	48 hours

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES					
Solids-Total Solids	160.3	250 mL	P,G	1	7 days
Solids-Total Volatile (TVS)	160.4	250mL	P,G	1	7 days
Solids-Volatile Filterable Residue (VDS)	160.1/160.4	250 mL	P,G	1	7 days
Solids-Volatile Nonfilterable Residue (VSS)	SM 2540 F	500 mL	P,G	1	7 days
Specific Conductance-Wheatstone Bridge	120.1	100 mL	P,G	1,2	28 days
Sulfate-Turbidimetric	375.4	100 mL	P,G	1	28 days
Sulfide-Iodometric	376.1	500 mL	P,G	1,7	7 days
Sulfite-Titrimetric	377.1	500 mL	P,G	1,9	ASAP
Tannin/Lignin-Colorimetric	SM 5550 B	100 mL	P,G	1	7 days
TKN-Auto Block Digest, Spect.	351.2	100 mL	P,G	1,3	28 days
Total Inorganic Carbon	415.1	(2) 40 mL	VOA vial	1	28 days
Total Inorganic Carbon if with TOC	415.1	(2) 40 mL	VOA vial	1	28 days
Total Organic Carbon-Oxidation	415.1	(2) 40 mL	VOA vial	1,3	28 days
Total Organic Halogen	9020	500 mL	Amber Glass	1,3	28 days
Turbidity	180.1	100 mL	P,G	1	48 hours
ELEMENTAL ANALYSES					
Chromium, Hexavalent	7196/6010	500 mL	P,G	1,9	24 hrs
GFAA(Furnace) Elements	SM 3113/ 200 series	500 mL	P,G	4	6 months
ICP Elements	200.7/6010	500 mL	P,G	4	6 months
ICP MS Elements	200.8/6020	500 mL	P,G	4	6 months
Low Level Mercury	1631	500 mL	G	NA	90 days
Mercury	245.1/7470	500 mL	P,G	4	28 days
GC ORGANIC ANALYSES					
BTEX & MTBE	602 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
EDB, DBCP & 1,2,3-TCP	504.1	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Extractable Petroleum Hydrocarbons	MADEP/EPH	(2) 1000 mL	Amber Glass	12	14days/40days
Formaldehyde	556	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Fuel Oil in Water	8015Modified	(2) 1000 mL	Amber Glass	1,8	7days/40days
Fuel Oil in Water	ME HETL 4.1.25	(2) 1000 mL	Amber Glass	1,8	7days/40days
Gasoline in Water	8015Modified	(2) 40 mL	VOA vial	1,8	14 days
Gasoline in Water	ME HETL 4.2.17	(2) 40 mL	VOA vial	1,8	14 days
Glycols	8015Modified	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Herbicides	8151	(2) 1000 mL	Amber Glass	1	7days/ 40days
Methane, Ethane & ethene	RSK 175	(2) 40 mL	VOA vial	1,8,9	14 days(~)
PCB's	608 & 8082	(2) 1000 mL	Amber Glass	1	7days/40days

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

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	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	100 mL	P,G	1,6	30 hours
E-coli	SM9213D	100 mL	P,G	1,6	6 hours
Heterotrophic Plate Count	SM9215B	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	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 1

EXAMPLE OF KATAHDIN CHAIN-OF-CUSTODY FORM

Katahdin
600 Technology Way
Scarborough, ME 04074
Tel: (207) 874-2400
Fax: (207) 775-4029

CHAIN of CUSTODY

PLEASE BEAR DOWN AND PRINT LEGIBLY IN PEN

Page ____ of ____

Client | Contact | Phone # ( ) | Fax # ( )

Address | City | State | Zip Code

Purchase Order # | Proj. Name / No. | Katahdin Quote #

Bill (if different than above) | Address

Sampler (Print / Sign) | Copies To:

LAB USE ONLY | WORK ORDER #:

KATAHDIN PROJECT NUMBER _____

REMARKS: _____

SHIPPING INFO: ☐ FED EX ☐ UPS ☐ CLIENT

AIRBILL NO: _____

TEMP'C _____ ☐ TEMP BLANK ☐ INTACT ☐ NOT INTACT

ANALYSIS AND CONTAINER TYPE PRESERVATIVES

Sample Description	Date / Time coll'd	Matrix	No. of Cntrs.	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N
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COMMENTS

Relinquished By: (Signature)	Date / Time	Received By: (Signature)	Relinquished By: (Signature)	Date / Time	Received By: (Signature)
Relinquished By: (Signature)	Date / Time	Received By: (Signature)	Relinquished By: (Signature)	Date / Time	Received By: (Signature)

THE TERMS AND CONDITIONS ON THE REVERSE SIDE HEREOF SHALL GOVERN SERVICES, EXCEPT WHEN A SIGNED CONTRACTUAL AGREEMENT EXISTS

ORIGINAL

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 2

EXAMPLE OF KATAHDIN SAMPLE RECEIPT LOGBBOK

KATAHDIN ANALYTICAL SERVICES, INC.

SAMPLE LOG IN

Date Received	Time Received	Date Logged In	Time Logged In	Work Order	Client	Initials
				SA 0094		
				SA 0095		
				SA 0096		
				SA 0097		
				SA 0098		
				SA 0099		
				SA 0100		
				SA 0101		
				SA 0102		
				SA 0103		
				SA 0104		
				SA 0105		
				SA 0106		
				SA 0107		
				SA 0108		
				SA 0109		
				SA 0110		
				SA 0111		
				SA 0112		
				SA 0113		
				SA 0114		
				SA 0115		
				SA 0116		
				SA 0117		
				SA 0118		
				SA 0119		
				SA 0120		
				SA 0121		
				SA 0122		
				SA 0123		
				SA 0124		

Signed By:_____ Date:_____

Reviewed By:_____ Date:_____

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 3

EXAMPLE OF SAMPLE RECEIPT CONDITION REPORT FORM

Katahdin Analytical Services, Inc. **Sample Receipt Condition Report**

Client:	SDG #:	Cooler: _____ of _____
Project:	KAS PM:	Sampled By:
COC#:	KIMS Entry By:	Delivered By:
KAS Work Order#:	KIMS Review By:	Received By:

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?					Temp (°C):
6. Samples received at < 6 °C w/o freezing? Ice or ice packs present? Y or N					Cooler temp. (°C): (if no temp blank)
7. Volatiles free of headspace?					
Aqueous: No bubble larger than a pea					
Soil/Sediment: Received in airtight container?					
Received in methanol?					
Methanol covering soil?					
8. Trip Blank present in cooler?					
9. Proper sample containers and volume?					
10. Samples within hold time upon receipt?					
11. Samples properly preserved? Metals, COD, NH3, TKN, O/G, phenol, TPO4, N+N, TOC, DRO, TPH – pH <2					
Sulfide - >9					
Cyanide – pH >12					
12. Corrective Action Report Filed?					

* Log-In Notes to Exceptions: document any problems with samples or discrepancies or pH adjustments

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 4

EXAMPLE OF KATAHDIN SAMPLE FILTRATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
Sample Filtration Logbook

Katahdin Sample No. List Individually	Site ID (Optional)	Filtration Requested By:		Filtered and Preserved By:		
		Initials	Date	Initials	Date	Time

Reviewed and Approved by: _____ **Date:** _____

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 5

MEASURED TURBIDITY AND PRESERVATION OF INCOMING SAMPLES LOGBOOK

KATAHDIN ANALYTICAL SERVICES

Measured Turbidity and Preservation of Incoming Samples

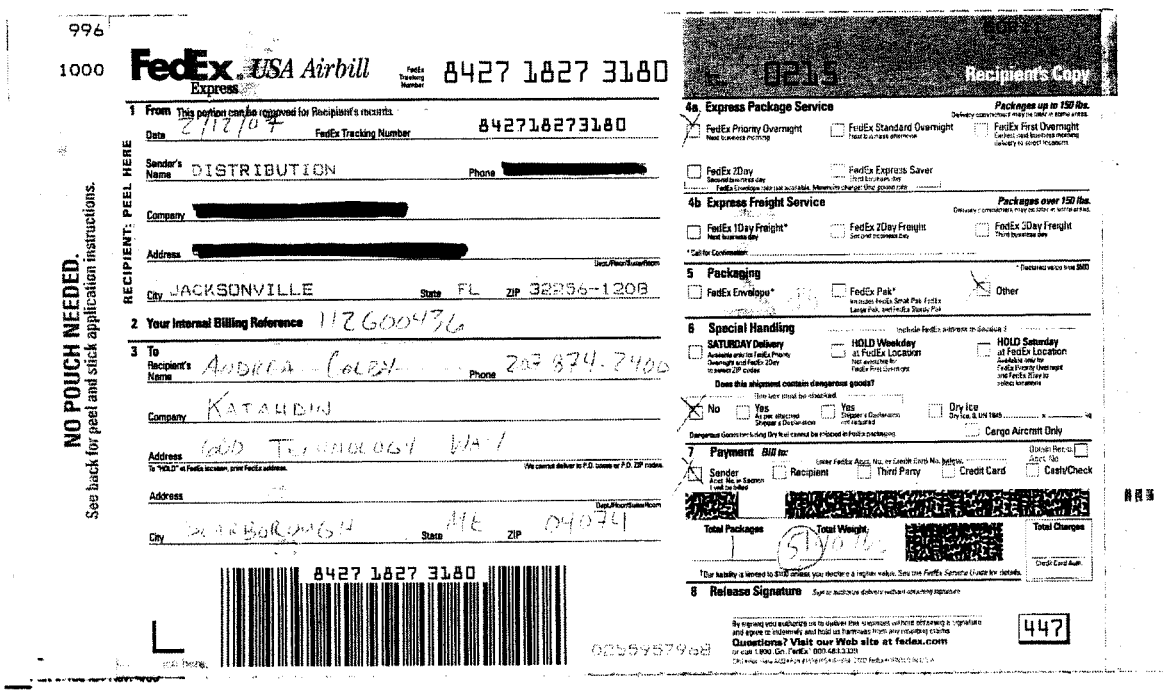
KAS Lab Sample ID	Measured Turbidity (NTU)	Turbidity Date	Turbidity Analyst	Preservation Date	Preservation Time	Preservation Analyst
WW6757-9A	0.22	12-19-06		12/14/06	~~1830~~ 1400	DM
↓ -10A	0.18	↓		↓	↓	↓
WW6758-1A	4.80	↓	↓	↓	↓	↓
WW6776-1A	0.68	12-20-06		12/20/06	1000	DM
WW6855-1A	0.92	12-26-06		12/26/06	1600	DM
WW6860-1A	2.40	↓	↓	↓	↓	↓
SA0027-1A	0.52	1-3-07		01/03/07	1445	DM
SA0028-1A	0.72	1-3-07		↓	↓	↓
SA0087-1A	0.26	1-5-07		01/05/07	1645	DM
SA0088-1A	10.4	1-5-07		↓	↓	↓
SA0110-1A	0.47	1-8-07		01/08/07	1430	DM
SA0109-1A	0.32	1-8-07		↓	↓	↓
SA0138-1A	0.12	1-08-07		01/09/07	1700	DM
SA0262-1A	0.14	1-18-07		01/18/07	0930	DM
SA0263-1A	0.33	1-18-07		↓	↓	↓
SA0267-1A	0.30	1-18-07		01/18/07	0950	DM
SA0313-1A	0.58	1-22-07		01/22/07	1030	↓
SA0316-1A	1.70	1-22-07		01/22/07	1345	DM

REVIEWED BY: DATE:

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 6

EXAMPLE SHIPPING FORM – FEDERAL EXPRESS AIRBILL



996

1000 FedEx Express USA Airbill 8427 1827 3180

1 From This portion can be removed for Recipient's records.
Date 2/12/07 FedEx Tracking Number 842718273180
Sender's Name DISTRIBUTION Phone [redacted]
Company [redacted]
Address [redacted]
City JACKSONVILLE State FL ZIP 32256-1208

2 Your Internal Billing Reference 112600436

3 To
Recipient's Name ANDREA COLEY Phone 207 874.2400
Company KATAHDIN
Address 600 TECHNOLOGY WAY
Address
City SCARBOROUGH State ME ZIP 04074

8427 1827 3180

NO POUCH NEEDED.
See back for peel and stick application instructions.

RECIPIENT: PEEL HERE

0215 Recipient's Copy

4a Express Package Service — Packages up to 150 lbs.
FedEx Priority Overnight
FedEx Standard Overnight
FedEx First Overnight
FedEx 2Day
FedEx Express Saver

4b Express Freight Service — Packages over 150 lbs.
FedEx 1Day Freight*
FedEx 2Day Freight
FedEx 3Day Freight

5 Packaging
FedEx Envelope*
FedEx Pak*
Other

6 Special Handling
SATURDAY Delivery
HOLD Weekday at FedEx Location
HOLD Saturday at FedEx Location
Does this shipment contain dangerous goods?
No
Yes
Dry Ice
Cargo Aircraft Only

7 Payment Bill to:
Sender
Recipient
Third Party
Credit Card
Cash/Check

Total Packages 1
Total Weight 5 lbs
Total Charges

8 Release Signature

Questions? Visit our Web site at fedex.com

447

025997968

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 7

EXAMPLE OF LABORATORY INCOMING SAMPLE INFORMATION SHEET (ISIS)

KATAHDIN ANALYTICAL SERVICES, INC. – INCOMING SAMPLE INFORMATION SHEET

CLIENT:	ATTENTION:
ADDRESS:	
TELEPHONE:	FAX:
OTHER CLIENT CONTACTS:	
REPORT CC:	PM/SALES CONTACT:
PO #:	CLIENT ACCOUNT:
KAS QUOTE #:	KIMS QUOTE #:
PROJECT NAME:	KIMS PROJECT REF.:
SDG:	APPLICABLE CERTIFICATION:
RECEIPT DATE:	MEANS OF DELIVERY:

METHOD OR KIMS PRODUCT	MATRIX	# OF SAMPLES	SPECIAL INSTRUCTIONS (I.E LIMITS, PREPS)

SUBCONTRACT PARAMETER	SUBCONTRACT LABORATORY

QC LEVEL:	EDD:
RUSH/VERBAL TAT:	HARDCOPY TAT:
HISTORY:	
OTHER:	

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 8

EXAMPLE OF NON-EPA CLP DC-1 FORM

SAMPLE LOG-IN SHEET

Lab Name: Katahdin Analytical Services, Inc. Page_____of_____

Received By (Print Name):_____ Log-in Date:_____

Received By (Signature):_____

Case Number: _____			CORRESPONDING		
Sample Delivery Group No.: _____ SAS Number:		CLIENT SAMPLE #	SAMPLE TAG #	ASSIGNED LAB #	REMARKS: CONDITION OF SAMPLE SHIPMENT, ETC.
REMARKS:					
1. Custody Seal(s)	Present/Absent*				
	Intact/Broken				
2. Custody Seal Nos.:	_____				
	_____				
3. Chain-of-Custody	Present/Absent*				
Records					
4. Traffic Reports or	Present/Absent*				
Packing List					
5. Airbill	Airbill/Sticker				
	Present/Absent*				
6. Airbill No.:	_____				
	_____				
7. Sample Tags	Present/Absent*				
Sample Tag	Listed/Not Listed				
Numbers	on Chain-of-				
	Custody				
8. Sample Condition:	Intact/Broken*/				
	Leaking				
9. Does information on					
custody records, traffic					
reports, and sample					
tags agree?	Yes/No*				
10.Date Received at Lab:	_____				
11. Time Received:					
Sample Transfer					
Fraction: _____	_____				
Area #: _____	_____				
By: _____	_____				
On: _____	_____				

* Contact Client and attach record of resolution

Reviewed By:_____ Logbook No.:_____

Date:_____ Logbook Page No.:_____

FORM DC-1

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 9

EXAMPLE OF WET CHEMISTRY SHORTS AND RUSHES LOGBOOK

WET CHEMISTRY SHORTS & RUSHES **Receipt Date:**

HOLDING TIME					Immediate			24 Hr		48 Hr								
Work Order / Client	Matrix	Earliest Sampling Date	Earliest Sampling Time	Rush Parameters	pH	DO	Sulfite	Fe +2	Cr+6	Total BOD	Carbon BOD	Color	Nitrate	Nitrite	OPO4	Set. Solids	Turbidity	Comments (Quick TAT, MS/MSD, etc.)

0000004

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 10

EXAMPLE OF KATAHDIN WORK ORDER/LOGIN COC REPORT

Katahdin
ANALYTICAL SERVICES

Katahdin Analytical Services
Login Chain of Custody Report (Inc1)
Jan. 26, 2007
03:51 PM

Page: 1 of 1

Login Number: SA0395
Account:KATAHD001 Web
Katahdin Analytical Services

Project:

Primary Report Address:
Leslie Dimond
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Primary Invoice Address:
Accounts Payable
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Report CC Addresses:
Invoice CC Addresses:

Login Information

ANALYSIS INSTRUCTIONS	:
CHECK NO.	:
CLIENT PO#	:
COOLER TEMPERATURE	: n/a
DELIVERY SERVICES	: In House
EDD FORMAT	:
MAIL DATE	:
PM	: LAD
PROJECT NAME	: QC Holding Blanks
QC LEVEL	: I
REGULATORY LIST	:
REPORT INSTRUCTIONS	:
SDG ID	:
SDG STATUS	:

Laboratory Sample ID	Client Sample Number	Collect Date/Time	Receive Date	PR	Verbal Date	Due Date	Comments
SA0395-1	WHITE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	
Matrix	*Product*	*Hold Date (shortest)*	*Bottle Type*		*Bottle Count*		
Aqueous	S SW8260FULL5ML	09-FEB-07			2		
SA0395-2	BLUE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	
Matrix	*Product*	*Hold Date (shortest)*	*Bottle Type*		*Bottle Count*		
Aqueous	S SW8260FULL5ML	09-FEB-07			2		

Total Samples: **2** ***Total Analyses:*** **2**

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 11

EXAMPLE OF LABORATORY INTERNAL CHAIN-OF-CUSTODY FORM

Katahdin Analytical Services, Inc.
Internal Custody Record

EXTRACTIONS (AQ) Work Order #:_____

Sample Numbers Involved	Placed in refrigeration by: Initials
	Date
	Time

Sample Numbers	Analysis	Removed			Returned			Consumed?	
		Initials	Date	Time	Initials	Date	Time	Yes	No

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 12

EXAMPLE OF REFRIGERATOR TEMPERATURE LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

SAMPLE RECEIPT WALK-IN TEMPERATURE LOG

Corrective Action: Note in the "comments" column and notify the QAO or supervisor; document corrective actions taken and return to control.

Thermometer Location		Sample Receipt Walk-in 1	Comments
Acceptance Criteria		2 to 6 °C	
Date	Initials	Temp (°C)	
02/2/07	NDA	3.7	
02/05/07	DWM	5.8	
02/06/07	DWM	5.8	
02/07/07	DWM	5.8	
02/08/07	DWM	3.8	
02/09/07	DWM	3.5	
02/12/07	DWM	3.5	
02/13/07	DWM	3.5	

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 13

EXAMPLE OF IMMEDIATE INTERNAL COC LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
INTERNAL CUSTODY RECORD FOR IMMEDIATES

CLIENT	PROJECT	CLIENT ID &/or WORK ORDER #	ANALYSIS	OUT date/time	IN date/time	INIT	Consumed?
Jacobs		WW4813-1A,-2A	ICP	9/13/06 0930 →	0935	DJJ	yes no
Jacobs		WW4883-1A	ICP	9/16/06 0900 →	1000	DJJ	yes no
CES		WW4965	BOD	9/20/06 0900	9/20/06 1000	CP	yes no
CCAB		WW4969	BOD	9/20/06 1000	↓	CP	yes no
GEMF		WW4970	BOD	↓	↓	CP	yes no
Jacobs		WW4962-1A,-2A	ICP	9/20/06 0900 →	1000	DJJ	yes no
Irving		WW4994	BOD	9/21/06 1000	9/21/06 1045	CP	yes no
Highliner		WW4992	BOD	9/21/06 1045		CP	yes no
National		WW5000	TS, Perox, DO, pH, Sp. Cond	9/21/06 1100	9/21/06 1257	[illegible]	yes no
W+C		WW5001	BOD	9/21/06 1300		CP	yes no
Aliens		WW5016	NO3	9/22/06 1300	09/22/06 1100	[illegible]	yes no
RAS04		WW5010	↓	↓	↓	↓	yes no
EcoMaine		WW5029	BOD	9/22/06 1100		CP	yes no

QAQC143

0000095

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: SD-903
Revision History
Cover Page
Page 1

TITLE: **SAMPLE DISPOSAL**

Prepared By: _____ Date: 2/01

Approved By:

Group Supervisor: _____ Date: _____

Operations Manager: _____ Date: 2/01

QA Officer: Deborah J. Nadeau Date: 2·01

General Manager: _____ Date: 2/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, ~~added pollution prevention~~, added updated log-book and greater detail on dis-posal.	DN	2·01	2/01
02	Major rewrite to include more detail on hazardous waste regu-lations + to reflect current practices.	DN	02/05	02/05
03	Rewrite of section 7 to comply with current practices in new facility. Updated Figures 1 to 3.	DN	02·08	02·08

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-903-03**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-903-03**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

TITLE: SAMPLE DISPOSAL

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services, Inc. requires strict adherence to specific procedures for the disposal of samples. The procedures are designed to categorize waste materials, provide for their safe and timely disposal and to ensure compliance with local and federal regulations pertaining to disposal of chemicals and environmental samples. Any other means of disposal not described in this SOP is prohibited without consent from the Katahdin Environmental Health & Safety Officer and/or the Katahdin Environmental Compliance Officer.

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical personnel for the disposal of samples. These procedures apply to the disposal of all samples received or processed by Katahdin. Refer to the current revision of Katahdin SOP CA-107 regarding the disposal of spent preparation and analysis reagents, standards, sample extracts, distillates, or digestates.

1.1 Definitions

Hazardous Waste – A "Solid Waste" which displays a hazardous characteristic or is specifically listed as hazardous waste.

Solid Waste – Any discarded material that is not excluded from the definition of hazardous waste.

Discarded Material – Material that is abandoned, recycled or inherently waste-like.

Waste (State of Maine) –

- Any useless, unwanted, or discarded substance or material, whether or not such substance or material has any other future use.
- Any substance or material that is spilled, leaked, pumped, poured, emptied or dumped onto the land or into the water or ambient air.
- Materials which are used in a matter constituting disposal, burned for energy recovery, reclaimed, or accumulated speculatively.

Ignitable Hazardous Waste – EPA Waste Code D001

- Liquids with a flash point less than 140°F or 60°C.
- Solids capable of spontaneous combustion under normal temperature and pressure.
- Ignitable compressed gas.
- Oxidizers.

Corrosive Hazardous Waste - Liquids with a pH less than or equal to 2.0 or greater than or equal to 12.5. EPA waste code D002.

Reactive Hazardous Waste – EPA waste code D003.

- A material that reacts violently with water.
- A material that generates toxic gases or fumes.
- Explosives.

Toxic Hazardous Waste – A material that exceeds certain concentration levels based on the toxicity characteristic leaching procedure (TCLP). See Figure 3 for the chemicals and concentration levels covered under this definition.

Listed Wastes – Lists of chemicals that are considered hazardous based on the following criteria

- Virgin chemical or unused product.
- Sole active ingredient.
- Single substance spill debris.

Listed wastes are divided into 5 subcategories

- F-wastes – Describe hazardous waste from non-specific sources usually containing halogenated and non-halogenated solvents.
- K-wastes – Describe hazardous wastes created by specific processes.
- U-wastes – Describe toxic or non-acute hazardous wastes.
- P-wastes – Describe acute hazardous wastes. (Note: Maine considers a material to be a P-listed waste if it contains 10% or more of any P-listed chemical.
- State listed wastes – Maine lists any material with a concentration of greater than 50 ppm Polychlorinated Biphenyls (PCB) as a hazardous waste.

Organics hit – A liquid sample containing greater than 1 mg/L of organic contaminants or a soil sample containing greater than 20 mg/kg of organic contaminants.

1.2 Responsibilities

Only designated analysts/technicians trained in these procedures may dispose of samples or analytical by-products. Each analyst or technician must be familiar with Katahdin Analytical safety procedures. Gloves, safety glasses, lab coats and/or other protective clothing must be worn at all times.

It is the responsibility of the designated Katahdin personnel involved in the disposal of samples to read and understand this SOP, to adhere to the procedures outlined, to properly document their activities in the appropriate lab notebook and file the

necessary manifests and reports to outside agencies in the required manner. Refer to Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of the Department Managers to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

It is the responsibility of the Katahdin Environmental Health & Safety Officer (EHSO) to manage the proper classification and disposal of samples. Katahdin is responsible for regulatory compliance of Katahdin's waste storage areas (less than 90 day storage). The EHSO ensures compliance of the waste storage areas with applicable state and federal regulations. The EHSO is responsible for providing the appropriate training to all individuals involved in the proper classification and/or disposal of samples. The EHSO is responsible for working with the Laboratory Operations Manager/Environmental Compliance Officer to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate unresolved problems and concerns to the Laboratory Vice President.

It is the responsibility of the Operations Manager/Environmental Compliance Officer to oversee adherence to Katahdin sample disposal and hazardous waste practices by all laboratory groups under his/her authority, to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate problems and concerns to the EHSO and/or the Laboratory Vice President.

It is the responsibility of the Laboratory Vice President to provide the necessary resources to meet the regulatory requirements of proper classification and disposal of samples.

2.0 SUMMARY OF METHOD

Not applicable.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

Not applicable.

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.

7.5 Once you are ready to dispose of the samples of interest (the oldest samples that have been purged), these samples must be sorted, logged, and the classification/category (sample knowledge) information recorded.

Sample storage times (as listed in section 7.2) and space should be taken into consideration when purging samples. It is important to make room for future samples, but to make sure that samples are not purged too early. Samples should be pulled from the walk-in or the volatiles refrigerators to make room for new samples. When purging, chose a section that needs extra space the most and remove the oldest samples.

Safety glasses, nitrile gloves, lab coat, and a splash apron must be worn when handing samples during disposal

7.6 Remove the designated purge samples from the shelf one by one and line them up on the countertop in the log-in area. Generally, removing two cartloads at a time is a good amount to purge at one time. For volatile samples in 40mL vials, 5 or 6 vial trays should be purged at a time. Samples should be lined up across the counter with the earliest sample to the left and building up to the right, organizing the samples according to work order and sample number. After the samples are lined up, they should be recorded in the Sample Disposal Logbook (SDL). Refer to Figure 1 for an example SDL page. The location the samples were removed from should also be recorded. Sample storage areas are recorded with the following designations:

VOA (Aq)	Aqueous Volatiles(VOA)
VOA (SL)	Solid Volatiles(VOA)
M	Metals
EXT	Extractables (Organic)
TOC	Total Organic Carbon
WC	Wet Chemistry
S	Soils

7.7 The next step is to use the sample disposal list to determine the earliest release date of the reports and to determine each samples appropriate waste classification/characterization. As stated in section 7.3, the primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide.

Using the information from the KIMS disposal list, record the appropriate classification for each sample in the SDL. If multiple categories are identified as being present then a single category is selected as controlling. The order of precedence is PCB's, metals and then organics. If another scenario is found, the individual should bring it to the EHSO for a determination of the acceptable waste stream designation or a determination that it should be lab packed separately.

If samples have been sorted that have not been in storage for the 30 days beyond the release date (60 or 90 for certain clients), then these samples need to be placed back in storage and it should be noted in the SDL.

7.8 As stated above, a sample may be categorized into a waste stream based upon the analytes it contains as determined by laboratory testing. In addition, many samples are also categorized as hazardous waste based upon the preservative that they contain. Since many samples contain preservatives, caution must be used when dumping samples. It is also important to ensure that the sample container is empty. This can be accomplished by holding the container upside down and shaking gently until liquid is no longer observed coming out of the container.

7.9 Once waste categories have been determined and entered into the SDL, The following waste categories are disposed of as follows:

7.9.1 Dumping non-hazardous samples (as determined by laboratory testing)

Non-hazardous samples (non-preserved) are poured directly into the sink in the warehouse.

Non-hazardous solid samples are disposed of with the general trash, which is picked up by commercial trash collectors and ultimately disposed of in a waste-to-energy incinerator.

Sample containers from non-hazardous samples are disposed of with the general trash.

7.9.2 Dumping Samples with high Organics (as determined by laboratory testing)

Aqueous samples get dumped into waste stream "K". Containers are disposed of with general trash. Solid samples are placed into waste stream "I" with their containers. The disposal date is recorded in the SDL.

7.9.3 Dumping Acidic Samples or samples high in metals (except Mercury)

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 Basic samples (as determined by the basic preservative or by laboratory testing)

Aqueous samples get disposed of in waste stream "NHi". Containers are disposed of with general trash. The disposal date is recorded in the SDL.

7.9.5 Dumping samples with high mercury (as determined by laboratory testing)

Aqueous samples get disposed of in waste stream "S". Containers are disposed of with general trash. Solid samples get disposed of in waste stream "L" with their containers. 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 flashoints (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream "O". Containers are disposed of with general trash. Solid samples get disposed of in waste stream "I" with their containers. The disposal date is recorded in the SDL.

7.9.8 Dumping samples with high cyanide (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream "NHi". Containers are disposed of with general trash. Solid samples should be set aside for labpack. The disposal date is recorded in the SDL.

7.9.9 Miscellaneous Disposal (as determined by the preservative)

Sodium Bisulfate: Sodium Bisulfate often comes in vials, but may also come in the 2-4oz glass jars. Dump the Sodium Bisulfate out of the container into waste stream "A". There may be remaining soil left in the sample container. The soil's waste stream and dump date will be dictated by the SDL. The disposal date is recorded in the SDL.

Methanol / Free Products: This often comes in vials, but may also come in the 2-4oz glass jars. Dump the methanol out of the container into the mix-flammables accumulation. When this satellite accumulation container gets full it can be dumped into the "O" waste stream. There may be remaining soil left in the sample container. The soil's waste stream and dump date will be dictated by the SDL. Lastly, samples marked "free product" on the Katahdin sample ID label can be dumped into the mixed flammables stream. The disposal date is recorded in the SDL.

7.10 Every 4 to 6 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.

7.11 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 Vice President or 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

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.

TITLE: SAMPLE DISPOSAL

Maine Department of Environmental Protection (ME DEP) Hazardous Waste Management Rules

ME DEP modified EPA Form 8700-13A

LIST OF TABLES AND FIGURES

Figure 1 Example of Sample Disposal Logbook
Figure 2 Example of KIMS Generated Waste Disposal Report
Figure 3 Example Of Hazardous Waste Area Daily Check Documentation
Figure 4 Characteristic Toxic Hazardous Waste and TCLP concentrations

TITLE: SAMPLE DISPOSAL

FIGURE 1

EXAMPLE OF SAMPLE DISPOSAL LOGBOOK (SDL)

KATAHDIN ANALYTICAL SERVICES, INC. –SAMPLE STORAGE/DISPOSAL LOGBOOK

WORK ORDER/ SAMPLE NUMBERS	DEPARTMENT	EARLIEST RELEASE DATE	< CRITERIA	SAMPLE KNOWLEDGE									DATE DISPOSED	INITIALS
			CLEAN	WL	ORG	METS	CN	FP	HG	PCBS				
SAS783-1	WC	10-17-07	✓										1-22-08	GN
SAS786-1		10-17-07	✓											
SAS787-1,2,4		10-17-07	✓											
SAS790-1		10-19-07		✓										
SAS793-1		10-17-07	✓											
SAS795-1-9		10-23-07	✓											
SAS797-1		10-25-07	✓											
SAS798-1,2		10-25-07			✓									
SAS799-1-5		10-31-07	✓											
SAS804-1,2		10-25-07	✓											
SAS809-1,2		10-23-07	1			2				2				
SAS810-1-4		10-23-07	✓											

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TITLE: SAMPLE DISPOSAL

FIGURE 2

EXAMPLE OF KIMS GENERATED WASTE DISPOSAL REPORT

SAMPLE DISPOSAL REPORT

Query by: Login SA6501 to SA7000
Date : 15-JAN-08

Sample	SDG	Status	Mail Date	Parameter	Value		
SA6605-1		NEED	12/02/07				
SA6606-1		NEED	12/02/07				
SA6607-1		NEED	11/15/07				
SA6608-1		NEED	12/06/07				
				ORG	1.17	MG/L	(HIGH)
SA6608-1		NEED	12/06/07				
SA6608-2		NEED	12/06/07				
				AA	13	MG/KG	(HIGH)
SA6609-1		NEED	11/26/07				
SA6609-1		NEED	11/26/07				
SA6610-1		NEED	11/30/07				
SA6611-1	FCS-020	NEED	12/07/07				
SA6611-2	FCS-020	NEED	12/07/07				
SA6611-3	FCS-020	NEED	12/07/07				
SA6611-4	FCS-020	NEED	12/07/07				
SA6611-5	FCS-020	NEED	12/07/07				
SA6611-6	FCS-020	NEED	12/07/07				
SA6611-7	FCS-020	NEED	12/07/07				
SA6611-8	FCS-020	NEED	12/07/07				
SA6612-1	NSA-030	NEED	12/07/07				
SA6612-2	NSA-030	NEED	12/07/07				
SA6612-3	NSA-030	NEED	12/07/07				
SA6612-4	NSA-030	NEED	12/07/07				
				ORG	1.70735	MG/L	(HIGH)
SA6612-5	NSA-030	NEED	12/07/07				
				ORG	1.0481	MG/L	(HIGH)

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

Item/Day:	Monday	Tuesday	Wednesday	Thursday	Friday
1. Are containers closed? (Except when waste is being added)	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
2. Are containers properly labeled with a hazardous waste label?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
3. Do you have access to each container and can you read the label? (36" aisle?)	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
4. Is each container marked with the date storage began?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
5. Are the dates on the containers less than 90 days old?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
6. Is container free of dents, bulges, rust, spills or leaks?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
7. Are all containers on a firm working surface?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
8. Inspection by: Name (No Initials)	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
9. Time of Inspection	16:35	15:00	14:45	14:15	16:25
10. Verification of Inspection (Name/Date)	[illegible] 1-28-08	[illegible] 1-29-08	[illegible] 1-30-08	[illegible] 1-31-08 1415	[illegible] 1-02 1700

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

Particle Size Analysis
ASTMD422-63

Approvals (Signature/Date):

William S. Cicero Laboratory Director	12/27/07 Date	Name Current Department Manager	12/27/07 Date
Kirstin L. McCracken Quality Assurance Manager	12/27/07 Date	Bryce E. Stearns Technical Director	12/27/07 Date
Bryce E. Stearns Interim Health & Safety Coordinator	12/27/07 Date		

This cover page documents management approval for the following changes to this SOP:

- **This SOP has been renamed and was previously identified as SOP No. LM-SL-D422**
- **Any reference within this document to Severn Trent Laboratories, Inc. or STL, should be understood to refer to TestAmerica Laboratories, Inc. (formerly known as Severn Trent Laboratories, Inc.)**
- **The effective date of this SOP has been changed to January 1, 2008.**

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STANDARD OPERATING PROCEDURE
PARTICLE-SIZE ANALYSIS OF SOILS
ASTM D422-63
Applicable Matrix: Soil

APPROVAL SIGNATURES

Laboratory Director:	Christopher A. Ouellette	Date: August 5, 2005
QA Manager:	Kirstin L. McCracken	Date: August 5, 2005
Department Manager:	William S. Cicero	Date: August 5, 2005
Techncial Director: Geotechnical Laboratoy	Matthew R. Duquette	Date: August 5, 2005

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1.0 SCOPE AND APPLICATION

1.1. This SOP describes the laboratory procedure for the determination of particle size distribution in soil samples that contain sand, silt, clay and gravel.

2.0 SUMMARY OF METHOD

2.1. A soil sample submitted for particle size analysis is prepared according to laboratory SOP LM-SL-D421 *Dry Preparation of Soil for Particle Size Analysis* or LM-SL-D2217 *Wet Preparation of Soil Samples for Particle Size Analysis*. Particles greater than 75um (gravels to fine sands) are determined by sieve analysis while particles less than 75um (silts and clays) are determined by sedimentation using a hydrometer followed by sieve analysis.

After wet or dry sample preparation, the sample is passed through No.10 sieve. The particles retained on the No.10 sieve (greater than 2.00mm) are further separated by sieve analysis. A portion of the sample that passed through the No.10 sieve is transferred to a glass sedimentation cylinder to which distilled water has been added. Seven hydrometer readings are taken over 24 hours. After the final hydrometer reading, the suspension is rinsed over a No. 200 (75 um) sieve, dried, and further separated by sieve analysis.

Particle size determinations for each sieve measurement and hydrometer reading are calculated and corrected for hygroscopic moisture and specific gravity. Unless a separate analysis for specific gravity is requested, the specific gravity is s assumed to be 2.65.

2.2. This procedure is based on ASTM D422-63.

3.0 DEFINITIONS

Not Applicable

4.0 INTERFERENCES

Not Applicable

5.0 SAFETY

5.1. Care should be taken to avoid exposure to the sample matrix since all environmental samples are potentially hazardous. Protective clothing, eye protection and disposable gloves should be worn when handling samples. All laboratory personnel must be familiar with the environmental health and safety plan described in the STL Chemical Safety Manual.

6.0 EQUIPMENT AND SUPPLIES

6.1. Top-Loading Balance sensitive to 0.01 g

6.2. Mechanical Stirring Apparatus and Dispersion Cup

6.3. Sedimentation Cylinder(s) 1000 mL

6.4. Hydrometer: ASTM 151H in specification E 100.

6.5. Thermometer: Accurate to 0.5°C

6.6. Mortar and Rubber Tipped Pestle

6.7. Sieves of the following size(s):

3.0 in (75.00mm)	No. 20 (850.0um)
2.0 in (50.00mm)	No. 40 (425um)
1.5 in (37.50mm)	No. 60 (250.0um)
1.0 in (25.00mm)	No. 80 (180.0um)
3/4 in (19.00mm)	No. 100 (150.0um)
3/8 in (9.50mm)	No. 200 (75.0um)
No. 4 (4.75mm)	
No. 10 (2.00mm)	

6.8. Oven with temperature range of 60° C to 110° C

6.9. Timing Device with second hand and capable of counting up to 25 hours

6.10. Stainless steel spatulas, spoons, metal and bristle brushes

6.11. Ro-tap machine

7.0 REAGENTS AND STANDARDS

7.1. Reagents

Deionized (DI) Water: Milli-Q System

Sodium Hexametaphosphate Solution: Combine 2940 g of DI water with 120 g of sodium hexametaphosphate in an appropriate container. Mix until the solution is homogeneous. Assign an expiration date of 30 days from date of preparation.

8.0 SAMPLE HANDLING AND PRESERVATION

8.1. At least 500 grams of soil sample should be collected in glass or polyethylene jars. Immediately following collection the sample should be sealed and cooled to 4°C in order to preserve the moisture content of the sample.

8.2. Samples are stored from the time of receipt in the laboratory until 30 days after delivery of the reconciled data package report. Unless otherwise specified by a federal, state or

client-specific protocol, samples are disposed of after 30 days in a manner that complies with all applicable regulations.

9.0 QUALITY CONTROL

Not Applicable

10.0 CALIBRATION AND STANDARDIZATION

10.1. Calibrate the balance on each day of use, prior to use.

10.2 Calibrate the hydrometers every two years following the procedure given in LM-SL-001.

11.0 PROCEDURE

11.1 Sample Preparation

Prepare the sample following either laboratory SOP LM-SL-D421 (Dry Preparation) or LM-SL-D2217 (Wet Preparation).

11.2 Sample Analysis

11.2.1 Hydrometer Test

Transfer the sample/sodium hexametaphosphate mixture into a dispersion cup ensuring a quantitative transfer using DI water. Fill the dispersion cup ~half full with DI water. Mix the sample for one minute using the immersion blender.

Pour the contents of the dispersion cup through a #10 sieve into a 1000 mL sedimentation cylinder (1000 mL graduated cylinder). Rinse the cup with DI water, to ensure that the entire sample is transferred to the sedimentation cylinder.

Add DI water to the sedimentation cylinder until the volume is 1000 mL then cover the cylinder with a sheet of parafilm. Allow the sample to stabilize to ambient temperature.

Transfer the material retained on the No. 10 sieve to a labeled medium-size aluminum dish, and place the aluminum dish into an oven maintained at a temperature of 105ºC for a minimum of 16 hours.

After up to 12 sedimentation cylinders have been prepared, ensure that each cylinder is filled to the reference line with DI water, covered with parafilm, and that there is sufficient clean DI water available to rinse the hydrometer.

Record the ID of the hydrometer that you intend to use. Record the start time and set the timer for elapsed time.

Use the hydrometer reading table used to perform the activities as indicated (shake, place or read) for each 1000 mL cylinder.

To shake, rotate the flask up and down for one minute approximating at least 60 turns (one turn upside down and then right side up constitutes two turns).

To take a reading, gently insert the hydrometer into the cylinder then wait ~ 20 seconds. Read the hydrometer at the top of the meniscus to the nearest 0.0005. Enter the hydrometer reading into the appropriate cell on the benchsheet. Clean the hydrometer by twisting and dropping into a clean DI water bath.

Insert a temperature sensor into the cylinder to the depth the hydrometer reached. Read the temperature to the nearest 0.5°C. Enter the temperature reading into the appropriate cell on the benchsheet. After reading, rinse the sensor in a DI water bath.

Take readings every 2, 5, 15, 30, 60, 240 and 1440 minutes. Record each reading on the benchsheet, then transfer this information into the appropriate cell of the EXCEL worksheet.

11.2.2 Large Sieves

Enter a brief description of the type of non-soil material (e.g.- sticks, grass, wood, plastic) in the “Description of >#10 particles” of the EXCEL worksheet in the cell labeled “Non-soil Material”.

Tare the balance and weigh each of the 3/4”, 3/8”, #4 and #10 sieves. Record the weight measurements in the EXCEL worksheet in the cells labeled “Sieves (Tares)”. Also weigh any larger sieves if necessary.

Stack the sieves then transfer the soil material retained on the #10 sieve into the sieve stack. Shake for 2 minutes. If there is greater than ~30 g of material, place the sieve stack into the Ro-tap machine and shake for 10 minutes.

Weigh each sieve along with the material retained on it. Enter these weight measurements in the “Sieve + Sample Weights” section of the Excel worksheet.

Determine the average hardness of the particles retained on the #10 sieve by dropping a hammer on the particle from a height of approximately one foot. Hardness qualifiers are hard, soft or brittle. Record the hardness qualifier in the “Description of >#10 Particles” section of the Excel worksheet.

Observe and record the shape of the particles in the “Description of >#10 Particles” section of the Excel worksheet. Shape qualifiers are well rounded, rounded, subrounded, subangular, and angular.

11.2.3 Small Sieves

When the hydrometer test is complete, transfer the soil from the sedimentation cylinder to a #200 wet wash sieve.

Wash the soil through the #200 sieve until the water from the bottom of the sieve runs clear. Carefully transfer the material retained on the sieve to a labeled 250 mL glass beaker.

Place the beaker into the oven. Dry at a temperature of 105ºC for at least 16 hours. After 16 hours, remove the beaker from the oven and allow it to cool.

Gently mix the dried contents of the beaker with a rubber-tipped pestle to break any soil aggregates that may have formed during the drying stage.

Tare the balance and weigh each of the sieves between #20 and #200. Record the weight measurements in the EXCEL worksheet in the cells labeled “Sieves (Tares)”.

Transfer the dry sample into the sieve stack, ensuring that all material is transferred. Use hair or wire brushes to clean the beaker.

Place the sieve stack on the Rotap machine and shake for ten minutes.

Weigh each sieve along with the material retained on it. Enter these weight measurements in the “Sieve + Sample Weights” section of the Excel worksheet.

Determine particle size using the following formula.

12.0 CALCULATIONS

12.1. Sample Used (SU)

<u>Wet Method</u>

$$SU = (pan + wet\ sample - pan) \otimes PS$$

Where:

PS = Percent solids

Note: for hydrometer SU, subtract the dry weight of any material retained on the No. 10 sieve.

<u>Dry Method</u>

$$SU = (pan + dry\ sample - pan) - (pan + non - soil\ material - pan) \otimes HMCF$$

Where:

HMCF = Hygroscopic moisture correction factor

12.2 Sieve Analysis (Percent Finer = PF)

Large Sieves:

3 inch: PF = 100-100 (Sieve and Sample (3 inch) - Sieve (3 inch))/SU*

2 inch: PF = PF (3 inch) - 100(Sieve and Sample (2 inch) - Sieve (2 inch))/SU and so on through the #10 Sieve.*

Small Sieves:

#20: PF = PF(#10) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#20) - sieve(#20))/sample used*

#40: PF = PF (#20) - 100(mass passing #10/sample mass (Hyd))*(sieve and sample (#40) - sieve (#40))/sample used and so on up through #10 sieve.*

12.5 Hydrometer Analysis

Particle size, Micron

1000*sqrt [930*viscosity/980*(SG-1))*(effective depth/time)]

Viscosity at sample temperature, poises
Effective Depth, cm = 16.29-264.5*(actual Hydrometer reading - 1) above equation for effective depth based on equation found with table 2 in method, in which 16.29 = 0.5*(14.0-67.0/27.8)+10.5 and 264.5 = (10.5-2.3)/0.031
Time, minutes = Time of hydrometer reading from beginning of sedimentation
Sqrt - square root
SG - Specific Gravity of soil
Viscosity - is the resistance of a liquid to flow

12.6 Percent Finer (PF):

PF = Constant*(actual hydrometer reading - hydrometer correction factor - 1)

Where:
Constant = (100,00/W)*SG/(SG-1)
W = (Total sample used *sample used for hydrometer analysis*HMCF)/Amount of total sample passing #10 sieve
Hydrometer Correction = slope*sample temperature + Intercept
Slope = ((low temp. reading -1)-(high temp. reading -1)/(low temp. - high temp.))
Intercept = (low temp. reading -1) - (low temp. * slope)

13.0 DATA ASSESSMENT, CRITERIA & CORRECTIVE ACTION

13.0. Complete the sample preparation benchsheet and EXCEL spreadsheet. Document any problems encountered during sample analysis so they may be properly addressed in the project narrative. Perform primary and secondary data review following the guidance given in laboratory SOP for Data Review.

14.0 METHOD PERFORMANCE

Not Applicable

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1. The laboratory optimizes technology to minimize pollution and reduce the production of hazardous waste whenever possible.

15.2. The laboratory procedures for waste management comply with applicable federal, state and local regulations and are described in SOP LP-LB-001HAZWD.

16.0 REFERENCES

16.1. Standard Test Method for Particle-Size Analysis of Soils, ASTM D422-63, Volume 04.08 Soil and Rock, American Society for Testing and Materials, Philadelphia, Pa., 1998.

17.0 REVISION HISTORY

17.1 Procedures to separate the non-soil particles from the sample were removed.

18.0 TABLES, DIAGRAMS, FLOWCHARTS

18.1 Table 1: Hydrometer Reading Table

Table 1: Hydrometer Reading Table (For up to 12 Sedimentation Cylinders)

Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)	Elapsed Time (hr:min)	Task	Cyl. No.	Actual Time (min)
0:00	Shake	1		1:01	Read	10	5
0:01	**Place**	**1**		**1:02**	Shake	11	
0:01	**Shake**	**2**		**1:03**	Place	11	
0:02	Place	2		1:04	Read	9	15
0:03	Read	1	2	1:05	Read	11	2
0:04	Read	2	2	1:06	Read	7	31
0:06	Read	1	5	1:07	Read	3	58
0:07	Read	2	5	1:08	Read	11	5
0:08	Shake	3		**1:09**	Shake	12	
0:09	**Place**	**3**		**1:10**	Place	12	
0:09	**Shake**	**4**		1:11	Read	10	15
0:10	Place	4		1:12	Read	12	2
0:11	Read	3	2	1:13	Read	4	63
0:12	Read	4	2	1:14	Read	8	32
0:14	Read	3	5	1:15	Read	12	5
0:15	Read	4	5	1:18	Read	11	15
0:16	Read	1	15	1:19	Read	9	30
0:17	Read	2	15	1:21	Read	5	60
0:20	Shake	5		1:25	Read	12	15
0:21	Place	5		1:26	Read	10	30
0:23	Read	5	2	1:27	Read	6	59
0:24	Read	3	15	1:33	Read	11	30
0:25	Read	4	15	1:34	Read	7	59
0:26	Read	5	5	1:41	Read	12	31
0:27	Shake	6		1:42	Read	8	60
0:28	Place	6		1:52	Read	9	63
0:30	Read	6	2	1:53	Read	10	57
0:31	Read	1	30	2:06	Read	11	63
0:32	Read	2	30	2:07	Read	12	57
0:33	Read	6	5	4:17	Read	1	256
0:34	Shake	7		4:18	Read	2	256
0:35	Place	7		4:19	Read	3	250
0:36	Read	5	15	4:20	Read	4	250
0:37	Read	7	2	4:21	Read	5	240
0:38	Read	3	29	4:22	Read	6	234
0:39	Read	4	29	5:00	Read	7	265
0:40	Read	7	5	5:01	Read	8	259
0:41	Shake	8		5:02	Read	9	253
0:42	Place	8		5:03	Read	10	247
0:43	Read	6	15	5:04	Read	11	241
0:44	Read	8	2	5:05	Read	12	235
0:47	Read	8	5	24:01	Read	1	1440
0:48	Shake	9		24:02	Read	2	1440
0:49	Place	9		24:03	Read	3	1434
0:50	Read	7	15	24:04	Read	4	1434
0:51	Read	9	2	24:05	Read	5	1424
0:52	Read	5	31	24:06	Read	6	1418
0:54	Read	9	5	24:07	Read	7	1412
0:55	Shake	10		24:08	Read	8	1406
0:56	Place	10		24:09	Read	9	1400
0:57	Read	8	15	24:10	Read	10	1394
0:58	Read	10	2	24:11	Read	11	1388
0:59	Read	6	31	24:12	Read	12	1382
1:00	**Read**	**1**	**59**				
1:00	**Read**	**2**	**58**				

Source: Laboratory Prepared Reference Document