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NSWC INDIAN HEAD
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Quality Assurance Project Plan
Sampling Analysis Plan
for
Munitions Response Program
Site Inspections at
Five Small Arms/Skeet Ranges

Naval Support Facility
Indian Head-Stump Neck Annex
Indian Head, Maryland

Naval Facilities Engineering Command
Washington

Contract Number N62467-04-D-0055
Contract Task Order 423

April 2009

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

QUALITY ASSURANCE PROJECT PLAN
SAMPLING ANALYSIS PLAN
FOR
MUNITIONS RESPONSE PROGRAM
SITE INSPECTIONS AT FIVE SMALL ARMS/SKEET RANGES
NAVAL SUPPORT FACILITY INDIAN HEAD-STUMP NECK ANNEX
INDIAN HEAD, MARYLAND

APRIL 2009

Prepared for:
Naval Facilities Engineering Command Washington
1314 Harwood Street, SE
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Prepared under:
Comprehensive Long-Term Environmental Action Navy (CLEAN) Contract
No. N62467-04-D-0055
Contract Task Order 0423

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Approval Signatures:

NAVFAC QAO/Chemist Signature
Printed Name/Title/Date
Approval Authority

_____

EXECUTIVE SUMMARY

Tetra Tech NUS has prepared this Quality Assurance Project Plan QAPP Sampling and Analysis Plan SAP has been prepared under CLEAN Contract No. N62467-04-D-0055 Contract Task Order 423. The Navy has conducted various testing, training, and disposal activities related to military munitions at the Naval Support Facility located in Indian Head, Maryland, since it was established in 1890 as a Naval Ordnance Station. The general locations of Naval Support Facility Indian Head (NSF-IH), Main Installation and the Stump Neck Annex are shown on Map 1-1. The Stump Neck Annex covers approximately 1,100 acres on the Stump Neck peninsula at the confluence of the Potomac River and Chicamuxen Creek in Charles County, Maryland. The Indian Head Main Installation is northeast of the Stump Neck Annex, across Mattawoman Creek. Much of Stump Neck peninsula lies within the Valley Impact Fan (Map 1-2). General Smallwood State Park and private property are east of Stump Neck Annex.

As a result of the Navy's explosives and munitions training activities, munitions and explosives of concern (MEC) and munitions constituents (MC) may be present at various sites throughout the Stump Neck Annex. The term MEC includes Discarded Military Munitions (DMM), Unexploded Ordnance (UXO), and MC in high enough concentrations to pose an explosive hazard. The Navy is following the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) process for the investigation of these sites.

Closed, transferred, and transferring military ranges and sites not located on an operational range are considered other than operational. The initial phase of the CERCLA process, the Preliminary Assessment (PA) was completed in September 2005 and identified for further investigation at the Stump Neck Annex five small arms and trap/skeet ranges classified as "other than operational range" sites or Munitions Response Areas or Munitions Response Sites (MRA/Ss) . The five MRA/Ss described in the Stump Neck Annex PA Report are indicated on Map 1-3. The PA used five primary sources of information to support the facility data collection effort, including historical archives, personal interviews, installation data repositories (including the Administrative Record), visual surveys, and off-facility data sources and repositories, such as local libraries and museums.

The primary objective of this Site Inspection (SI) is to determine whether further response actions or remedial investigations are appropriate for any of the sites identified in the PA to restore the sites to an acceptable environmental condition. The SI considers the background information provided in the PA and collects supplemental site-specific environmental data to further characterize the nature and extent of MEC and MC at the sites identified in the PA Report.

This QAPP SAP has been prepared in accordance with the Department of Defense requirements for developing QAPPs for the management of environmental data collection and use as described in the Uniform Federal Policy for Quality Assurance Project Plans (UFP QAPPs aka as UFP SAPs). Therefore this UFP SAP consists of the thirty seven UFP SAP Work Sheets with text, figures, maps and appendices added as necessary to provide the required information.

The information provided in the Work Sheets was developed based on the results of three project scooping meetings among the planning team The planning team consisted of representatives of the Navy, United States Environmental Protection Agency (EPA) Region III, Maryland Department of Environment, and Tetra Tech NUS (See Work Sheet 9 for attendees). Work Sheet 10 contains the general and site-specific Conceptual Site Models (CSMs) for each of the five sites. These CSMs were used as the basis for the development of the site-specific project Data Quality Objectives, which are contained in Work Sheet 11. The remainder of the Work Sheets describes the sampling, analytical and data evaluation procedures including quality requirements.

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Appendix A: Field Standard Operating Procedures
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Appendix D: Sample Size Calculations

ACRONYMS

%D	Percent deviation
%R	Percent recovery
°F	Degree Fahrenheit
AOC	Areas of Concern
AR	Administrative record
ARAR	Applicable or Relevant and Appropriate Requirements
BERA	Baseline Ecological Risk Assessment
bgs	Below ground surface
BTAG	Biological Technical Assistance Group
CA	Corrective Action
CAS	Chemical Abstract Service
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-Term Environmental Action Navy
CLP	Contract Laboratory Program
COC	Chain-of-custody
COPC	Contaminant of potential concern
CPR	Cardiopulmonary resuscitation
CSM	Conceptual Site Model
CTC	Cost-to-complete
CTO	Contract Task Order
DI	Deionized
DMM	Discarded Military Munitions
DoD	Department of Defense
DPT	Direct-push technology
DQI	Data Quality Indicator
DQO	Data Quality Objective
DVM	Data Validation Manager
EDD	Electronic data deliverable
EOD	Explosive Ordnance Device
FCR	Field Change Request
FOL	Field Operations Leader
FS	Feasibility Study

ft^2/d	Square foot per day
FTMR	Field Task Modification Request
GC/MS	Gas Chromatograph/Mass Spectrometer
GIS	Geographic Information System
GPS	Global Positioning System
HASP	Health and Safety Plan
HDPE	High-density polyethylene
HSM	Health and Safety Manager
IAS	Initial Assessment Study
IC	Initial Calibration
IDL	Instrument Detection Limit
IDQTF	Intergovernmental Data Quality Task Force
IDW	Investigation-derived waste
IR	Installation Restoration
IS	Internal Standard
ITRC	Interstate Technology and Regulatory Council
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LFB	Laboratory Fortified Blank
LIMS	Laboratory Information Management Systems
MARLAP	Multi-Agency Radiological Laboratory Analytical Protocols (Manual)
MARSSIM	Multi-Agency Radiation Survey and Site Investigation Manual
MC	Munitions Constituents
MCB	Marine Corps Base
MCL	Maximum Contaminant Level
MDE	Maryland Department of the Environment
MDL	Method detection limit
MEC	Munitions and Explosives of Concern
mi^2	Square mile
MOU	Memorandum of Understanding
MPC	Measurement Performance Criterion
MQO	Measurement Quality Objectives
MRA/S	Munitions Response Area or Munitions Response Site
MRP	Munitions Response Program
MS/MSD	Matrix Spike/Matrix Spike Duplicate
msl	Mean sea level

MSR	Management Systems Review
NAVEODTECHDIV	Naval Explosion Ordnance Disposal Technical Division
NAVFAC	Naval Facilities Engineering Command
NAVSCOLEOD	Naval School, Explosive Ordnance Disposal
NAVSEA	Naval Sea Systems Command
NCP	National Contingency Plan
NDW	Naval District Washington
NEIC	National Enforcement Investigations Center
NFA	No Further Action
NFESC	Naval Facilities Engineering Service Center
NG	Nitroglycerin
NIRIS	Naval Installation Restoration Information Solution
NIST	National Institute of Standards and Technology
NPDES	National Pollutant Discharge Elimination System
NPL	National Priorities List
NSF	Naval Support Facility
OSHA	Occupational Safety and Health Administration
PA	Preliminary Assessment
PAH	Polynuclear aromatic hydrocarbon
PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparability
PCB	Polychlorinated biphenyl
PDF	Portable Document Format
PG	Professional Geologist
PM	Project Manager
POC	Point of Contact
PPE	Personal Protective Equipment
PQO	Project Quality Objective
PRP	Potentially Responsible Party
PRQL	Project-Required Quantitation Limit
PT	Proficiency Testing
QA	Quality Assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit

QMP	Quality Management Plan
QS	Quality System
QSM	Quality Systems Manual
RAGS	Risk Assessment Guidance for Superfund
RBC	Risk-based Concentrations
RBSL	Risk-based Screening Levels
RCRA	Resource Conservation and Recovery Act
RI	Remedial Investigation
RIC	Reconstructed Ion Chromatogram
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RSD	Relative Standard Deviation
RT	Retention Time
RTM	Remedial Technical Manager
RTW	Retention Time Window
SAP	Sampling and Analysis Plan
SD	Standard Deviation
SDG	Sample Delivery Group
SDWA	Safe Drinking Water Act
SDZ	Safety Danger Zone
SI	Site Inspection
SIM	Selective Ion Monitoring
SOP	Standard Operating Procedure
SQL	Structured Query Language
SRM	Standard Reference Material
SSL	Soil Screening Level
SSO	Site Safety Officer
SVOA	Semivolatile Organic Analytes
SVOC	Semivolatile Organic Compounds
SW	Surface Water
SWMU	Solid Waste Management unit
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
Tetra Tech	Tetra Tech NUS, Inc.
TOC	Total Organic Carbon
TSA	Technical Systems Audit

U.S. EPA	United States Environmental Protection Agency
UFP	Uniform Federal Policy
USACE	United States Army Corps of Engineers
USCS	Unified Soil Classification System
USDA	United States Department of Agriculture
UXO	Unexploded Ordnance
VOA	Volatile Organic Analyte
VOC	Volatile organic compound
VSP	Visual Sample Plan
WMA	Wildlife Management Area
XRF	X-ray fluorescence

SAP Worksheet #2 -- SAP Identifying Information

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number: Naval Support Facility (NSF) Indian Head-Stump Neck Annex, Maryland
Operable Units: UXO-14, -15, -16, -17, and -25
Contractor Name: Tetra Tech, Inc.
Contract Number: N62467-04-D-0055
Contract Title: Comprehensive Long-Term Environmental Action Navy (CLEAN)
Work Assignment Number (optional): **Contract Task Order** (CTO) 0423

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. EPA, 2005) and *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, QAMS (2002)*.

2. Identify regulatory program: Department of Defense (DoD) Military Munitions Response Program (MRP) using the general CERCLA process.

3. This SAP is a **project-specific SAP**.

4. List dates of scoping sessions that were held:

Scoping Session	Date
Meeting No. 1 - Development of Conceptual Site Model (CSM) and Data Quality Objectives (DQOs)	November 7 to 9, 2007
Meeting No. 2 - Development of CSM and DQOs	December 18 and 19, 2007
Meeting No. 3 - Development of CSM and DQOs	January 28 and 29, 2008
Meeting No. 4 - Site Walk	February 11, 2008

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

Title	Date
Not Applicable – This is the initial MRP SI	

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

United States Environmental Protection Agency (U.S. EPA) Region 3 – Regulatory Oversight
Maryland Department of the Environment (MDE) – Regulatory Oversight

7. Lead organization (see WS 7 for detailed list of data users)
Naval Facilities Engineering Command (NAVFAC) Washington
NSF Indian Head

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:

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
A. Project Management		
Documentation		
1	Title and Approval Page	NA
2	Table of Contents SAP Identifying Information	NA
3	Distribution List	NA
4	Project Personnel Sign-Off Sheet	NA
Project Organization		
5	Project Organizational Chart	NA
6	Communication Pathways	NA
7	Personnel Responsibilities and Qualifications Table	NA
8	Special Personnel Training Requirements Table	NA
Project Planning/Problem Definition		
9	Project Planning Session Documentation (including Data Needs tables) Project Scoping Session Participants Sheet	NA
10	Problem Definition, Site History, and Background. Site Maps (historical and current)	NA
11	Site-Specific Project Quality Objectives	NA
12	Measurement Performance Criteria Table	NA
13	Sources of Secondary Data and Information, Secondary Data Criteria and Limitations Table	NA
14	Summary of Project Tasks	NA
15	Reference Limits and Evaluation Table	NA
16	Project Schedule/Timeline Table	NA
B. Measurement Data Acquisition		
Sampling Tasks		
17	Sampling Design and Rationale	NA
18	Sampling Locations and Methods/Standard Operating Procedure (SOP) Requirements Table Sample Location Map(s)	NA
19	Analytical Methods/SOP Requirements Table	NA
20	Field Quality Control Sample Summary Table	NA
21	Project Sampling SOP References Table, Sampling SOPs	NA
22	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	NA
Analytical Tasks		
23	Analytical SOPs, Analytical SOP References Table	NA
24	Analytical Instrument Calibration Table	NA
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	NA

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

SAP Worksheet #3 -- Distribution List

(UFP-QAPP Manual Section 2.3.1)

Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-Mail or Mailing Address	Document Control Number (Optional)
Joseph Rail	Navy Remedial Project Manager (RPM)	NAVFAC Washington	202.685.3105	joseph.rail@navy.mil	NA
Jeffrey Bossart	Environmental Division Director, NSF-IH	NSF Indian Head	301.744.4705	jeffrey.bossart@navy.mil	NA
Curtis DeTore	MDE RPM	MDE	410.537.3791	cdetore@mde.state.md.us	NA
Dennis Orenshaw	U.S. EPA Region 3 RPM	U.S. EPA Region 3	215.814.3361	orenshaw.dennis@epa.gov	NA
John Trepanowski	Program Manager	Tetra Tech	610.491.9688	john.trepanowski@tetratech.com	NA
Ralph Basinski	Project Manager (PM)	Tetra Tech	412.921.8308	ralph.basinski@tetratech.com	NA
Ralph Brooks	UXO/MEC Manager	Tetra Tech	706.224.4690	ralph.brooks@tetratech.com	NA
Fred Ramser	Field Operations Leader (FOL)	Tetra Tech	412.921.8838	fred.ramser@tetratech.com	NA
Ed Sedlmyer	Project Chemist	Tetra Tech	412.921.8704	edward.sedlmyer@tetratech.com	NA
Andrea Colby	Lab Project Manager	Katahdin Analytical Services, Inc.	207.874.2400	acolby@katahdinlab.com	NA
Scott Brunk	Vice President (VP) Corporate Operations	Analytical Laboratory Services, Inc.	717.944.5541 ext. 3147	sbrunk@analyticallab.com	NA

Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-Mail or Mailing Address	Document Control Number (Optional)
Anna Milliken	Operations Manager	Analytical Laboratory Services, Inc.	717.944.5541 ext. 3135	amilliken@analtyicallab.com	NA
TBD	Direct-Push Technology (DPT)/ Driller	Subcontractor	***TBD***	***TBD***	NA

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

(UFP-QAPP Manual Section 2.3.2)

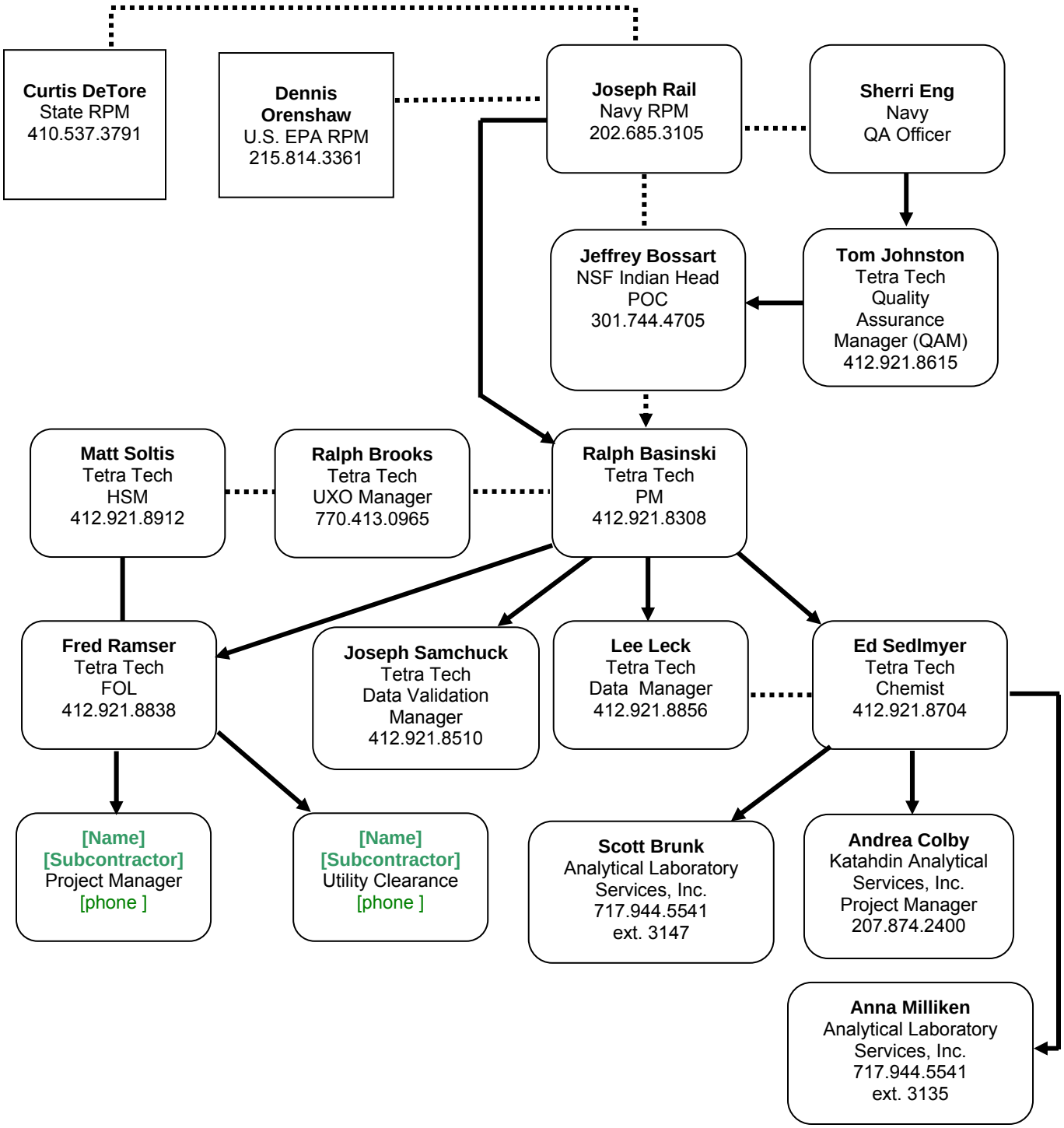
Name	Organization/Title/Role	Telephone Number (optional)	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Navy and Regulator Project Team Personnel					
Joseph Rail	Navy RPM	202.685.3105	joseph.rail@navy.mil	All	
Jeffrey Bossart	Environmental Division Director, NSF-IH	301.744.4705	jeffrey.bossart@navy.mil	All	
Curtis DeTore	State (MDE) RPM	410.537.3791	cdetore@mde.state.md.us	All	
Dennis Orenshaw	U.S. EPA Region 3 RPM	215.814.3361	orenshaw.dennis@epa.gov	All	
Tetra Tech Project Team Personnel					
Ralph Basinski	PM	412.921.8308	ralph.basinski@tetratech.com	All	
Fred Ramser	FOL	412.921.8838	fred.ramser@tetratech.com	All	
Ed Sedlmyer	Project Chemist	412.921.8704	edward.sedlmyer@tetratech.com	WS #s 12, 14, 15, 19, 20, 23-28, 30, 34-37	
Matt Soltis	Health and Safety Manager (HSM)	412.921.8912	matt.soltis@tetratech.com	HASP	
Joseph Samchuck	Data Validation Manager (DVM)	412.921.8510	joseph.samchuck@tetratech.com	WS #s 12, 14, 15, 19, 20, 23-28, 30, 34-37	

Name	Organization/Title/Role	Telephone Number (optional)	Signature/E-Mail Receipt	SAP Section Reviewed	Date SAP Read
Subcontractor Personnel					
Leslie Dimond	Lab (Katahdin Analytical Services, Inc.) QAM	207.874.2400	ldimond@katahdin.com	WS #s 12, 14, 15, 19, 20, 23-28, 30, 34-37	
Scott Brunk	Analytical Laboratory Services, Inc. (VP Corporate Operations)	717.944.5541 ext. 3147	sbrunk@analtyicallabs.com	WS #s 12, 14, 15, 19, 20, 23-28, 30, 34-37	
Anna Milliken	Analytical Laboratory Services, Inc. (Operations Manager)	717.944.5541 ext. 3135	amilliken@analtyicallabs.com	WS #s 12, 14, 15, 19, 20, 23-28, 30, 34-37	
TBD	DPT (TBD) Supervisor	***TBD***	***TBD***		

SAP Worksheet #5 -- Project Organizational Chart

(UFP-QAPP Manual Section 2.4.1)

Lines of Authority ——— Lines of Communication ·····



SAP Worksheet #6 -- Communication Pathways

(UFP-QAPP Manual Section 2.4.2)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure
Field Task Modification Requests (FTMRs) – Sampling	Tetra Tech FOL	Fred Ramser	412.921.8838	Immediately obtain approval from Tetra Tech PM. Document via Field Task Modification Request (FTMR) Form.
QAPP Amendments	Navy RPM	Joseph Rail	202.685.3105	Send scope change to Tetra Tech Program Management Office.
Schedule Changes	Tetra Tech PM	Ralph Basinski	412.921.8308	Inform Navy via schedule impact letter as soon as impact is realized.
Field issues that require changes in the scope or implementation of field work	Tetra Tech PM Tetra Tech FOL	Ralph Basinski Fred Ramser	412.921.8308 412.921.8838	FOL informs PM, PM informs Navy RPM, Navy RPM issues scope change, if warranted; Scope change to be implemented before work is executed.
Stop work recommendations, for example, to protect workers from unsafe conditions or situations or to prevent a degradation in quality of work	Tetra Tech PM Tetra Tech FOL Tetra Tech QAO Navy RPM	Ralph Basinski Fred Ramser Kelly Carper Joseph Rail	412.921.8308 412-921-8838 412.921.7273 202.685.3105	Responsible party immediately informs subcontractors, Navy, and project team.
Field or laboratory data issues	Analytical Laboratory Tetra Tech Project Chemist	Matt Kraus	412.921.8729	Immediately notify Tetra Tech Project Chemist when issue is related to chemical data. Notify Data Validation Staff and Tetra Tech PM if necessary.

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)
John Trepanowski	Program Manager	Tetra Tech	Overseas CLEAN Program	M.S. Mining Engineering, B.S. Mining Engineering, 27 years of engineering experience
Ralph Basinski	Project Manager	Tetra Tech	Oversees project, budget, schedule, and technical day-to-day management of the project.	B.S. Chemistry, 30 years of environmental experience
Fred Ramser	FOL, Lead Geologist, Project Safety Officer	Tetra Tech	Supervises, coordinates, and performs field sampling activities. Prepares all geological interpretations and report text.	B.S. Geology, 16 years experience as a geoscientist
Matt Soltis	HSM	Tetra Tech	Oversees Navy CLEAN program safety.	B.S. in Industrial Safety Science, CSP, CIS.
Matt Kraus	Project Chemist	Tetra Tech	Participate in scoping, prepare laboratory scope, coordinate with laboratory, and data quality review.	B.S. environmental chemistry, 2 years environmental experience
Andrea Colby	Laboratory Project Manager	Katahdin Analytical Services, Inc.	Coordinate analyses with laboratory chemists, ensure the scope is followed, QA data packages, communicate with Tetra Tech staff.	Can be provided upon request

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
Scott Brunk or Anna Milliken	Laboratory Management	Analytical Laboratory Services, inc.	Coordinates analyses with laboratory chemist, ensures that scope is followed, QA data packages, communicates with Tetra Tech staff	Can be provided upon request
TBD	DPT/Driller	Subcontractor	Soil boring and sampling, groundwater well installation and sampling.	
TBD	Utility Location	Subcontractor	Utility location.	

SAP Worksheet #8 -- Special Personnel Training Requirements Table

(UFP-QAPP Manual Section 2.4.4)

The following table is used to identify and describe any specialized/non-routine project specific training requirements or certifications needed by personnel in order to successfully complete the project or task. OPNAV 5090.1 instructions are not considered specialized training; the OPNAV training requirements represent routine, minimum requirements that are mandatory for all DON projects.

Project Function	Specialized Training by Title or Description of Course	Training Provider	Training Date	Personnel / Groups Receiving Training	Personnel Titles / Organizational Affiliation	Location of Training Records / Certificates[1]
Field Technicians	40-hour HAZWOPER 8-hour HAZWOPER Refresher	Various	Current	UXO and field sampling personnel	All field staff / Tetra Tech	Tetra Tech project office and field office
FOL	Same as field technician HAZWOPER requirements plus Supervisor Training	Various	Current	FOL	FOL / Tetra Tech	Tetra Tech project office and field office
X-Ray Fluorescence (XRF) Technician	Operation of XRF	Previously trained personnel	Current	XRF Technician	XRF Technician / Tetra Tech	Tetra Tech project office and field office
Health and Safety Officer	First Aid/Cardiopulmonary Resuscitation (CPR) Training	Red Cross	September 2007	Field Personnel	Tetra Tech	Tetra Tech project office and field office

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

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) Quality Assurance Program and described in the Department of Defense Quality Systems Manual (DoD QSM), January 2006.

SAP Worksheet #9 -- Project Scoping Session Participants Sheet

(UFP-QAPP Manual Section 2.5.1)

Project Name: Stump Neck Annex SI Projected Date(s) of Sampling: August 2009 Project Manager: Ralph Basinski	Site Name: Five Other Than Operational MRP Sites Site Location: NSF Indian Head—Stump Neck Annex, Charles County, Maryland

Date of Session: November 7 to 9, 2007
Scoping Session Purpose: Develop CSM and DQOs

Name	Title	Affiliation	Phone Number	E-Mail Address	Project Role
Joseph Rail	Navy RPM	NAVFAC Washington	202. 685.3105	joseph.rail@ navy.mil	Navy NAVFAC project management
Shawn Jorgensen	NSF Indian Head RPM	NSF Indian Head	301. 744.2263	shawn.a. jorgensen@ navy.mil	NSF Indian Head facility project management
Ralph Basinski	Project Manager	Tetra Tech	412. 921.8308	ralph.basinski@ tetratech.com	Tetra Tech project managmenet
Ralph Brooks	UXO Manager	Tetra Tech		ralph.brooks@ tetratech.com	UXO support
Jim Coffman	Geophysicist	Tetra Tech	412. 921.8244	jim.coffman@ tetratech.com	On-site geopysics measurements
Rick Barringer	Technical Lead	Tetra Tech	412. 921.8524	rick.barringer@ tetratech.com	Technical support

November 7-9, 2007 Meeting Notes

The November 7 – 9, 2007 scoping session addressed 16 sites at IH-Stump Neck Annex. Five Small Arms Range sites (UXO-14, UXO-15, UXO-16, UXO-17, UXO-25) are addressed in this SAP and are highlighted in the following general meeting notes. Information relevant to these sites is included in Worksheet # 9. The full meeting minutes are available in the project file.

IH-Stump Neck Annex MRP Site Locations	General Notes
A. Marine Rifle Range (UXO-14)	• Two discrete target butts on east end of rifle range. Target butt soil (subsurface) to be field-screened using XRF instrumentation to identify subsoil lead concentrations. • Lead is the marker compound. Need reference concentration–either health-based or risk-based for lead in soil to evaluate samples against. • Emphasis on field analyses, with limited laboratory analysis to confirm lead/other metal concentrations in target butt subsurface soil. • Consider limited soil sampling in front of target butts (undershot) and beyond target butts (overshot) to delineate zones of projectile and/or metals accumulation beyond the range target butts.
B. Old Skeet and Trap Range (UXO-15)	• Superimpose maximum fall zone for shot onto MRP site map. Much of the maximum fall zone may be in the Potomac River (underwater). • Surface soil samples to be field-screened using XRF instrumentation to identify subsoil lead concentrations. • Lead is the marker compound. Need reference concentration–either health-based or risk-based for lead in soil to evaluate samples against. • Soil samples to be evaluated for PAHs-clay pigeons are a source for these compounds, and many fragments are present on this range. • Emphasis on field analyses, with limited laboratory analysis to confirm lead/other metal concentrations (and PAHs) in surface soil.
C. Small Arms (Pistol) Range (UXO-17)	• The earthen slope behind the former range targets is on east end of rifle range. Slope soil (subsurface) to be field-screened using XRF instrumentation to identify subsoil lead concentrations. There has been some limited slumping (slope failure) along this hillside slope, so sample collections may have to penetrate through uncontaminated soil originally from the head of the slope. • Lead is the marker compound. Need reference concentration–either health-based or risk-based for lead in soil to evaluate samples against. • Emphasis on field analyses, with limited laboratory analysis to confirm lead/other metal concentrations in range soil. • Additional sediment samples to be collected from drainage on the south (or north) side of range to evaluate particulate transport of lead by surface water

IH-Stump Neck Annex MRP Site Locations	General Notes
D. Rum Point Skeet Range (UXO-16)	• Superimpose maximum fall zone for shot onto MRP site map. • Surface soil samples to be field-screened using XRF instrumentation to identify subsoil lead concentrations. • Lead is the marker compound. Need reference concentration–either health-based or risk-based for lead in soil to evaluate samples against. • Soil samples to be evaluated for PAHs since clay pigeons are a source for these compounds. Many clay pigeon fragments and plastic shot gun wads were observed at surface and are present on this range. • Emphasis on field analyses, with limited laboratory analysis to confirm lead/other metal concentrations in range surface soil. • Additional sediment samples to be collected from drainage on the western side of range to evaluate particulate transport by surface water
E. Roach Road Rifle Range (UXO-25)	• Historical aerial photographs will be used to verify the location of the rifle range. The PA verified that the location of Roach Road was modified in this area (road shifted in 1982). • The 1963 memorandum indicates a barricade (target butt) was constructed behind the targets using earth and railroad timbers. • Range soil to be field-screened using XRF instrumentation to identify subsoil lead concentrations. • Lead is the marker compound. Need reference concentration–either health-based or risk-based for lead in soil to evaluate samples against. • Emphasis on field analyses, with limited laboratory analysis to confirm lead/other metal concentrations in range soil.

Date of Session: December 18 and 19, 2007
Scoping Session Purpose: Develop CSM and DQOs

Name	Title	Affiliation	Phone Number	E-Mail Address	Project Role
Joseph Rail	Navy RPM	NAVFAC Washington	202. 685.3105	joseph.rail@ navy.mil	Navy NAVFAC project management
Curtis DeTore	State RPM	MDE	410. 537.3791	cdetore@mde. state.md.us	Regulatory
Dennis Orenshaw	USEPA Region III RPM	U.S. EPA Region III	215. 814.3361	orenshaw. dennis@epa.gov	Regulatory
Ralph Basinski	PM	Tetra Tech	412. 921.8308	ralph.basinski@ tetratech.com	Tetra Tech project management
Ralph Brooks	UXO Manager	Tetra Tech		ralph.brooks@ tetratech.com	UXO support
Jim Coffman	Geophysicist	Tetra Tech	412. 921.8244	jim.coffman@ tetratech.com	On-site geopysics measurements
Rick Barringer	Technical Lead	Tetra Tech	412. 921.8524	rick.barringer@ tetratech.com	Technical support

December 18 and 19, 2007 Meeting Notes

The sixteen MRP sites were toured over the period of two days. The planning team agreed that, due to the number of sites (16) and complexity of the UFP SAP, additional meetings would be necessary. The agreement was made that the small arms ranges would be addressed separately from the MEC sites and two UFP SAPs would be prepared.

The planning team approved the general approach for the five small arms/skeet ranges, which was described in the November 7-9 meeting notes.

Preparation of a technical memorandum for No Action at Test Area 2 (UXO-22) was approved on the basis that Test Area 2 was never used.

A second planning meeting was scheduled for January 28 and 29 in the MDEL headquarters in Baltimore, MD for the ten MEC sites.

Date of Session: January 28 and 29, 2008
Scoping Session Purpose: Develop CSM and DQOs

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Joseph Rail	Navy RPM	NAVFAC Washington	202. 685.3105	joseph.rail@ navy.mil	Navy NAVFAC project management
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Ralph Brooks	UXO Manager	Tetra Tech		ralph.brooks@ tetratech.com	UXO support
Jim Coffman	Geophysicist	Tetra Tech	412. 921.8244	jim.coffman@ tetratech.com	On-site geopysics measurements
Rick Barringer	Technical Lead	Tetra Tech	412. 921.8524	rick.barringer@ tetratech.com	Technical support

January 28 and 29, 2008 Meeting Notes

This meeting dealt primarily with the ten MEC sites. No major decisions were made for the five small arms ranges.

Date of Session: February 11, 2008
Scoping Session Purpose: Site Walk

Name	Title	Affiliation	Phone Number	E-Mail Address	Project Role
Ralph Basinski	PM	Tetra Tech	412. 921.8308	ralph.basinski@ tetratech.com	Tetra Tech project management
Ralph Brooks	UXO Manager	Tetra Tech		ralph.brooks@ tetratech.com	UXO support
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February 11, 2008 Meeting Notes

A site walk, performed on February 11, 2008, was attended by the Tetra Tech Project Manager, the UXO Manager, and the Field Operations Leader (FOL). The objectives of the site walk were as follows:

- Familiarize the FOL with the sites.
- Develop coordination activities between the UXO Manager and the FOL.

Participants also identified and reviewed logistical considerations of the field investigation including:

- Site access.
- Sequencing of surface clearance, geophysical investigation, and MC sampling activities.
- Navy restrictions on working.
- Vegetation clearing requirements.
- Coordination of MEC investigation and MC sampling activities.

SAP Worksheet #10 -- Problem Definition, Site History and Background

(UFP-QAPP Manual Section 2.5.2)

A detailed CSM is presented for each of the five "other than operational" small arms skeet ranges located at the Stump Neck Annex, Naval Support Facility-Indian Head (NSF-IH). The initial CSMs for each site were developed as part of the PA (Malcolm Pirnie, 2005), which is one of the initial steps in the CERCLA process. The CSMs, which were presented in the PA, have been reviewed and modified as necessary based on additional information obtained during the Tetra Tech site visits and scoping meetings conducted during the preparation of this UFP SAP for the SI. The CSMs for each of the sites will be updated as additional information is obtained. Updated versions of the CSMs, which incorporate data obtained in the field effort, will be presented in the report prepared after all SI fieldwork is completed.

This introductory section for Problem Definition, Site History and Background (Worksheet #10) includes general information on the site background, facility history, and general description of the geology, hydrology, endangered species, cultural settings, and natural resources on an installation-wide basis at the Stump Neck Annex. The site-specific Problem Definition worksheet for each of the five sites addressed in this SAP have been prepared and is presented here (under Worksheet #10) to more completely describe the CSM and define the problem for each of the five MC sites (Worksheets #10.1 through #10.5). SAP Worksheet #10 includes the Problem Definition in terms of the site-specific CSM and contains in detail the initial step (Step 1) of the DQO process, with site-specific maps developed for each site. A brief summary of the problem, which is detailed in Worksheet #10, is included as Step 1 in Worksheet #11 [Project Quality Objectives (PQOs)].

10-1 GENERAL INTRODUCTION AND PROBLEM DEFINITION FOR WORKSHEET #10

The Navy has conducted various testing, training, and disposal activities related to military munitions at NSF Indian Head, Maryland, since it was established in 1890 as a Naval Ordnance Station. The general locations of the NSF-IH Main Installation and Stump Neck Annex are shown on Map 1-1. NSF-IH is located in northwestern Charles County, Maryland, approximately 25 miles southwest of Washington, D.C. The Stump Neck Annex covers approximately 1,100 acres on the Stump Neck peninsula at the confluence of the Potomac River and Chicamuxen Creek in Charles County, Maryland. Stump Neck Annex was acquired by the Navy in 1901 to support activities at the 2,300-acre Indian Head Main Installation. These two Navy properties are not contiguous; the Indian Head Main Installation is northeast of the Stump Neck Annex across Mattawoman Creek. General Smallwood State Park and private property parcels are located east and southeast, respectively, of Stump Neck Annex, and the Chicamuxen Wildlife Management Area (WMA) is to the south across Chicamuxen Creek.

The Valley Impact Area and Stump Neck Impact Area are both located on the Stump Neck Annex (Map 1-2). These two areas received fire from various caliber munitions (14-inch through 16-inch) from the Valley Gun Proving Site located on the Main Installation of NSF-IH from 1891 through 1921. Various caliber guns (4-inch through 16-inch) were fired into these two areas. The firing fan from the Main Installation to the impact areas covers several of the small arms ranges discussed in this UFP SAP. In addition to fire from the gun proving site, the Stump Neck Impact Area received impacts from a firing range set up in the vicinity of Rum Point. Marine Corps Base (MCB) Quantico was also permitted to fire large artillery at the Stump Neck Annex for several years until 1934.

As a result of the Navy's explosives and munitions training activities, Munitions and Explosives of Concern (MEC) and MC may be present at various sties throughout the Stump Neck Annex. The term MEC includes DMM, UXO, and MC in high enough concentrations to pose an explosive hazard. This UFP SAP will cover the small arms ranges. A separate UFP SAP has been prepared to investigate the MEC sites located on the Stump Neck Annex. The Navy is following the CERCLA process for the investigation of these sites.

Although no MEC is believed to be in any of the small arms ranges, the possibility exists, and precautions will be taken. The Valley Impact Area and Stump Neck Impact Area are both included under a separate UFP SAP concerning MEC sites at Indian Head Stump Neck Annex.

The initial phase of the CERCLA process, the PA, was completed in September 2005 and identified 17 "other than operational range sites" or Munitions Response Areas/Munitions Response Sites (MRA/Ss) at the Stump Neck Annex for further investigation (Malcolm Pirnie, 2005). Closed, transferred, and transferring military ranges and sites not located on an operational range are considered "other than operational."

The small range MC site locations are shown on Map 1-3. The Malcolm Pirnie (2005) PA Report used five primary sources of information to support the facility data collection effort, including historical archives, personal interviews, installation data repositories (including the Administrative Record), visual surveys, and off-facility data sources and repositories such as local libraries and museums.

Table 10-1 summarizes the five other than operational ranges at the Stump Neck Annex that were evaluated in the installation's PA report (Malcolm Pirnie, 2005).

10-2 GENERAL STUMP NECK ANNEX PROBLEM DEFINITION INFORMATION BASED ON THE PA REPORT

Established in 1890, NSF-IH is the Navy's oldest ordnance station. Throughout its long and distinguished history, the facility has proved guns, armor, and propellants, developed and manufactured powder and propellants, and is recognized as a leader in energetics research and development. Shortly after operations commenced, additional property was acquired by the Navy to increase the size of the installation. The most notable acquisition was of Stump Neck Annex as an impact area and safety buffer in 1901.

With the opening of the nearby Dahlgren Naval Proving Ground in the early 1930s, the primary focus of Indian Head turned to powder manufacturing. Additional acquisition and improvement to the installation continued through the 1960s to increase operational capacity and safety buffers required for the manufacture, testing, and storage of energetic materials.

Stump Neck Annex has played a key role in the development of Explosive Ordnance Disposal (EOD) research and training. In 1941, Stump Neck Annex was chosen as an isolated location for the practical instruction syllabus associated with the Navy's Advanced Mine School in Washington, D.C. The remote location and proximity to the new Explosives Investigation Laboratory (Main Installation) made Stump Neck an ideal choice.

Shortly thereafter, the requirements for "bomb disposal," ordnance demilitarization (enemy and allied), and reverse engineering of foreign ordnance grew increasingly important, mainly due to World War II. The newly formed EOD unit at Stump Neck Annex responsible for these tasks had grown such that individual EOD detachments could be deployed for mission support. Stump Neck Annex could then be used to support the powder plant and a school for EOD officers and enlisted personnel. In 1953, the Naval EOD Technical Center was formed at the Stump Neck Annex with the purpose of training EOD personnel in all service branches. The training function was renamed the Naval School, Explosive Ordnance Disposal (NAVSCOLEOD). In 1993, the Naval EOD Technical Center was renamed the Naval Explosive Ordnance Disposal Technical Division (NAVEODTECHDIV), a division of the newly established Naval Ordnance Center. The NAVEODTECHDIV operated until 1999, at which time it was relocated to Eglin Air Force Base, Florida. Stump Neck Annex is home of the Improved Explosive Device (IED) School where research is conducted on render safe technologies for items encountered in hostile situations by law enforcement and security personnel. EOD Technology Division is a tenant of Stump Neck Annex. Their mission is to utilize technology to develop and deliver EOD information, tools, and equipment and to meet the needs of Joint Services EOD operating forces and other customers.

In recent decades, NSF-IH has come to be known as a center of excellence in the development and manufacturing of specialized energetic materials used in demolition and propulsion. Now under the direction of the Naval Sea Systems Command (NAVSEA), the current mission of NSF-IH is as follows:

- To provide services in energetics for all warfare centers through engineering, fleet and operational support, manufacturing technology, limited production, and industrial base support.

- To provide research, development, testing, and evaluation of energetic materials, ordnance devices, and components, and other engineering standards including chemicals, propellants, propulsion systems, explosives, pyrotechnics, warheads, and simulators.

- To provide support to all warfare centers, military departments, and the ordnance industry for special weapons, explosives safety, and ordnance environmental issues.

Table 10-2 summarizes the key milestones in the history of NSF-IH.

10-3 PHYSICAL AND ENVIRONMENTAL CHARACTERISTICS

The following sections provide general information for Stump Neck Annex including climate, topography, geology, soil and vegetation types, hydrology, hydrogeology, cultural and natural resources, and threatened, endangered, and protected species.

10-3.1 Climate

Stump Neck Annex, located on the eastern shore of the Potomac River in Charles County, Maryland, has a continental-type climate with four well-defined seasons. Located in the middle latitudes of North America, atmospheric flow is from west to east. The Potomac River and its tributaries significantly affect the climate, moderating extreme temperatures and causing higher humidity in the region. In the winter, the Blue Ridge and Appalachian mountain ranges located west of the Stump Neck Annex obstruct the cold, continental air. The coldest period occurs in late January and early February, with low temperatures averaging 29 degrees Fahrenheit (ºF). July is the warmest month, with average high temperatures of 85ºF. Annual precipitation is well distributed, with July and August as the wettest months. Average annual precipitation is 44 inches. Maximum snow accumulation averages 9 inches between November and March. The growing season lasts approximately 190 days, starting in mid-April.

10-3.2 Topography

NSF-IH occupies two peninsulas along the eastern shore of the Potomac River. The Stump Neck Annex is on the southern peninsula, and the Main Installation is located on the northern peninsula. The two peninsulas are separated by Mattawoman Creek. The general topography of the mainland areas of Charles County can be described as gently rolling lands with a few steep slopes. These areas include many drainage swales and streams. Shoreline areas at NSF-IH are generally steeply sloped.

The Stump Neck Annex has a relatively low topographic profile. The highest point is the northeastern portion of the peninsula at an elevation of approximately 140 feet above mean sea level (msl). The lowest points lie along the shorelines of the Stump Neck Annex adjacent to Mattawoman Creek and Chicamuxen Creek. These areas are mostly flat, tidal marsh areas, although several 50- to 60-foot bluffs exist along Mattawoman Creek.

10-3.3 Geology

The Stump Neck Annex lies within the Atlantic Coastal Plain Physiographic Province 8 to 10 miles east of the Fall Line that marks the western extent of the physiographic province. The regional geology consists of a sedimentary wedge of Cretaceous to Quaternary fluvial and marine deposits overlying crystalline Precambrian metamorphic and igneous bedrock. The sedimentary wedge dips and thickens eastward and ranges in thickness from 550 feet to 900 feet in the vicinity of the Stump Neck Annex (Vroblesky, and Fleck, 1991; Hiortdahl, 1990). It lies nonconformably on the crystalline basement rock surface, which dips to the east. The geologic units underlying NSF-IH, in stratigraphically ascending order, are the Lower Cretaceous Potomac Group, Tertiary Aquia Formation of the Pamunkey Group, fluvial-estuarine deposits of Tertiary to early Quaternary age, and undivided Quaternary deposits.

The lithology of the Potomac Group consists of interbedded clay, silt, sand, and gravel deposited in fluviodeltaic environments (Hiortdahl, 1990). The Potomac Group ranges in thickness from 650 to 750 feet in the vicinity of the Stump Neck Annex (Vroblesky and Fleck, 1991; Harsh and Laczniak, 1990) and consists of three geologic units (in ascending stratigraphic order): the Patuxent Formation, Arundel Formation, and Patapsco Formation. The Patuxent Formation consists of sand and pebbles with thin clay interbeds and is 300 to 400 feet thick in the study area. The Arundel Formation generally consists of a massive clay with abundant lignite and siderite concretions and is less than 100 feet thick beneath most of the study area. The Patapsco Formation generally consists of sand and silt separated by thick clay layers. The interpreted thickness of the Patapsco Formation in the study area varies from about 200 feet to more than 450 feet (Hiortdahl, 1997).

The Aquia Formation (Upper Paleocene) consists of marine deposits of olive black to olive gray, micaceous, glauconitic quartz sand interbedded with sand, silt, and clay. The formation is 0 to 80 feet thick in the NSF-IH peninsula area. The younger units of the Pamunkey Group and the Chesapeake Group have been removed by erosion in the study area.

Overlying the Aquia Formation are fluvial-sedimentary deposits consisting of gravel, sand, and loam. These sediments are referred to as "upland deposits" and range in age from Pliocene to early Pleistocene (Hiortdahl, 1997). The upland deposits crop out at the surface in the northern portion of NSF-IH where surface elevations exceed 40 feet above msl. However, beneath most of the study area, the surficial sediments consist of Pleistocene paleochannel deposits and Holocene alluvial and paludal deposits (Hiortdahl, 1997). These deposits consist of gravel, sand, silt, clay, and peat mixtures with irregular bedding and with an aggregate thickness of 0 to approximately 40 feet. The Aquia Formation and younger upland deposits are missing in many locations in the NSF-IH peninsula area due to erosion and deposition in Pleistocene and Holocene paleochannels. Where this occurs, the overlying Quaternary deposits directly overlie the Cretaceous formations.

10-3.4 Soil and Vegetation Types

Charles County is located in the inner Potomac Coastal Plain geologic province. The soils in this area are derived from unconsolidated marine sediments that vary from sandy to clayey in texture and from excessively well drained to poorly drained. Hydric and erodible soils are prevalent. High water tables, severe erosion, earthslides, and hardpans are common.

The United States Department of Agriculture (USDA) mapped the soils of the Stump Neck Annex in the Soil Survey of Charles County, Maryland (1974). The main soil series in this area are the Beltsville, Keyport, and Elkton silt loams. Some additional soil types found at the Stump Neck Annex are cut-and-fill land, gravelly land, tidal marsh, and Mattawan soil. The following discussion is a description of the soil types at the Stump Neck Annex.

The eastern area of the Stump Neck Annex is primarily composed of the Beltsville silt loam, with a small area of gravelly land. Beltsville series soils consist of silt and sand with moderate amounts of clay. They are nearly level to moderately sloping, moderately deep, strongly acidic, slowly permeable, and well drained. Gravelly land is composed of gravelly deposits with unidentifiable soil types due to severe erosion.

The western and central areas of the peninsula are primarily composed of tidal marsh and Keyport silt loam. Tidal marshes consist of materials ranging from sand to clay, with occurrences of peat and muck. The Keyport silt loam is a clayey silt loam that is slowly permeable.

A small area in the western end of the Stump Neck Annex is comprised of Mattawan loamy sand and cut-and-fill land. The Mattawan Series consists of soils that are nearly level to gently sloping, moderately well drained to well drained, and slowly permeable. These soils formed on uplands in a sandy mantle over loamy sediment. Cut-and-fill lands are areas where native soils have been removed and graded or filled with other material or soil.

The land around the Stump Neck Annex is heavily vegetated. There are five basic vegetation types present including pine, hardwood, pine-hardwood mix, tidal and non-tidal wetlands, and urban landscape. The hardwoods and pine-hardwood mix can be further subdivided into upland and wetlands divisions. Most of the forested land is either second or third growth; little, if any, virgin forest remains. The most abundant trees are Virginia pine, sweet gum, red oak, and yellow poplar.

Hardwood forest dominates approximately 1,075 acres (nearly 50 percent) of NSF-IH. Species common to the upland portions of hardwood forests include red, white, and chestnut oak, tulip poplar, and Hickories. The wetland portion is typically comprised of red maple, sweet gum, green ash, and American sycamore.

Along the shoreline of the Potomac River, the following species are common: black persimmon, grape, sea myrtle, false indigo, poison ivy, Virginia creeper, and phlox. In addition, the following grasses are present: gama grass, panic grass, bermuda grass, and finger grass. Marsh areas dominate along the shores of Mattawoman Creek and are characterized by jewelweed, alger, marsh cattail, weedgrass, sedge, three square bulrush, wild rice, saltmarsh cordgrass, smartweed, and marsh mallow.

10-3.5 Hydrology

The three primary waterways in the area are the Potomac River, Mattawoman Creek, and Chicamuxen Creek. The Potomac River and Mattawoman Creek border the Main Installation, and the Potomac River, Mattawoman Creek, and Chicamuxen Creek border the Stump Neck Annex. The Potomac River is a continuous, slow-moving, slightly brackish, tidal tributary to the Chesapeake Bay. Mattawoman Creek and Chicamuxen Creek are tributaries to the Potomac River and are also tidally influenced. Both have large floodplains and contain large expanses of tidal wetlands and swamps. Many small streams cross the area, most of which drain directly into one of the three major waterways.

The Stump Neck Annex is bordered by and contains large tracts of both tidal and non-tidal wetlands. Wetlands and floodplains are valuable habitats for wildlife, important groundwater recharge areas, and filters for surface water runoff, thus minimizing siltation and erosion. They are also important aesthetic buffers, scientific resources, and in some cases, recreational areas.

Based on the drainage divides presented in the 1983 Initial Assessment Study (IAS) conducted at NSF-IH, the majority of the natural drainage at the Stump Neck Annex flows to both Mattawoman Creek and Chicamuxen Creek (reference for IAS). Treated wastewater effluent is discharged directly to the Potomac River or Mattawoman Creek and is also discharged from outfalls to tributaries of these two waterways. The wastewater consists of industrial, sanitary, and storm effluents, or combinations thereof.

10-3.6 Hydrogeology

The hydrogeologic framework of the Indian Head area consists of a surficial aquifer and three major underlying confined aquifers: the lower Patapsco aquifer, upper Patuxent aquifer, and lower Patuxent aquifer. Although underlying the surficial aquifer, the upper Patapsco aquifer is considered a poor producer of groundwater in the area and is not considered to be a major aquifer at NSF-IH. Rather than continuous bodies of sands, the individual confined aquifers consist of multiple sand layers interbedded with lower permeability layers. The aquifers are described in detail below.

Shallow, unconfined to semi-confined groundwater at the NSF-IH occurs in the surficial aquifer from near surface to approximately 45 feet below ground surface (bgs), with water table elevations ranging from sea level to approximately 65 feet above msl. Depending on location, the surficial aquifer is composed of Quaternary paleochannel deposits, Tertiary to Quaternary upland deposits, the Aquia Formation, or sediments of Patapsco Formation. Typically, shallow groundwater occurs in perched water-bearing zones and is recharged from infiltration (Hart, 1983). In some lowland areas, surface water intrusion may be an additional source of recharge of the shallow aquifer along the edges of water bodies and during periods of high tide. Shallow groundwater flow follows topography and discharges to local surface water bodies.

The descriptions and hydrogeologic properties of the confined aquifers presented in this section are derived primarily from Andreasen (1999).

The top of the lower Patapsco aquifer lies at 70 to 200 feet below sea level in the study area, with a thickness ranging from 65 to 140 feet. The transmissivity of the lower Patapsco aquifer ranges from

about 190 to 700 square feet day (ft^2/d) near Indian Head. The aquifer is underlain by relatively low permeability sediments of the Patapsco Formation and by the low-permeability Arundel Clay. In most places the Arundel Clay serves as an effective confining unit between the lower Patapsco and upper Patuxent aquifers, although a hydraulic connection occurs where the Arundel Clay is thin or more heterogeneous.

The lower Patapsco aquifer is the principal water-supply aquifer at NSF-IH, and its potable water wells are typically screened in multiple sand layers within this aquifer at an average depth of 200 to 300 feet. These potable water wells serve approximately 4,050 people, including civilian and enlisted Navy employees and contractor employees. According to the installation, there are two potable wells located on the Stump Neck Annex. Well #42 SN was installed in 1945 and currently has minimal output. The other well, #2012 SN, was installed in 1953. NSF-IH is considering several rehabilitation options including closing #42 SN and using #2012 SN as the main well or installing of an additional well on Stump Neck Annex. There are also several private wells near Rum Point. These wells are tested quarterly or monthly, if regularly used. Although none of the NSF-IH wells supply reserves or residences beyond the facility boundaries, the lower Patapsco aquifer is used extensively for domestic and municipal water supplies in northwestern Charles County. Several production wells are screened in this aquifer northeast of NSF-IH, in and near the Towns of Indian Head and Potomac Heights.

The upper Patuxent aquifer lies at 400 to 600 feet below sea level in the study area and is about 50 to 70 feet thick. The transmissivity of the upper Patuxent aquifer ranges from about 150 to 2,600 ft^2/d in northwestern Charles County. Relatively low-permeability sediments of the Patuxent Formation underlie this aquifer.

The top of the lower Patuxent aquifer lies at 800 to 1,000 feet below sea level in the study area and is about 100 feet thick. Few potable water wells are screened in the lower Patuxent aquifer due to availability of water from the overlying confined aquifers. The lower Patuxent aquifer is underlain by crystalline basement rock. Water levels in the upper and lower Patuxent aquifer are generally similar due to the leaky nature of the intervening confining unit.

10-3.7 Cultural and Natural Resources

A Phase I Cultural Resources Survey and Supplemental Architectural Investigations were conducted at Stump Neck in 1996. As a result, 33 sites were identified and investigated. Only 17 of 33 were recommended for Phase II evaluations to determine their eligibility for listing on the National Register of

Historic Places. Five of these 17 sites that are considered to potentially overlap with former ranges discussed in this report are as follows:

- Site 18CH388 – overlaps with the Old Skeet and Trap Range (UXO-15) and the Marine Rifle Range (UXO-14)
- Sites 18CH391, 18CH628, and 18CH630 – overlap with the Rum Point Skeet Range (UXO-16)
- Site 18CH644 – overlaps with the Small Arms Range (UXO-17)

The architectural evaluation verified three National Register-eligible historic districts and identified one additional district, as follows: the Indian Head Residential Historic District containing 64 contributing resources; the Naval Powder Factory District containing 124 contributing resources; the Naval Proving Ground Historic District containing no contributing resources; and the Extrusion Plant Historic District containing 63 contributing resources. An additional area of the base, the Polaris facility, was recommended for further research, as it was considered potentially eligible for the National Register as an exceptionally significant area. The exact locations of the contributing resources within these districts was not available.

10-3.8 Endangered and Special Status Species

According to the 1997 Wildlife Management Plan, three endangered species are reported to be located within the Stump Neck Annex. Two of these three species, the rainbow snake and joint-vetch (flowering plant of the pea family), are federally endangered. The third species, the scaly blazing-star (perennial herb), is a species of special concern in the State of Maryland. As of the July 2001 Threatened and Endangered Plant/Animal Species of Charles County, Maryland report, the Maryland Department of Natural Resources Wildlife and Heritage Service still listed these three species as federally and state endangered for Charles County.

Protected species that are known to or have the potential to inhabit Stump Neck Annex are listed in Table 10-3.

10-4 NEARBY POPULATIONS

Charles County contains approximately 261.5 people per square mile (mi^2) according to the 2000 United States Census. NSF-IH and its tenant commands employ approximately 3,600 military and civilian personnel. Indian Head is the county's largest employer. Over 76 percent of the employees at the base live within Charles County. Approximately 500 military and family members live on the installation;

however, no military or family members live on Stump Neck Annex. Recreation on and around the installation includes hunting and fishing by permit. The Chicamuxen WMA is located approximately 245 feet downstream of the Marine Rifle Range, and it is used by recreational hikers, hunters, and fishers. Residential properties are located 2 miles south of the range.

TABLE 10-1

**SUMMARY OF OTHER THAN OPERATIONAL RANGES
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND**

Site Name	NORM Site No.	Size (acres)	Use	Dates of Use
Marine Rifle Range	UXO 14	30.44	Rifle range	1911-1918
Old Skeet and Trap Range	UXO 15	29.33	Skeet and trap range	1966-1991
Roach Road Rifle Range	UXO 25	0.27	Rifle range	1967-1980s
Rum Point Skeet Range	UXO 16	33.45	Skeet and trap range	1991-2001
Small Arms (Pistol) Range	UXO 17	2.41	Pistol range	Mid-1980s - 1991

TABLE 10-2

**TIMELINE OF HISTORICAL EVENTS
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND
PAGE 1 OF 2**

Time Period	NSF-IH Milestones
1890 - 1900	• Constructed on 659 acres on Cornwallis Neck in 1890 as the Naval Proving Ground to test guns, armor, shells and mounts. • Within 1 year, added the 222.75-acre Mount Pleasant Farm.
1900 - 1910	• Factory constructed for smokeless powder production. • Stump Neck Annex property purchased in 1901 to extend firing range.
1910 -1920	• Work gradually increased from proving of guns and armor to include standardization of shells and powder. • Acquired 1,160 acres of land adjacent to the Main Installation in 1918. • 161 acres acquired for a railroad right-of-way running from the Naval Proving Ground to the Pennsylvania Railroad junction at White Plains, Maryland; 13.8-mile railroad spur constructed.
1920 - 1940	• Mission gradually shifted from a Naval gun proving ground to a chemical factory, research laboratory, and Explosive D factory. • Facility changed name to the Naval Powder Factory. • All proving ground activities were moved to Dahlgren, Virginia.
1940 - 1950	• Navy established Explosives Investigation Laboratory where extensive examination of captured enemy ordnance was performed. • Practical applications for the EOD School moved from Washington, D.C. to Stump Neck Annex. • Joint forces EOD School led by Navy formed in 1947. • Propellant research and development added to installation mission. • Jet Propulsion Research Lab founded (1940-1944).
1950 - 1960	• Facility changed its name to the Naval Propellant Plant. • Research and development on the Polaris and other rocket programs began.
1960 - 1980	• Rum Point, an 80-acre promontory in Mattawoman Creek, was acquired by condemnation in 1966. • Bullets Neck, a separate 47-acre promontory in Mattawoman Creek, was purchased in five small acquisitions (1965-1966). • The Naval Propellant Plant changed its name to the Naval Ordnance Station to reflect the diversification from propellants into related fields of chemistry, engineering, and production contract management.

TABLE 10-2

TIMELINE OF HISTORICAL EVENTS AT NSF-IH
NSF-IH
INDIAN HEAD, MARYLAND
PAGE 2 OF 2

Time Period	NSF-IH Milestones
1980 - 1990	• Full-scale production at the Naval Ordnance Station concentrated on several processes/products too unprofitable, too dangerous, or too difficult for the private sector. • The Naval Ordnance Station became the center of excellence for the following technologies: guns, rockets and missiles; energetic chemicals; ordnance devices; missile weapon simulators; explosive process development engineering; and explosive safety, occupational safety and health, and environmental protection.
1990 - present	• EOD School on Stump Neck closed in 1999. • Currently, the mission of NSF-IH is to ensure operational readiness of U.S. and allied forces by providing the full spectrum technical capabilities necessary to rapidly move any “energetics” product from concept through production to operational deployment.

TABLE 10-3

**SUMMARY OF KNOWN OR POTENTIAL PROTECTED SPECIES
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND**

Ecological Receptor	Species
Federal Endangered	• Rainbow snake
	• Joint-vetch
Federal Threatened	None reported
State Endangered	Scaly blazing-star
State Threatened	None reported

Sources of data include:

- NSF-IH Wildlife Management Plan, 1997.
- Threatened and Endangered Plant/Animal Species of Charles County, Maryland, July 2001, Maryland Department of Natural Resources.

REFERENCES (WORKSHEET #10)

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United States Department of Agriculture Soil Conservation Service, 1974. Soil Survey of Charles County, Maryland. Prepared by: Richard L. Hall and Earle D. Matthews, Soil Conservation Service, Prepared for: Department of Agriculture in cooperation with the Maryland Agriculture Experiment Station, July.

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INDIVIDUAL MRP SITE-SPECIFIC CHARACTERISTICS/PROBLEM DEFINITIONS

The following SAP worksheets (#10.1 through #10.5) provide specific information about each of the other than operational ranges located on Stump Neck Annex, NSF-IH, Maryland, including history and range description, land use, access controls and restrictions, visual survey observations and results, contaminant migration routes, and receptors.

SAP WORKSHEET #10.1:
Problem Definition/Conceptual Site Model for Marine Rifle Range (Site UXO-14)

10.1 MARINE RIFLE RANGE

10.1.1 History and Site Description

The Marine Rifle Range was used for small arms training from approximately 1911 to 1918 and was identified in the Navy Range Inventory. The estimated limits of the Marine Rifle Range, shown on Map 10.1-1, were mapped based on field reconnaissance, additional data collection, and historical maps. Based on this evaluation, the size of the Marine Rifle Range was estimated in the PA at 30.44 acres. The boundaries of the range were altered from the Navy Range Inventory to incorporate the firing lines and target butts identified on historical maps. The boundaries were also changed to exclude the areas occupied by other MRP sites. The Safety Danger Zone (SDZ) developed for the Marine Rifle Range covers approximately 1,369 acres.

Figure 10.1-1: View of Marine Rifle Range looking West toward Building 2195.

Site walks completed during the preliminary SI and review of historical records and photographs indicated that the hillside embankment located behind the main targets should be included in the site because it most likely received the majority of bullets passing through or over the intended targets (Map 10.1-2). Bullets landing below the targets impacted the target berms.

The range is located on the Stump Neck Annex, south of Archer Avenue, and parallel to Archer Avenue from the causeway west to Building 2105 (Figure 10.1-1). The western portion of the range is developed, and the central and eastern portions of the range are wooded with sporadic structures. Earthen mounds

are present in the woods along Archer Avenue and east of Building 2075. The Marine Rifle Range is also surrounded by the Air Blast Pond to the south, Torpedo Burial Site to the south, and Old Skeet and Trap Range to the north. When the Marine Rifle Range was an active training area, two range houses, barracks, and associated support buildings were present at the range.

The estimated period of use, 1911 to 1918, is based on historical maps and documents. The Marine Rifle Range, as identified on maps from 1913, 1915 and 1918, is also referred to as the Rifle Range, Winthrop, Maryland. The range was identified on a January 1913 Government Reservation at Stump Neck, Maryland map, which was obtained from the National Archives (Figure 10.1-2). Photographs of the conditions at the Marine Rifle Range in 1913 were also obtained from the National Archives (Figures 10.1-3, 10.1-4, 10.1-5, and 10.1-6). The 1913 map obtained from the National Archives (as well as the installation and Navy historian) provides details of the layout of the Marine Rifle Range, including buildings, target butts, and firing lines. The 1913 map shows the location of the range within the installation and a rough outline of the range (see Figure 10.1-2). According to the 1918 Annual Report from the Commandant of the Marine Corps, this range was used for Marine recruits from the Norfolk and Philadelphia depots for small arms training with rifles. An April 1917 memorandum from the Marine Corps states that the range was still in use and that it was dangerous to enter the portion of the reservation to the rear of the rifle butts. The 1918 Annual Report of the Chief of the Bureau of Ordnance states that “the buildings vacated by the Marines on Stump Neck were moved to Machodoc Creek (Virginia) to form the nucleus of the station.” A 1931 memorandum from the Major General Commandant of the Marine Corps states that the buildings at Winthrop were “understood to be quite dilapidated and of no value.” A 1949 map showing the conditions at Stump Neck lists this area as “cultivated.” Therefore, it is assumed that the range was closed between April 1917 and 1918.

Mr. Miller, retired Marine Corps EOD, stated that the rifle range paralleled the Potomac River. He also stated that pistols may have been used at the range and that an embankment was present behind the targets, as seen on Figure 10.1-6.

Details from the 1913 map are shown on Figure 10.1-2. Two target butts were located at the eastern end of the range. Two sets of firing lines, one for each target butt, were located at 100-meter increments to a maximum of 1,000 meters. Range houses, barracks, and other support buildings were also identified on the 1913 map (Figure 10.1-4). According to historical photographs (Figure 10.1-6), there were 13 targets in each target butt. The numbers of targets, locations of firing lines, and recommended Small Arms Range Design and Construction guidance document were used to establish the SDZ, as shown on Map 10.1-1. The SDZ for the Marine Rifle Range extends over Chicamuxen Creek, Potomac River, and east

over the Stump Neck Annex and off post. These features were used to establish the size, shape, and orientation of the Marine Rifle Range as it appears in this UFP-SAP for the SI.

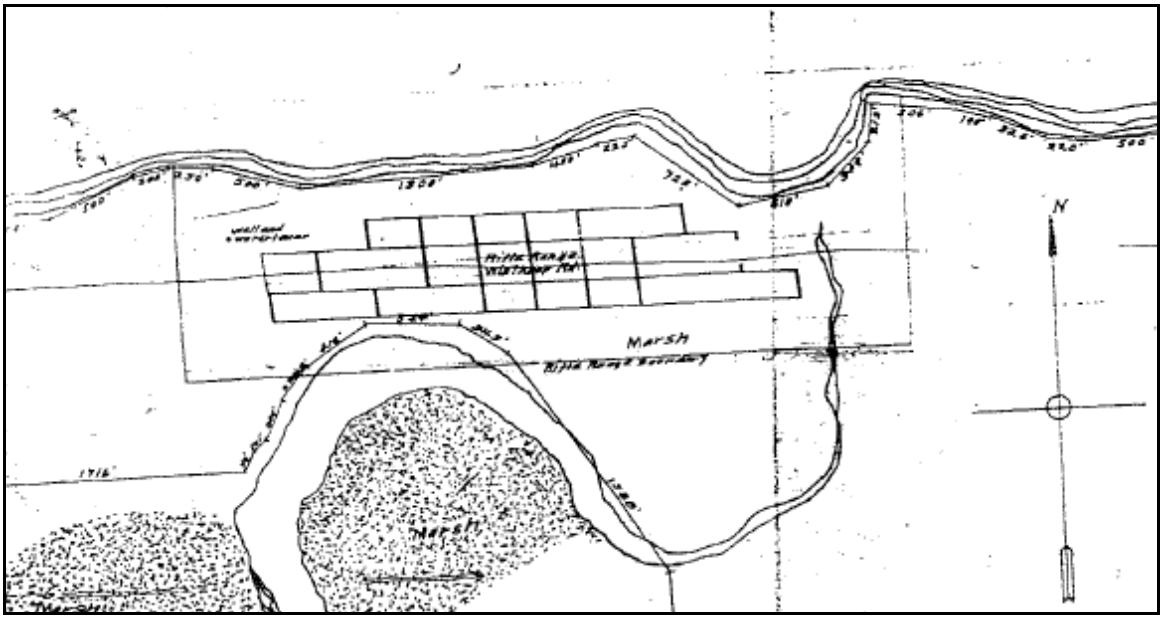

Figure 10.1-2: Rifle Range, Winthrop, Maryland as shown on 1913 installation map

Figure 10.1-3: October 19, 1913. Rifle Range at Winthrop, Maryland, showing firing line

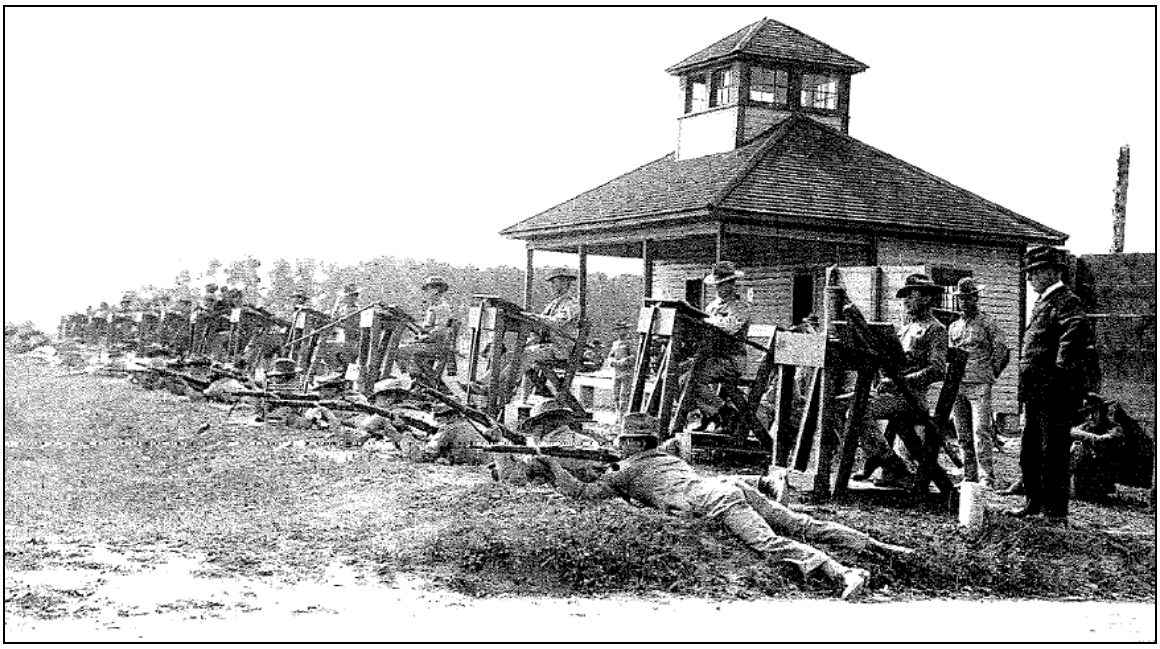

Figure 10.1-4: October 19, 1913. Rifle Range at Winthrop, Maryland, showing firing line and range house

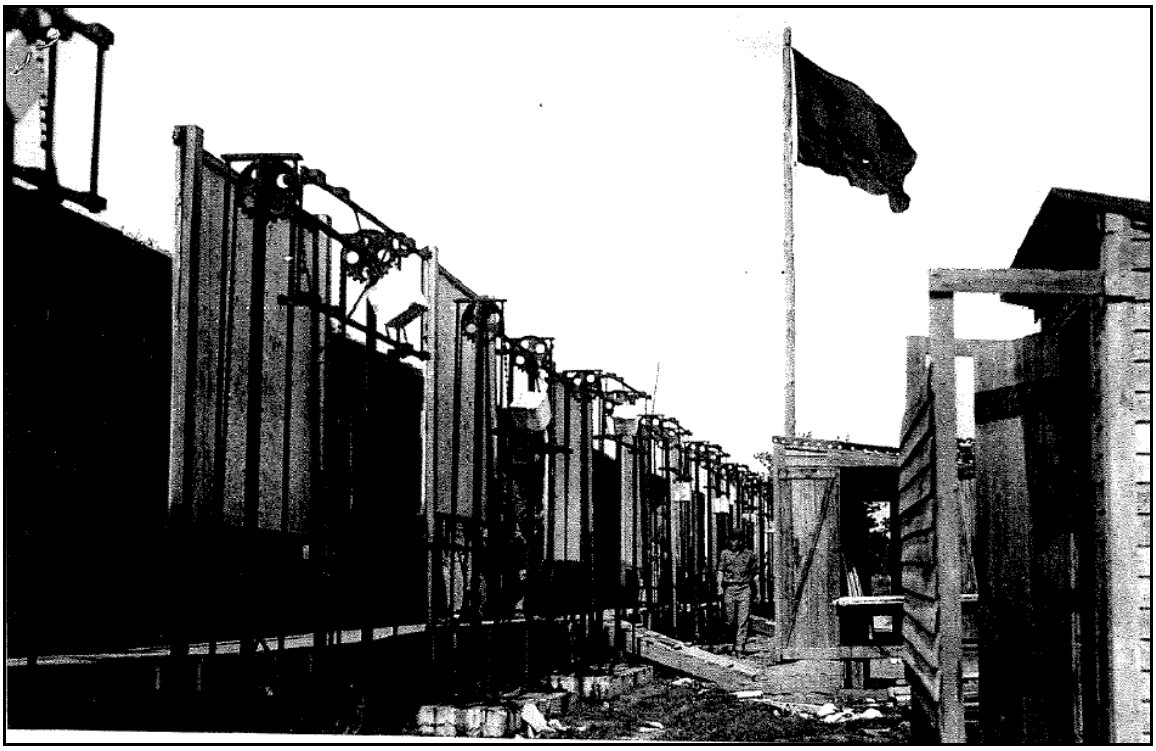

Figure 10.1-5: October 19, 1913. Rifle Range at Winthrop, Maryland, showing target mechanisms

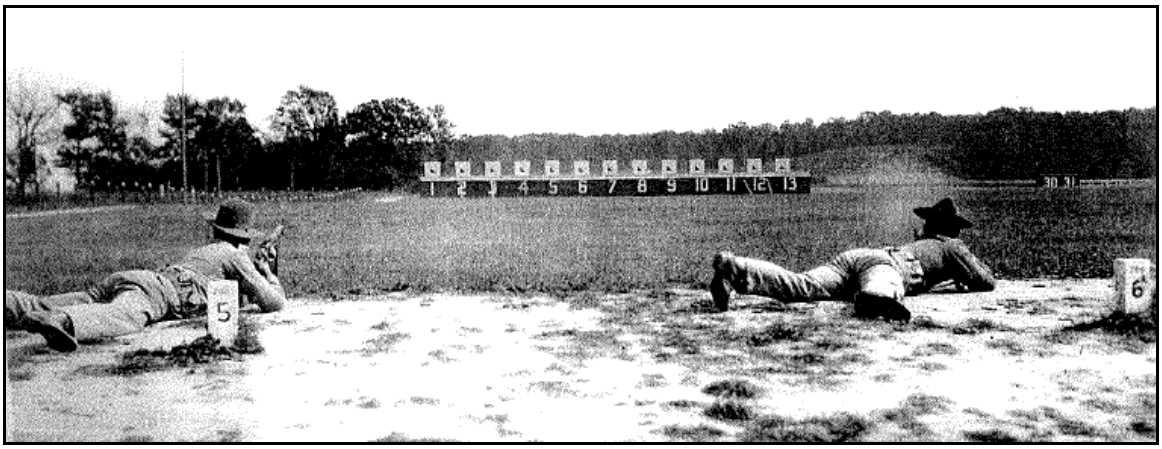

Figure 10.1-6: October 19, 1913. Rifle Range at Winthrop, Maryland, showing targets and embankment

The 1914 Annual Report stated that the Marine Rifle Range was a source of delay for the Proving Ground. Despite precautions taken at the range, the requirements at the rifle range made it necessary to suspend or curtail work at the Naval Proving Ground. However, work from the Proving Ground also caused delays and interferences with training at the rifle range when projectiles where fired toward Stump Neck. The dwellings and barracks on Stump Neck were close to the line of the 10-inch, 12-inch, and 14-inch guns and were inside the SDZ established by the Navy. Although stringent orders existed to vacate the Marine Rifle Range when firing over it was necessary, there was always an element of danger. According to a 1911 Bureau of Ordnance memorandum, no projectiles had been reported to have landed in the rifle range. The Marine Rifle Range was relocated to MCB Quantico in 1918 to eliminate the interferences between training at the rifle range and at the Proving Ground.

The Marine Rifle Range is not identified as a SWMU or as an Installation Restoration (IR) range; however, the following SWMUs are overlapped by or adjacent to the Marine Rifle Range:

- SWMU #6 – Air Blast Pond
- SWMU #8 – Tool Burial Site – Site 34
- SWMU #9 – Torpedo Burial Site – Site 35
- SWMU #12 – Waste Oil Storage Site
- SWMU #18 – Waste Pile

The overlapping areas are not included in the estimated acreage for the Marine Rifle Range.

Topography

Section 10-3.2 provides a general description of topography for the Stump Neck Annex. The terrain at the Marine Rifle Range is relatively flat. Elevations range from 5 to 30 meters above msl. The central and eastern portions of the range are relatively flat at an elevation of approximately 10 meters above msl. The western portion of the range has a slight slope from 10 to 30 meters above msl. Wetlands border the range to the east, and wetlands also surround an unnamed tributary in the central portion of the range. Due to the topography of the area and the complete vegetative ground cover, severe erosion is not expected to occur.

Geology

Section 10-3.3 provides a description of the geology at the Indian Head Stump Neck Annex, which is applicable to the Marine Rifle Range. No range-specific geological information was available for the Marine Rifle Range.

Soil and Vegetation Types

Section 10-3.4 provides a description of the soil and vegetation types at the Stump Neck Annex. Vegetation at this range is a mixture of mowed grass, grass fields, hardwood forest, and wetlands, as illustrated in Figure 10.1-7. The wetland area within the range is classified by the installation as a Palustrine forested broad-leaved deciduous wetland. According to the Soil Survey of Charles County, soils in the area consist of silty sand with gravel at the ground surface underlain by silty sand with clay. Specifically, Keyport silt (western portion), Elkton silt loam (central portion), and Mattapex silt loam (eastern portion) are present at the range.

Figure 10.1-7: View of grassland at Marine Rifle Range

Hydrology

Section 10-3.5 provides a description of hydrology at Indian Head and the Stump Neck Annex. Wetlands are located on and bordering the eastern portion and in the central portion of the Marine Rifle Range surrounding an unnamed tributary. Surface water drains to two unnamed tributaries, one in the central portion and one in the eastern portion of the range. Surface water also drains to drainage swales located along Archer Avenue and surrounding the building in the western portion of the range. The surface water drains to the wetlands and Chicamuxen Creek, both of which drain to the Potomac River. A portion of Marine Rifle Range along the wetlands is located within the 100-year floodplain.

Hydrogeology

Section 10-3.6 provides a description of hydrogeology for Indian Head and the Stump Neck Annex, which is applicable to the area of the Marine Rifle Range. It is assumed that shallow groundwater flow from the Marine Rifle Range follows topography and is connected to the area's dominant surface water bodies (Mattawoman Creek, Chicamuxen Creek, and Potomac River). No monitoring wells are located within the Marine Rifle Range; however, one of the potable production wells for the Stump Neck Annex, #2012 SN, is located across Archer Avenue.

Cultural and Natural Resources

General cultural and natural resources for Stump Neck Annex are discussed in Section 10-3.7. Because a portion of this range is undeveloped, there is the potential for wildlife to exist at the range. According to the 2003-2004 Stump Neck Annex Hunting Map, hunting is permitted in the undeveloped portions of the Marine Rifle Range. The Chicamuxen WMA is located approximately 75 meters south of the Marine Rifle Range.

The Phase I Cultural Resources Survey of Stump Neck Annex and Supplemental Architectural Investigations from August 1996 identified two sites within the Marine Rifle Range. The sites and associated locations of positive prehistoric and historic findings are identified on Figure 10.1-8. A reinforced concrete retaining wall approximately 130 feet long and 33 feet high was identified in the area bordering the marsh on the eastern edge of the Marine Rifle Range. No sites were identified within the range Maryland Register of Historic Places (State Register) or the National Register of Historic Places.

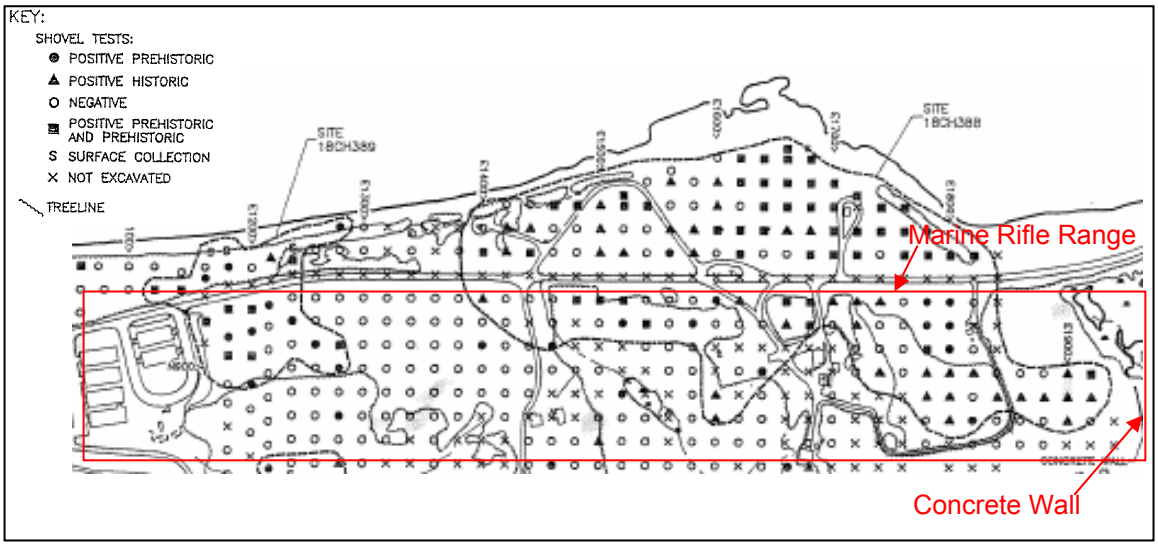

Figure 10.1-8: Cultural resources identified in shovel test pits

Endangered and Special Status Species

Endangered or special status species located on the Stump Neck Annex are discussed in Section 10-3.8. These species potentially inhabit the Marine Rifle Range.

10.1.2 Visual Survey Observations and Results

A visual survey of the Marine Rifle Range was conducted during a range visit on June 26, 2003. Malcolm Pirnie personnel who conducted the range visit included Mr. Dinh, Mr. Egholm, Ms. Tegtmeyer, Mr. Hains, Mr. McManus, Mr. Wiley, Mr. Baker, and Mr. Rice. Ms. Morgan and Mr. Jorgensen of the NSF-IH Environmental Office accompanied the team. The Marine Rifle Range was inspected through several transects across the former range. Numerous pieces of metallic and wood debris were identified in the woods during the visual survey. The metallic debris was determined to be non-ordnance or munitions related. Earthen mounds were observed in the woods along Archer Avenue and east of Building 2075, and these may be the remnants of the embankments discussed in Section 10.1.1. No ordnance, MEC, or related debris were observed during the range visit.

Figure 10.1-9: View of earthen mound identified during visual survey

Figure 10.1-10: Metal debris observed in the woods at the Marine Rifle Range

A series of site walks was conducted with the SI planning project team during the late fall and winter of 2007. An initial site walk of all of the Stump Neck Annex SI MRP sites was completed on September 18, 2007, by Ralph Basinski (Tetra Tech), Joe Rail (Navy RPM), and Shawn Jorgensen (Indian Head-Environmental Department) as part of the project kick-off meeting. A subsequent site walk was conducted on November 8 to 10, 2007, and included a larger contingent of the project team: Joe Rail (Navy RPM), Shawn Jorgensen (Indian Head Environmental Department), and several Tetra Tech personnel (Ralph Basinski, Ralph Brooks, Jim Coffman, George Latulippe, and Rick Barringer). Each MRP site was walked, and multiple photographs were taken to document conditions observed during the site visit (Figures 10.1-9 through 10.1-12). The site walks were performed in conjunction with technical meetings at the facility that reviewed the proposed approaches for the MC fieldwork programs.

Figure 10.1-11: Concrete Wall Backing Target Butt on the Former Marine Rifle Range Observed During SI Site Walk

An expanded site walk/meeting was conducted on December 18 and 19, 2007, and included previously identified Navy and Tetra Tech personnel and representatives from MDE (Curtis Detore) and USEPA Region 3 (Dennis Orenshaw). As before, MRP sites were walked and photographed to record the conditions observed at the sites. Project quality objectives for small arms ranges were discussed with the meeting attendees on December 19, 2007.

Figure 10.1-12: View from Concrete Wall Backing Target Butt on the Marine Rifle Range Looking into Adjacent Wetlands Area.

As related to the Marine Rifle Range, the following additional observations were made based on the conditions at the site. The PA Report used the 1915 range map to develop the site boundaries for the Marine Rifle Range. As illustrated in the historical photographs presented in the PA Report (Malcolm Pirnie, 2005), the small arms targets were raised and held above the target butts during firing by a series of chains and mechanical devices (Figure 10.1-5). Expended bullets passing through the targets or passing above the targets would continue on a trajectory that would continue beyond the boundary of the Marine Rifle Range as depicted in the PA Report. An improved range boundary proposed for the SI includes the downrange hillside on the eastern side of Roach Road that would most likely have received the fired ammunition from the Marine Rifle Range. As shown on Figure 10.1-12, a wetlands area is located between the southernmost target butt and downrange earthen hillside (embankment) adjacent to Roach Road.

Based on these observations, there should be two primary areas for the accumulation of expended small arms ammunition on the Marine Rifle Range. Those bullets missing below the range targets would tend to accumulate in the target butt soil below the targets. Those fired bullets that successfully hit the targets or overshot the targets most likely continued on a trajectory that would continue beyond the target butts and into the hillside embankment beyond the downrange wetlands (next to Roach Road) as shown on Map 10.1-2.

10.1.3 Munitions and Munitions-Related Materials Associated with the Site

The Marine Rifle Range was used for small arms training. Historical maps and documents state that rifles were used at the range. Reportedly pistols were also used at the range. Specific ordnance types used at the range are not known; however, general rifle ammunition from the early 1900s included .30-caliber ammunition. According to the Army Technical Manuals (AR 750-10, TM 9-855), the maximum range for .30-caliber ammunition is 10,350 feet and the muzzle velocity is 2,700 feet per second. The SDZ extends from the end of each firing line in a 30-degree angle for 3,000 feet down range, at which point it continues for an additional 8,700 feet parallel to the direction of fire. An example of a typical SDZ for a rifle range is provided on Figure 10.1-13. The SDZ for the Marine Rifle Range covers 1,369 acres and is shown on Map 10.1-1. The SDZ is the location where projectiles were fired over or may have landed.

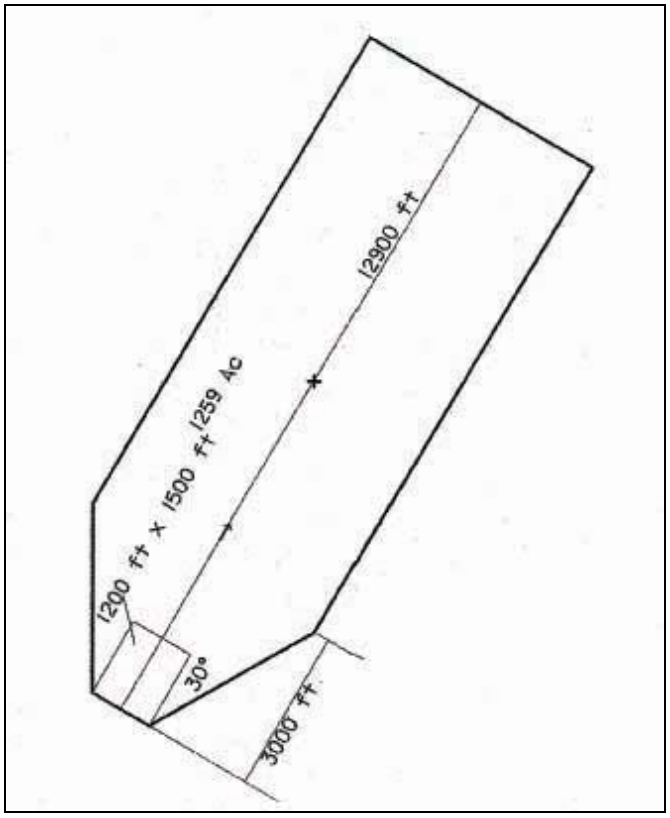

Figure 10.1-13: Typical SDZ for a 500-Yard Rifle Range

- Lead bullets/fragments is suspected within the target butts. Nitroglycerin (NG) from unburned powders and lead from primers is suspected at the firing points.

No munitions-related debris items were identified at the Marine Rifle Range. No bullets or metallic debris were reported to have been identified in the shovel tests conducted for the Phase I Cultural Resources Survey of Stump Neck Annex (reference).

Based on the information obtained during the data collection process, the Marine Rifle Range is not suspected to contain chemical weapons material (CWM) filled munitions, electrically fuzed munitions, or depleted uranium (DU)-associated munitions.

10.1.4 MEC Presence

The entire range has been subdivided and categorized into one of three levels of MEC presence including known MEC areas, suspected MEC areas, and areas where no evidence exists to indicate that MEC are known or suspected to occur. These categories are discussed below. The Marine Rifle Range is overlapped by the firing fan from the Valley located at NSF-IH, Main Installation. Thus, there is a potential for munitions associated with the Valley to be present at the Marine Rifle Range. However, a 1911 memo from the Bureau of Ordnance states that no projectiles were reported to have landed on the Marine Rifle Range. Technical data sheets can be found in Appendix D of the PA (Malcolm Pirnie, 2005). Only MEC presence specifically related to the Marine Rifle Range was considered.

Known MEC Areas

There are no readily apparent MEC areas associated with the Marine Rifle Range because expended small arms ammunition do not pose an explosive hazard.

Suspected MEC Areas

There are no suspected MEC areas expected at this range because expended small arms ammunition do not pose an explosive hazard.

Areas Not Suspected to Contain MEC

Expended small arms ammunition are not explosive; therefore, the entire 35-acre range is not suspected to contain MEC.

10.1.5 Ordnance Penetration Estimates

For small arms ranges, the Interstate Technology and Regulatory Council (ITRC) has prepared a document titled Characterization and Remediation of Soils at Closed Small Arms Firing Ranges, dated January 2003, to provide information on the general layout of small arms ranges, as well as information on areas that may be impacted with MC and/or MEC as a result of range use and the characteristics of the munitions used. According to the ITRC guidance, the penetration depth of small arms ammunition on the range floor is 1 foot or less. The document states that rounds that impact the range floor typically had a flat trajectory that fell short of or missed the target, or resulted from ricochet, and these fragments are usually found within the top 6 inches of soil. MC at the MR Range is expected to be concentrated in the target butts.

10.1.6 Munitions Constituents

For small arms ammunition, the primary MC of concern is lead from shot. Other associated MC less likely to be of concern may include antimony, arsenic, copper, nickel, and lead styphnate/lead azide. These MC components are not consumed when the munitions items function as they are designed. Therefore, these MC may exist at the Marine Rifle Range. NG from unburned propellants and lead from primers is suspected at the firing points. There was no record found of environmental sampling on the range.

10.1.7 Contaminant Migration Routes

Contaminants at the Marine Rifle Range may potentially migrate within soil, groundwater, surface water, and sediment. The Marine Rifle Range's proximity to Chicamuxen Creek and its unnamed tributaries provides possible migration routes to surface water. Storm water discharges to surface water via overland flow. Groundwater flow in the shallow water table aquifer also likely trends towards Chicamuxen Creek; therefore, MC leaching from soil into shallow groundwater may migrate to surface water. Sediments can act as contaminant repositories, and sediment mixing and dredging can act as migration routes to surface water. MC in surface soil and sediment may migrate via plant/animal uptake. Direct human or biota contact with surficial and subsurface soil is possible if the soil is disturbed. Based on a review of hydrogeological data, it is unlikely that MC in shallow groundwater would migrate to the deeper aquifers that are used as water supplies. However, shallow groundwater is still considered a potential exposure medium.

10.1.8 Receptors

Potential current and future human receptors include authorized Navy personnel (military and civilian), visitors, contractors, maintenance workers, recreational users, and trespassers. Plant and animal biota are also potential receptors. Examples of ecological receptors include deer, wild turkey, and waterfowl.

Buildings Near/Within Site

Building 2195 is located at the western edge of the range and is home to Code 45 as the Joint Services EOD Equipment Support Facility. Additional buildings surrounding Building 2195 are used for offices and storage. A fenced maintenance yard including Building 2156 and the former range of Buildings 2019 and 2101 are located in the central portion of the range. Building 2075 at the eastern edge of the range is a former shredder, which is currently condemned. The majority of the buildings were constructed after the Marine Rifle Range was closed.

Refer to Table 10.1-1 for details on the buildings at the Marine Rifle Range. The information was obtained from historical maps and the Indian Head building database.

Utilities On/Near Site

Electrical, telephone, sanitary sewer, and potable water lines run through the Marine Rifle Range. The primary electrical, sanitary sewer, and potable water lines are adjacent to and parallel Archer Avenue. Secondary utility lines service the buildings within the footprint of the Marine Rifle Range.

10.1.9 Land Use

Information on land use at the Marine Rifle Range prior to 1911 could not be found. From 1911 until 1918, the range was maintained as a rifle range for the Marines. After the Marines relocated, the buildings were abandoned and eventually demolished to make way for development of the Stump Neck Annex. Buildings within the Marine Rifle Range are primarily used as offices and storage. The west portion of the range contains Building 2195, which is used by the Joint Services EOD Equipment Support Facility. The central and eastern portions of the range are primarily undeveloped with sporadic structures (Buildings 2156 and 2075). There is a fenced maintenance yard in the central portion of the range that is used to store equipment (e.g., dump trucks, backhoes). The wooded area surrounding the existing buildings and the wetland area in the eastern portion of the range are currently unused. According to the installation personnel, there are no planned changes to activities at the Marine Rifle Range.

10.1.10 Access Controls/Restrictions

No public access is authorized at NSF-IH. Signs, partial fencing, locked/secured gates, login book/office check-in, and vehicle security patrols are used to control the entire facility. There are no access control features specific to the Marine Rifle Range. According to the 2003-2004 Stump Neck Annex Hunting Map, hunting is permitted within the undeveloped portions of the Marine Rifle Range.

The wetlands located on the eastern edge of the range are protected under United States Executive Order 11990, which prohibits construction in a wetland area unless there is no practicable alternative and all possible measures are taken to minimize the environmental impacts. Wetlands are also protected under Section 404 of the Clean Water Act, which requires a permit to be obtained from the United States Army Corps of Engineers (USACE) before any work in a wetland can commence. The wetlands found at the Marine Rifle Range are under the category of Palustrine forested broad-leaved deciduous wetland.

A portion of the Marine Rifle Range is located in the 100-year floodplain surrounding the wetlands. Executive Order 11988 restricts development within the 100-year floodplain to water-dependent activities. Any construction within the floodplain must be in accordance with regulations promulgated by the Federal Insurance Administration pursuant to the National Flood Insurance Act of 1968. Permits for construction within the 100-year floodplain are also required and are administered by the Waterway Permits Division of the Maryland Department of Natural Resources.

10.1.11 Conceptual Site Model

A general description of the CSM exposure pathway analysis is included in Section 10.1.7. For the purpose of this SAP, only MC associated with the Marine Rifle Range is considered in the CSM exposure pathway analysis.

For the Marine Rifle Range, historical and visual evidence indicate that MEC are not present. Therefore, no complete exposure pathways exist for MEC and no exposure pathway analysis was completed for MEC. Soil and surface water/sediment impacted by MC represents a primary potential source medium, as illustrated in the CSM (Map 10.1-2). Exposures to surface soil, subsurface soil, and surface water/sediment containing MC presents potentially complete pathways for human and ecological receptors. The MC exposure pathway analysis for the Marine Rifle Range is presented in Figure 10.1-14. All human and ecological receptors have potentially complete exposure pathways for direct contact with MC in surface soil at the embankment behind the former target butts. These pathways include dermal contact, ingestion, and inhalation (dust). Runoff, discharges, and/or erosion may transport the MC from

surface soil to surface water/sediment. Because there are two tributaries at the Marine Rifle Range, potentially complete pathways also exist for all human and ecological receptors of surface water/sediment.

Soil also represents an exposure medium when considering plant/animal uptake for biota (including game such as deer and wild turkey) and human receptors consuming the affected biota (e.g., hunted game). There is a potential for the MC present in surface soil to infiltrate to the subsurface. Potentially complete exposure pathways exist for MC in subsurface soil (direct contact, ingestion and inhalation during intrusive work activities) for all human and ecological receptors with the exception of trespassers. It is anticipated that trespassers would not come in contact with subsurface soil. Although confining layers are expected to prevent the migration of MC to the lower aquifers used for water supplies, potentially complete pathways exist for MC in shallow groundwater for human receptors.

10.1.12 Problem Definition Summary

The following is a summary of the problem definition that is detailed in Worksheet #10.1:

- Environmental contamination may exist at the Marine Rifle Range (30.5 acres) because the facility was an active range from 1911 through 1918. Specific small arms ammunition types and materials used at the Marine Rifle Range include general rifle ammunition from the early 1900s (e.g., .30-caliber ammunition). Expended ammunition is suspected along the range floor immediately in front of the target embankments, in the target embankments themselves, and in the hillside east of the target embankments, which would have been impacted by bullets shot through, over, or alongside the targets.

- MC consisting of metals (primarily lead and, to a lesser extent, antimony, arsenic, copper, tin, and zinc) and NG may be present in site soil (particularly surface soil [0 to 6 inch interval]) and surface water and sediment of any drainageways leading from the range to the adjoining unnamed tributaries, wetlands, and Chicamuxen Creek. Lead is assumed to be the primary metal MC of concern because it is the primary constituent in the spent munitions and because of its documented toxicity to both human and ecological receptors. It is anticipated that other metals contamination will be spatially correlated with lead concentrations because they are associated with the lead in the bullets. MC, if present in surface soil, may have migrated to subsurface soil and subsequently infiltrated to the underlying groundwater. These constituents may also have migrated to the aforementioned adjoining surface water bodies via surface soil runoff or the discharge of contaminated shallow groundwater to

surface water. However, the nature and extent of the potential environmental contamination is not known at this time (no environmental sampling has been conducted to date).

- The CSM (detailed in Worksheet #10.1) indicates that potentially complete exposure pathways exist for both human and ecological receptors under both current and hypothetical future land uses. However, potential human and ecological risks have not been characterized for receptors exposed to the potentially contaminated environmental media. Unacceptable levels of human and/or ecological risk may exist.

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FIGURE 10.1-14

MARINE RIFLE RANGE
MC EXPOSURE PATHWAY ANALYSIS
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

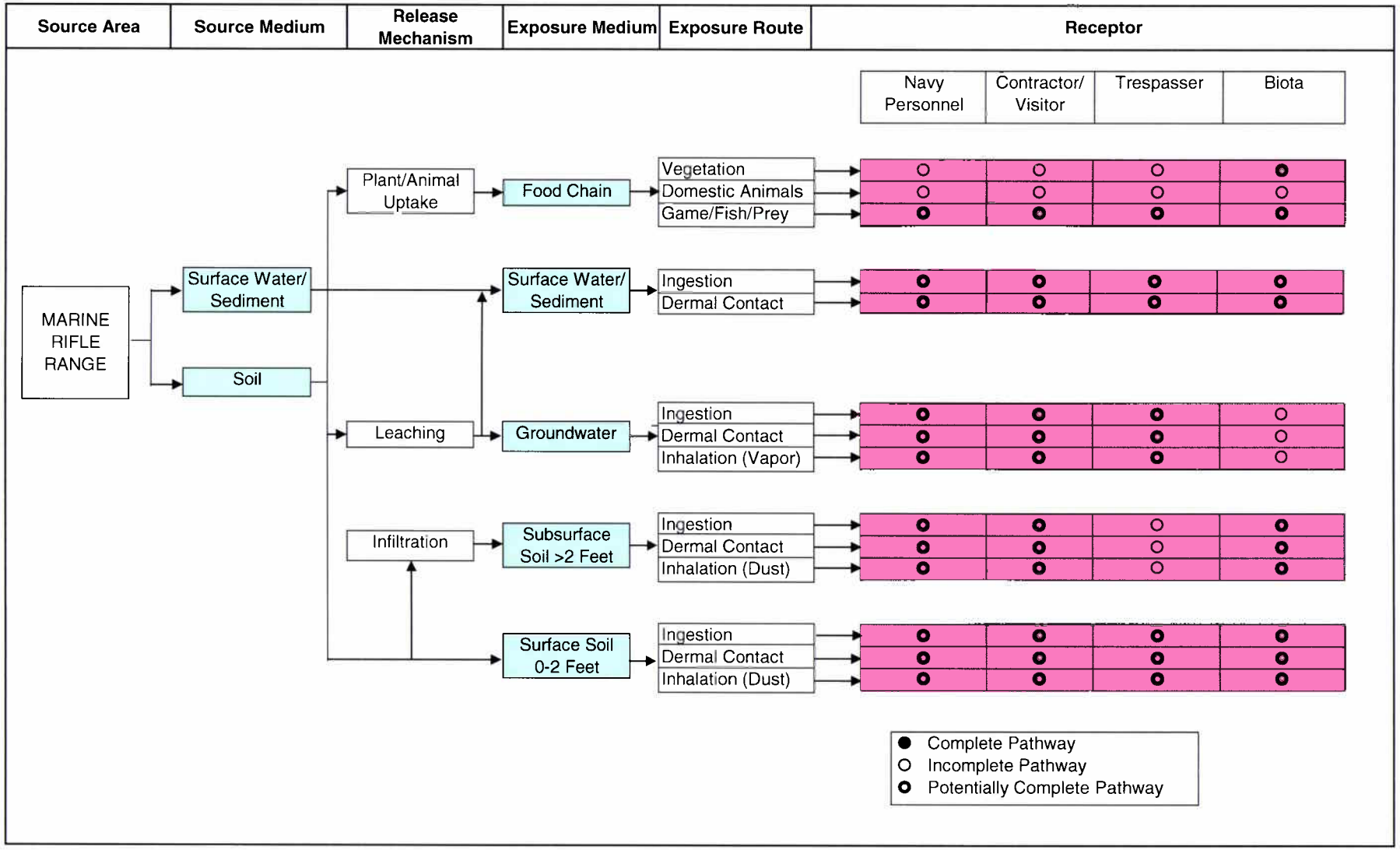


TABLE 10.1-1

**MARINE RIFLE RANGE BUILDING DESCRIPTIONS
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND**

Building	Location	Date Built	Use	Status
2019	Central portion	1962	Offices Shredder	Demolished
2075	Eastern portion	1949	Building	
2101	Central portion	1972	Shelter	Demolished
2105	Western portion	1974	Storage	Demolished
2128	Western portion	1988	Storage Equipment Maintenance	Demolished
2147		2002	Support	
2147	Western portion	1990	Storage	
2148	Western portion	1990	Storage	
2150	Western portion	1991	Office and storage	
2151	Western portion	1991	Storage	
2155	Western portion	1991	Offices	
2156	Central portion		Fenced storage yard	
2165	Western portion	1991	Air conditioning	Demolished
2195	Western portion	1991	Offices and storage	
D-21CSN	Eastern portion	1918	Testing building	Demolished

SAP WORKSHEET #10.2:
Problem Definition/Conceptual Site Model for Old Skeet and Trap Range (Site UXO-15)

10.2 HISTORY AND SITE DESCRIPTION

The Old Skeet and Trap Range is located in the north-central portion of Stump Neck Annex, north of Archer Avenue along the Potomac River. The eastern and western boundaries of the range consist of open fields (Map 10.2-1). The area is currently used for a helicopter pad and recreation (Figure 10.2-1). The Old Skeet and Trap Range comprises 29.33 acres and was reportedly used for small arms recreational activities. Firing may have occurred into the Potomac River. Through interviews, it was determined that the range was in use for approximately 25 years and closed in June 1991. The Old Skeet and Trap Range was identified on an April 1972 aerial photograph of Stump Neck Annex. The range does not appear on a previous aerial photograph, dated September 1967. Therefore, the Old Skeet and Trap Range was constructed between 1967 and 1972.

Figure 10.2-1: Current Conditions at the Old Skeet and Trap Range

The Old Skeet and Trap Range lies on what was initially identified as Solid Waste Management Unit (SWMU) #20, Disposal Area 2, in the Resource Conservation Recovery Act (RCRA) Corrective Action Plan (CAP), a SWMU for which no further action was recommended. However, this investigation took place while the range was still active. In 1992, the range was again added to the RCRA program as SWMU #28. At this time, the Old Skeet and Trap Range was inactive, and it was determined that lead shot still remained on the ground and in the Potomac River. The Old Skeet and Trap Range is overlapped by the firing fan from the Valley located at NSF-IH, Main Installation.

Topography

Section 10-3.2 provides a general description of topography for the Stump Neck Annex. The relief of the Old Skeet and Trap Range is relatively flat; the elevation of the entire range is approximately 16 feet above msl. Surface runoff is to the Potomac River either directly or via a drainage ditch along Archer Avenue.

Geology

Section 10-3.3 provides a description of the geology at Indian Head and the Stump Neck Annex. The Old Skeet and Trap Range is characterized by unconsolidated fluvial and marine deposits overlying older, Precambrian, igneous and metamorphic bedrock.

Soil and Vegetation Types

Section 10-3.4 provides a description of the soil and vegetation types at Indian Head and the Stump Neck Annex. According to the Soil Survey of Charles County, the soils at the Old Skeet and Trap Range consist of Mattapex fine sandy loam with 0 to 2 percent slopes. Soils in this area generally have a surface layer of sandy silt and are moderately well drained. Available moisture capacity is high, and permeability is moderately low. Vegetation at the range consists of a grass field.

Hydrology

Section 10-3.5 provides a description of hydrology at Indian Head and the Stump Neck Annex. Surface water at the Old Skeet and Trap Range drains directly into the Potomac River.

Hydrogeology

Section 10-3.6 provides a description of hydrogeology for Indian Head and the Stump Neck Annex. There is no range-specific hydrogeology information for the Old Skeet and Trap Range.

Cultural and Natural Resources

General cultural and natural resource information for the Stump Neck Annex is provided in Section 10-3.7. According to the 1996 Phase I Cultural Resources Survey of Stump Neck Annex (ref), there is one archaeological site located within the Old Skeet and Trap Range. Site 18CH388 contained a mix of prehistoric and historic artifacts. This site may correspond to Indian Head Quad File #4, a Late Woodland Indian village. A total of 645 artifacts were recovered at this site, which covers the Old Skeet and Trap Range and Marine Rifle Range.

Endangered and Special Status Species

As discussed in Section 10-3.8, endangered and special status species are reported to exist at the Stump Neck Annex. The reported endangered and special status species are expected to inhabit the Main Installation and thus have the potential to inhabit the Old Skeet and Trap Range.

10.2.2 Visual Survey Observations and Results

A visual survey of the Old Skeet and Trap Range was conducted on June 24, 2003. Personnel conducting the range visit included Mr. Dinh, Mr. Egholm, Ms. Tegtmeyer, Mr. Hains, Mr. McManus, and Ms. Morgan. The Old Skeet and Trap Range was inspected by a perimeter walk of the range (as previously identified during the inventory) and through several random transects across the range to visually inspect the entire location. The range consisted of a neatly mowed grassy area, which is now used for recreational purposes. There were no physical indications of where the firing points were located. No ordnance, MEC, or related debris were observed during the visual survey.

Facilities north of Archer Avenue consist of a recreational pavilion (Building 2174) located approximately 245 feet from the western boundary and a utility shed (Building 2012) approximately 245 feet from the eastern boundary. The former range is also currently designated as a helicopter landing pad.

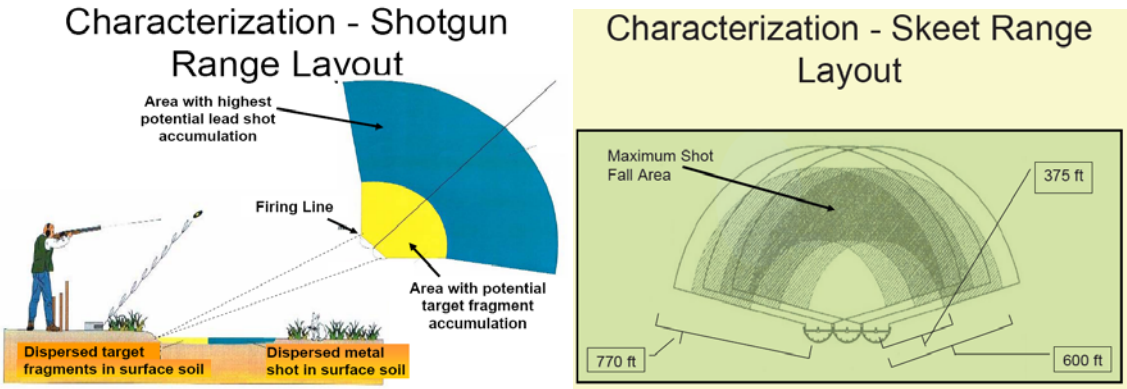
A series of site walks were conducted with the SI planning project team during the late fall and winter of 2007. An initial site walk of all of the Stump Neck Annex SI MRP sites was completed on September 18, 2007, by Ralph Basinski (Tetra Tech), Joe Rail (Navy RPM), and Shawn Jorgensen (Indian Head Environmental Department) as part of the project kick-off meeting. A subsequent site walk was conducted on November 8 to 10, 2007, and included a larger contingent of the project team: Joe Rail (Navy RPM), and Shawn Jorgensen (Indian Head Environmental Department), and several Tetra Tech personnel (Ralph Basinski, Ralph Brooks, Jim Coffman, George Latulippe, and Rick Barringer). Each MRP site was walked and multiple photographs were taken to document the site conditions observed during the site visit (Figures 10.2-2 and 10.2-3). The site walks were performed in conjunction with technical meetings at the facility that reviewed the proposed approaches for the MC fieldwork programs.

Figure 10.2-2: View of the Old Skeet and Trap Range Looking North in the General Direction of Fire Toward the Potomac River.

An expanded site walk/meeting was conducted on December 18 and 19, 2007, and included previously identified Navy and Tetra Tech personnel, as well as representatives from the MDE (Curtis Detore) and USEPA Region 3 (Dennis Orenshaw). As before, MRP sites were walked and photographed to record the conditions observed at the sites. Project quality objectives for small arms ranges were discussed among the meeting attendees on December 19, 2007.

Figure 10.2-3: View of the Old Skeet and Trap Range Looking South (Toward Archer Avenue) in the Vicinity of the Former Range Firing Locations.

As related to the Old Skeet and Trap Range, the following observations were made based on the conditions at the site. As indicated in the maps provided in the PA Report (Malcolm-Pirnie, 2005), much of the area within the boundary of the MRP site Old Skeet and Trap Range was within the Potomac River, and beyond the Stump Neck Annex installation boundary. Figures 10.2-4a and 10.2-4b present the expected distributions of range residues on the ground surface. A graded pattern is observed in skeet range residue (target fragments, shotgun shell pellets, etc.) based primarily on the mass, velocity, and general aerodynamic properties of the fired items and the resulting target fragments (Figure 10.2-4a). The zone of maximum shot fall begins approximately 375 feet from the firing point (Figure 10.2-4b).



Figures 10.2-4a and 10.2-4b: Views of Typical Skeet Ranges Showing Distribution of Range Residues and Areas of Maximum Shot Fall on the Range Surface.

During the initial SI site walk, target fragments were observed on the ground surface (Figure 10.2-5). The observed target fragments consisted of small pieces of clay targets that most likely had been hit by shotgun-fired pellets, shattered in flight, and fell to the surface in small shards.

Figure 10.2-5: Target Debris Observed on the Ground Surface at the Old Skeet and Trap Range during SI Site Walk.

The detection of these types of trap range target fragments on the ground surface near the firing point on the Old Skeet and Trap Range is consistent with the anticipated deposition point for clay target fragments as illustrated on Figure 10.2-6.

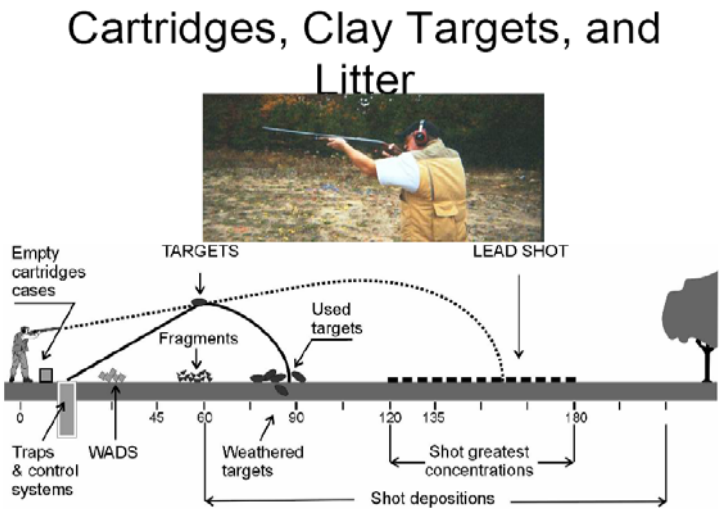


Figure 10.2-6: A Side-View of Typical Trap/Skeet Range Showing the Distribution of Range Residue (Shotgun Cartridge Wads, Target Fragments, Used Targets, and Shot Pellets) Located on the Ground Surface.

As illustrated on Figure 10.2-6, the clay pigeon targets were launched from a control system by means of a mechanical device. The target would pass across the field of view, downrange of the individual on the firing line, and the shotgun was aimed and shot to impact and break the clay target with the fired metal pellets. Expended shotgun shell pellets missing their intended targets would continue undisturbed on a standard trajectory to the north toward the Potomac River. Intact targets would land on the ground surface. Targets that were successfully hit with shot on the range would fragment and fall to the ground surface. The much denser shot pellets, after they impacted the clay targets, would continue a slightly revised trajectory and fall to the ground downrange.

Map 10.2-2 depicts the zone of maximum shot fall accumulation, which is presumed to occur in the Potomac River in an arc from the firing point.

10.2.3 Munitions and Munitions-Related Materials Associated with the Site

The data collection team was unable to locate specific records of the types and quantities of small arms ammunition used at the Old Skeet and Trap Range. However, an inventory listing from the Potomac River Gun Club for 1979 revealed the following on hand for resale: .410-, 28-, 20-, and 12-gauge shells; clay targets, 5-pound containers of bulk smokeless powder, and shot. These items would be consistent with munitions normally associated with a skeet and trap range. No evidence of ordnance was found at the range during the visual survey.

Based on the information obtained during the data collection process, the Old Skeet and Trap Range is not suspected to contain CWM-filled munitions, electrically fuzed munitions, or DU-associated munitions.

10.2.4 MEC Presence

The entire range has been subdivided and categorized into one of three levels of MEC presence including known MEC areas, suspected MEC areas, and areas where no evidence exists to indicate that MEC are known or suspected to occur. These MEC categories are discussed below. The Old Skeet and Trap Range is overlapped by the firing fan from the Valley, located at the NSF-IH, Main Installation. Thus, there is a potential for munitions associated with the Valley to be present at the Old Skeet and Trap Range. Technical data sheets can be found in Appendix D of the Final PA (Malcolm Pirnie, 2005). Only MEC presence specifically related to the Old Skeet and Trap Range was considered.

Known MEC Areas

Based on historical documents and information obtained during the data collection process, there is no evidence of MEC at the Old Skeet and Trap Range because only shotguns were used. Therefore, there are no known MEC areas associated with the Old Skeet and Trap Range.

Suspected MEC Areas

Based on historical documents and information obtained during the data collection process, there is no evidence of MEC at the Old Skeet and Trap Range because only shotguns were used. Thus, there are no suspected MEC areas at the Old Skeet and Trap Range.

Areas Not Suspected to Contain MEC

Based on historical documents and information obtained during the data collection process, there is no evidence of MEC at the Old Skeet and Trap Range because only shotguns were used. Therefore, the entire range is not suspected to contain MEC.

10.2.5 Ordnance Penetration Estimates

The depth to which munitions penetrate below the ground surface depends on many factors, including the type of soil, angle of impact, size of the munitions, velocity at impact, and range-specific environmental conditions.

For small arms ranges, the ITRC has prepared a document titled Characterization and Remediation of Soils at Closed Small Arms Firing Ranges, dated January 2003, to provide information on the general layout of small arms ranges, as well as information on areas that may be impacted with MC and/or MEC as a result of range use and the characteristics of the munitions used. According to the ITRC guidance, the penetration depth of small arms on the range floor is 1 foot or less. The document states that rounds that impact the range floor typically had a flat trajectory that fell short of or missed the target, or resulted from ricochet, and these fragments are usually found within the top 6 inches of soil. For trap and skeet ranges, the ammunition is dispersed as pellets over a small area in the direction of fire. According to the 1958 Programming Guide, the minimum safe range from a skeet/trap range is 900 feet. Pellets dispersed from a shotgun would be deposited on the ground surface and not significantly penetrate the ground unless disturbed.

10.2.6 Munitions Constituents

For shotgun ammunition and clay targets, the primary MC of concern include lead from shot and polynuclear aromatic hydrocarbons (PAHs) from pitch tar used in clay pigeons. Other associated MC less likely to be of concern may include antimony, arsenic, nickel, and lead styphnate/lead azide. These MC components are not consumed when the munitions items function as they are designed. Therefore, these MC may exist at the Old Skeet and Trap Range. NG from unburned propellants and lead from primers is suspected at the firing points.

10.2.7 Contaminant Migration Routes

Contaminants at the Old Skeet and Trap Range may potentially migrate within soil, groundwater, surface water, and sediment. Sediment contamination in Mattawoman Creek may have resulted from shooting into the direction of the creek. Although erosion on the range is considered to be minimal, contaminants may become mobile within the surface soil, particularly during extended periods of surface runoff. Runoff would occur in the direction of Mattawoman Creek. Direct human or biota contact with surficial and subsurface soil is possible if the soil is disturbed. Precipitation infiltration may provide for contaminant mobility through the subsurface to the shallow surficial groundwater aquifer, which is assumed to be connected to the nearby surface water bodies. Based on a review of hydrogeological data, it is unlikely that MC in shallow groundwater would migrate to the deeper aquifers used as a water supply. However, shallow groundwater is still considered a potential exposure medium.

10.2.8 Receptors

Potential human receptors include authorized Navy personnel (military and civilian), visitors, contractors, maintenance workers, recreational users, and trespassers. Plant and animal biota are also potential receptors. Examples of ecological receptors include deer, wild turkey, fish, and waterfowl (ducks and geese).

Buildings Near/Within Site

Currently, only five small structures are located in the vicinity of the range, both north and south of Archer Avenue. The facilities north of Archer Avenue consist of a recreational pavilion (Building 2174) located approximately 75 meters from the western boundary and a utility shed (Building 2012) approximately 75 meters from the eastern boundary. Building 2156 is south of Archer Avenue and about 75 meters from the southern boundary of the range. Buildings 2019 and 2101 are located approximately 100 and 175 meters southeast of the boundary, respectively. Both buildings are associated with the Marine Rifle Range and are not designed for a continuous dense population of personnel. The remaining area south of Archer Avenue is lightly wooded with some wetlands.

Utilities On/Near Site

There are no utilities located on the Old Skeet and Trap Range; however, above-ground electrical power lines run along Archer Avenue behind the range.

10.2.9 Land Use

The Old Skeet and Trap Range was used for small arms recreational activities. This range is currently a grass field, which is used for a helicopter pad and recreation. According to the NSF Indian head Master Plan, future land use is anticipated to continue as a grass field for a helicopter pad and for recreational activities.

10.2.10 Access Controls/Restrictions

No public access is authorized at NSF-IH, Stump Neck Annex. Signs, partial fencing, locked/secured gates, login book/office check-in, and vehicle security patrols are used to control the entire facility. There are no access control features specific to the Old Skeet and Trap Range. Access from the Potomac River is not controlled. There are no known land use/development restrictions for the range.

The Coastal Zone of Maryland includes all land and water lying within coastal counties, one of which is Charles County. Within the Coastal Zone, Maryland has defined an area within which strict land use management is needed to protect the Chesapeake Bay. The critical area is defined as a 1,000-foot-wide strip of land surrounding the bay and its tidal tributaries. Most construction within 100 feet of the Mean High Water Line (buffer) is prohibited. Most of the area of Old Skeet and Trap Range falls within the 100-foot buffer.

10.2.11 Conceptual Site Model

A general description of the CSM exposure pathway analysis is included in Section 10.2.7. For the purpose of this SAP, only MC associated with the Old Skeet and Trap Range is considered in the CSM exposure pathway analysis.

For the Old Skeet and Trap Range, historical and visual evidence indicate that MEC are not present. Therefore, no complete exposure pathways exist for MEC, and no exposure pathway analysis was completed for MEC. MC may be present; therefore, potential MC exposure pathways do exist. The MC exposure pathway analysis for the Old Skeet and Trap Range is presented on Figure 10.2-7. Soil, surface water, and sediment impacted by MC represents a primary potential source medium, as illustrated in the CSM (Map 10.2-3). Potential receptors include both human and ecological receptors that may disturb, unbury, or remove the source medium from the range. Potentially complete exposure pathways exist for surface soil through ingestion, dermal contact, and inhalation for both human and ecological receptors. Runoff, discharges, and/or erosion may transport the MC from surface soil to surface water/sediment, so potentially complete pathways also exist for all human and ecological receptors of surface water/sediment. Soil also represents an exposure medium when considering plant/animal uptake for biota (including game such as deer) and human receptors consuming the affected biota (e.g., fish and hunted game). Although hunting is not permitted on the Old Skeet and Trap Range, there are upland hunting areas nearby. Fishing is permitted in Mattawoman Creek. There is a potential for the MC present in the water to infiltrate to the subsurface soil or surficial groundwater. Potentially complete exposure pathways exist for MC in subsurface soil (direct contact, ingestion and inhalation during intrusive work activities) for all human and ecological receptors with the exception of trespassers. It is not anticipated that trespassers would come in contact with subsurface soil. Although confining layers are expected to prevent the migration of MC to the lower aquifers used for water supplies, potentially complete pathways exist for MC in shallow groundwater for human receptors.

10.2.12 Problem Definition Summary

The following is a summary of the problem definition that is detailed in Worksheet #10.2:

- Environmental contamination may exist at the Old Skeet and Trap Range (23.5 acres) because the facility was active for 25 years as a skeet and trap range. Specific small arms ammunition types and materials used at the range may have included .410-, .28-, .20-, and 12-guage shells, clay targets, 5 pound containers of smokeless powder, and shot. Based on the proximity of the site to the Potomac River, a significant amount of the shot is assumed to have fallen both in the water and along the shoreline of the river.

- MC consisting of metals (primarily lead and, to a lesser extent, antimony, arsenic, copper, tin, and zinc), NG, and PAHs (from pitch tar used in the clay pigeon targets) may be present in site soil (particularly surface soil [the 0-to 6-inch interval]) and surface water and sediment of the adjoining Potomac River as a result of military training activities. Lead is assumed to be the primary metal MC of concern in soil and sediment because it is the primary constituent of the spent munitions and because of its documented toxicity to human and ecological receptors. It is anticipated that other metals contamination will be spatially correlated with lead concentrations. MC in surface soil may have migrated to subsurface soil and subsequently to underlying groundwater. These constituents may have also migrated to adjoining surface water and sediment via surface soil run-off or the discharge of contaminated shallow groundwater to the Potomac River and direct fall out over the river. However, the nature and extent of potential environmental contamination at the site is not known at this time (no environmental sampling has been conducted to date).

- The CSM (detailed in Worksheet #10.2) indicates that potentially complete exposure pathways exist for both human and ecological receptors under both current and hypothetical future land uses. However, potential human and ecological risks have not been characterized for receptors exposed to the potentially contaminated environmental media. Unacceptable levels of human and/or ecological risk may exist.

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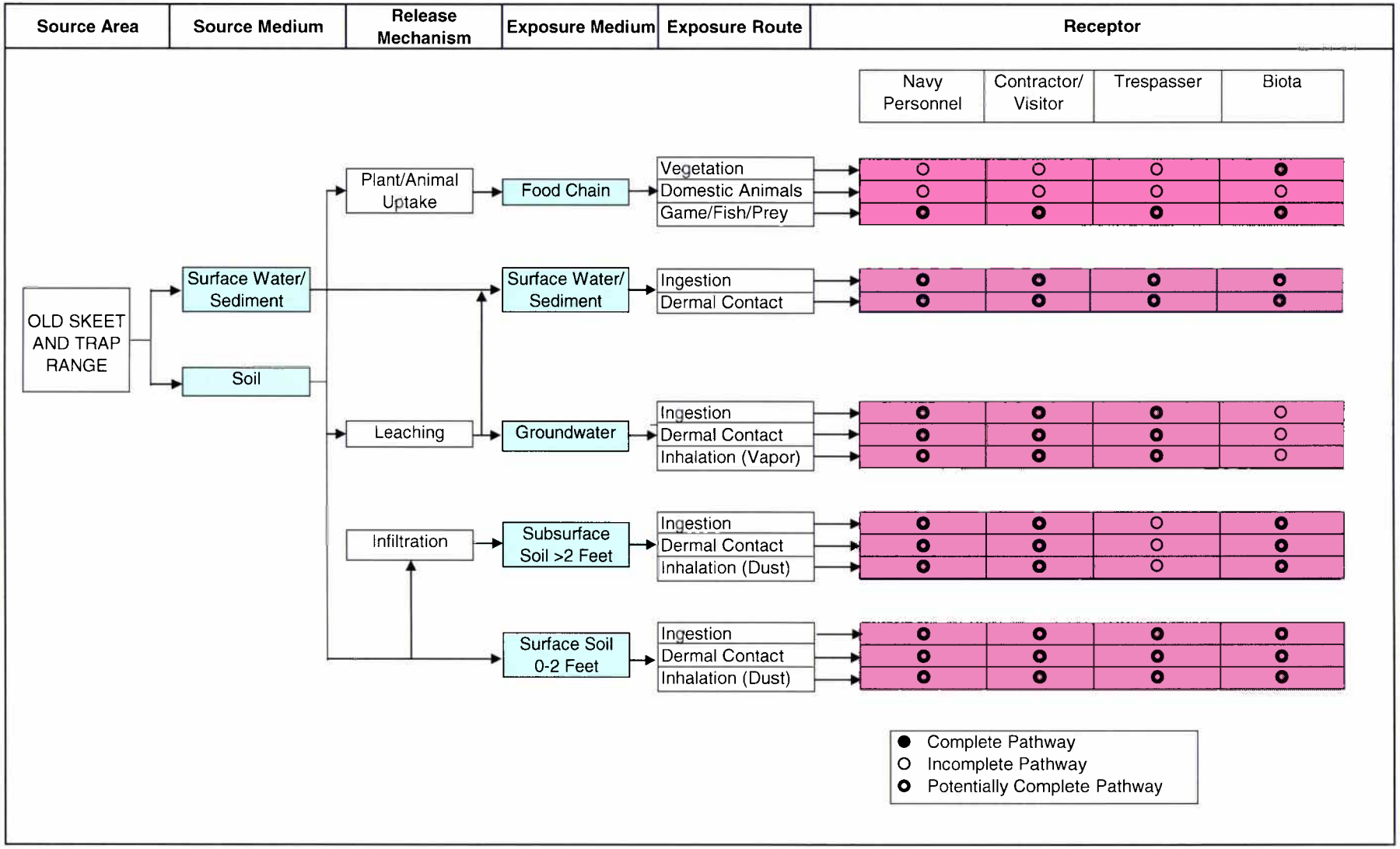
FIGURE 10.2-7

OLD SKEET AND TRAP RANGE
MC EXPOSURE PATHWAY ANALYSIS
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND



SAP WORKSHEET #10.3:
Problem Definition/Conceptual Site Model for Roach Road Rifle Range (Site UXO-25)

10.3 ROACH ROAD RIFLE RANGE

10.3.1 History and Site Description

The Roach Road Rifle Range is located in the central portion of Stump Neck Annex, immediately west of Roach Road. The 0.3-acre site was used for small arms training from approximately 1963 to 1986.

The Roach Road Rifle Range is first referenced in a 1963 memorandum from the Department of Public Works. The 1963 hand-drawn layout for the "new pistol range" includes eight firing stands, six targets constructed of railroad ties, and walkways, as shown on Figure 10.3-1. The plan indicates that the range is north of Roach Road and approximatley 830 feet east of Building 65SN (located at Area 8). According to an interview with Mr. John Bartellson, a retired EOD technician, he participated in the construction of the small arms range in the 1960s.

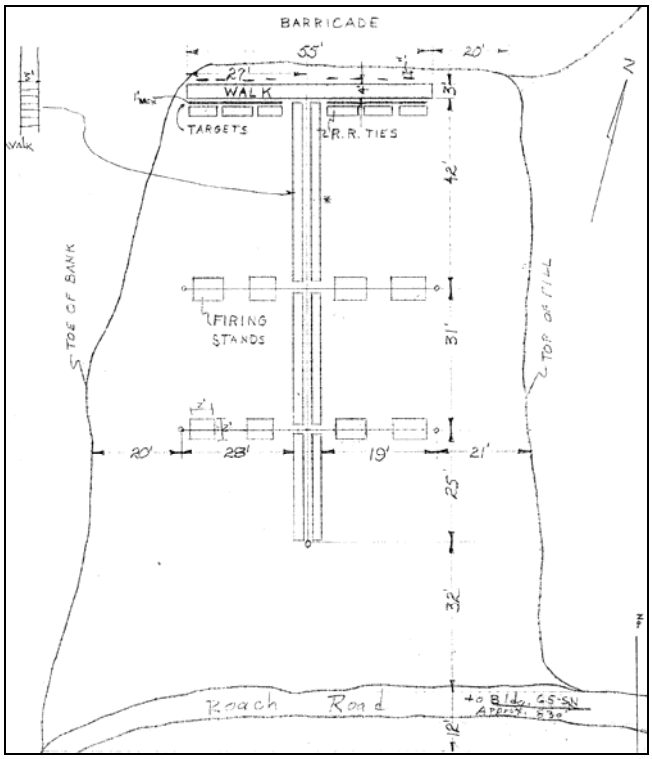


Figure 10.3-1 1963 Layout for 'New Pistol Range.'

The range is first visible on a 1967 aerial photograph as a cleared area surrounded by dense vegetation, and is still visible on a 1987 aerial photograph. The range is more distinct on a 1972 aerial photograph (Figure 10.3-2) and is shown as a cleared area immediately north of the 90 degree turn in Roach Road. On maps dating from 1967 to 1976, the range is labeled as #2070, a small bore and pistol range. On these maps, #2070 is located immediately north of the 90 degree turn in Roach Road, which corresponds to the aerial photographs from this time period. A cleared area is also visible across Roach Road on the 1972 aerial photograph (possibly a parking lot). The 1982 aerial photograph shows a southern shift in the road to the southern side of the cleared area. On maps dating from 1981 to 1988 (Figure 10.3-3), the rifle range is shown as a square area just south of the 90 degree turn in Roach Road. The 1987 aerial photograph shows vegetative cover over the range and Roach Road in its current location.

Figure 10.3-2: 1972 Aerial View of the Roach Road Rifle Range.

Due to the different locations of the range presented in the 1963 memorandum, on the maps from 1967 to 1976, and on the maps from 1981 to 1988, the location of the range was mapped for this UFP SAP using

the aerial photographs. The size of the range was established using the layout presented in the 1963 memorandum. The location of the range is provided on Map 10.3-1.

The Roach Road Rifle Range is not identified as a SWMU or IR range.

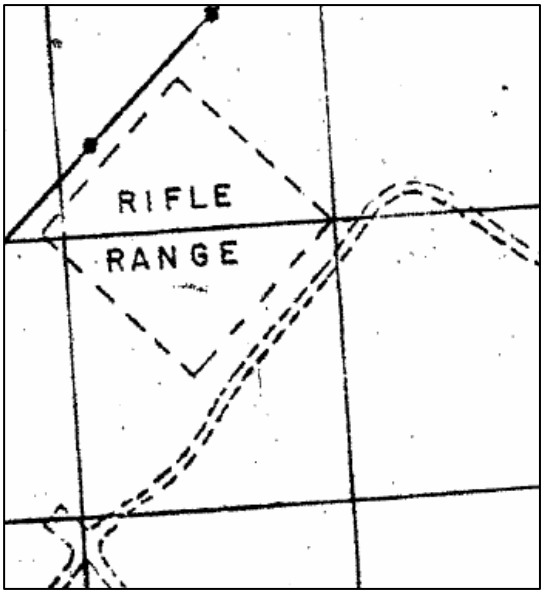


Figure 10.3-3: Range as Shown on the 1981 Map.

Topography

Section 10-3.2 provides a general description of topography for the Stump Neck Annex. The terrain at the Roach Road Rifle Range is relatively flat at an elevation of approximately 50 feet msl. The majority of the site is flat with a small downward slope toward the north. Due to the topography of the area and the predominant vegetative ground cover, the potential for erosion is limited.

Geology

Section 10-3.3 provides a description of the geology the Stump Neck Annex, which is applicable to the Roach Road Rifle Range.

Soil and Vegetation Types

Section 10-3.4 provides a description of the soil and vegetation types at the Stump Neck Annex. Vegetation at the Roach Road Rifle Range is a mixture of shrubs and forest as illustrated on Figure 10.3-4. According to the Soil Survey of Charles County, soils in the area consist of silty sand with gravel at the ground surface underlain by silty sand with clay. Specifically, Aura gravelly sandy loam is present at the range.

Figure 10.3-4: Vegetation at the Roach Road Rifle Range.

Hydrology

Section 10-3.5 provides a description of hydrology at the Stump Neck Annex. There are no surface water bodies present at the Roach Road Rifle Range. Based on topography, surface water flows to the north toward Chicamuxen Creek, which drains to the Potomac River.

Hydrogeology

Section 10-3.6 provides a description of hydrogeology for the Stump Neck Annex, which is applicable to the area of the Roach Road Rifle Range. It is assumed that shallow groundwater flow from the Roach Road Rifle Range follows topography and is connected to the area's dominant surface water bodies (Mattawoman Creek, Chicamuxen Creek, and the Potomac River). No monitoring wells are located within the Roach Road Rifle Range.

Cultural and Natural Resources

General cultural and natural resource information for Stump Neck Annex is provided in Section 10.3.7. Because a portion of this range is undeveloped, there is the potential for wildlife to exist at the range. According to the 2003-2004 Stump Neck Annex Hunting Map, hunting is permitted in the undeveloped portions of the Roach Road Rifle Range. The Chicamuxen WMA is located approximately 500 feet north of the Roach Road Rifle Range and is located within an eagle protection area.

The Phase I Cultural Resources Survey of Stump Neck Annex and Supplemental Architectural Investigations from August 1996 did not identify archeological sites within the Roach Road Rifle Range. Historic and prehistoric artifacts were recovered but not in sufficient concentrations to warrant a site designation. The locations of positive prehistoric and historic findings are identified on Figure 10.3-5. No sites were identified within the range Maryland Register of Historic Places (State Register) or the National Register of Historic Places.

Endangered and Special Status Species

Endangered or special status species located on the Stump Neck Annex are presented in Section 10-3.8. These species potentially inhabit the Roach Road Rifle Range.

10.3.2 Visual Survey Observations and Results

A visual survey of the Roach Road Rifle Range was conducted during a range visit on June 2, 2004. Malcolm Pirnie personnel walked the area north of Roach Road, which historically was the cleared area across from the range (possibly a parking lot). Because the location of the Roach Road Rifle Range

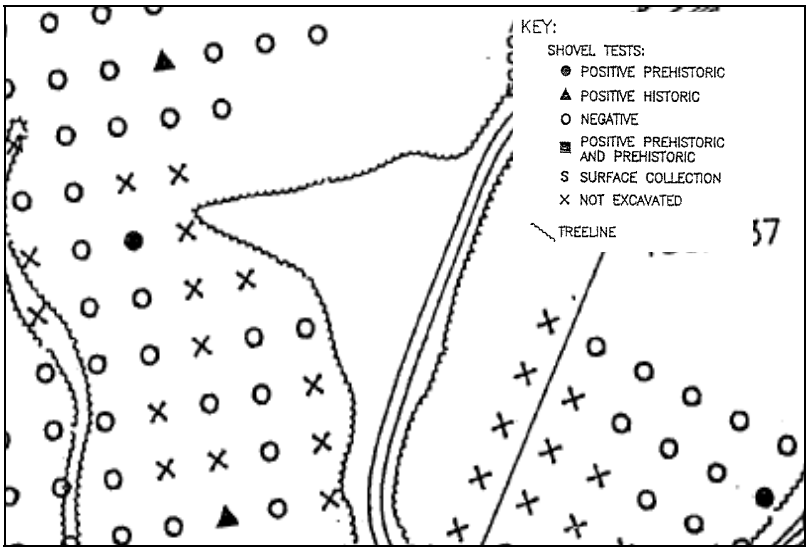


Figure 10.3-5: Cultural Resources Identified in Shovel Test Pits.

was determined by aerial photograph analysis after this site visit, the majority of the range was not inspected in the field. Thus, the presence or absence of former berms cannot be confirmed at that time. A wooded area with similar shrubs and trees was observed that appeared to have been cleared approximately 25 years ago. Larger trees and pines surrounded this area. The perimeter of this area was visually inspected; however, the survey was limited because of safety issues associated with swarms of yellow jackets in the area. No ordnance, MEC, or related debris were observed during the range visit.

Figure 10.3-6 Construction Debris Identified During the Site Visit.

The area inspected is currently used for storage of construction-related debris, which was observed during the visual survey. Construction debris/rubble including old concrete pipes and telephone poles was observed in the central portion of the range immediately north of Roach Road (Figure 10.3-6). Piles of dirt and gravel were also observed throughout the site. The central portion of the site is cleared and covered with gravel and dirt.

A series of site walks were conducted with the SI planning project team during the late fall and winter of 2007. An initial site walk of all of the Stump Neck Annex SI MRP sites was completed on September 18, 2007, by Ralph Basinski (Tetra Tech), Joe Rail (Navy RPM), and Shawn Jorgensen (Indian Head Environmental Department) as part of the project kick-off meeting. A subsequent site walk was conducted on November 8 to 10, 2007, and included a larger contingent of the project team: Joe Rail (Navy RPM), Shawn Jorgensen (Indian Head Environmental Department), and several Tetra Tech personnel (Ralph Basinski, Ralph Brooks, Jim Coffman, George Latulippe, and Rick Barringer). Each MRP site was walked and multiple photographs were taken to document conditions observed during the site walks (Figures 10.3-7 through 10.3-10). The site walks were performed in conjunction with technical meetings at the facility that reviewed the proposed approaches for the MC fieldwork programs.

Figure 10.3-7: General View of Vegetation Conditions Near the Roach Road Rifle Range as Observed During SI Site Walk.

Figure 10.3-8: Earthen Hillside Slope Backing Former Target Butt Location on the Roach Road Rifle Range as Observed During SI Site Walk.

An expanded site walk/meeting was conducted on December 18 and 19, 2007, and included previously identified Navy and Tetra Tech personnel, as well as representatives from the MDE (Curtis Detore) and USEPA Region 3 (Dennis Orenshaw). As before, MRP sites were walked and photographed to record the conditions observed at the sites. Project quality objectives for small arms ranges were conducted with the meeting attendees during December 19, 2007.

During the SI site walks, it became apparent that, since closure of the range in 1986 there had been significant placement of fill materials in the areas near present-day Roach Road. The piles of fill materials and the staging of construction materials, telephone poles, piping, and construction debris (e.g., corrugated metal pipe, etc.) in the general area of the Roach Road Rifle Range added to the difficultly in locating any remaining features of the former range. There was evidence of potential small arms usage at the Roach Road Rifle Range, which may have occurred after the 1986 closure of the range. As shown on Figures 10.3-9 and 10.3-10, small arms fire was directed at a coffee pot, and shot guns may also have

been used at the Roach Road Rifle Range (as evidenced by the presence of shot gun shells on the ground surface).

Figure 10.3-9: Evidence of Small Arms Usage at the Roach Road Rifle Range Observed During SI Site Walk.

Figure 10.3-10: Discarded Shotgun Shells on the Ground Surface at the Roach Road Rifle Range as Observed During SI Site Walk.

As related to the Roach Road Rifle Range, the following additional observations were made based on the conditions at the site. The PA Report relied on a sequence of historical aerial photographs and identified that the physical location of Roach Road in the immediate vicinity of the small arms range had shifted prior to 1982. The hazards from swarming yellow jackets prevented the PA project team from performing a detailed site walk for the Roach Road Rifle Range. The MRP site was mapped by the PA project team based on aerial photographic information, without the benefit of walking the site. However, during the SI site walks, it became apparent that the boundaries of former Roach Road Rifle Range needed to be redefined. Trees located in the mapped downrange area were large and appeared to be too mature to have grown during the 20-plus years since the end of range operations at this location in 1986. Furthermore, the mapped range area was located across a hillside slope and into a valley from Roach Road. Small arms ranges are generally level and have some sort of impact berm or hillside slope to collect and contain the expended small arms ammunition. There was no berm or hillside present in the mapped range position as presented in the PA, so the general range location seemed out of place with the present-day topography.

A review of the 1963 hand sketch (Figure 10.3-1) indicated range dimensions and the presence of a barricade behind the targets and specific notes on the left (toe of bank) and right (top of fill) perimeters of the range. Using that site-specific information, the range location was modified so that the hand-sketched range configuration was consistent with local topographic conditions. Map 10.3-1 shows the location of the Roach Road Rifle Range as originally indicated in the PA and as subsequently revised for the SI. The revised location for the Roach Road Rifle Range provides an improved match with the range layout as indicated in the 1963 hand sketch and provides a refined site location that is more consistent with conventional range design and range siting requirements.

The refined range location aids in establishing the site boundaries for the Roach Road Rifle Range and in identifying the barricade or bullet backstop where ammunition expended on the range would have accumulated. The topography of the hillside behind the Roach Road Rifle Range appears to have been contoured to accommodate the range, and the excavated earthen materials were most likely used to level the range surface and as a source of fill for the right side of the range (as viewed from Roach Road). Expended bullets passing through the targets or passing above the targets would have continued on a trajectory into the hillside (barricade) behind the range targets.

The updated Roach Road Rifle Range boundary proposed for the SI now includes a downrange hillside on the western side of Roach Road that would most likely have received ammunition fired on that range.

As shown on Figure 10.3-11, the downrange earthen hillside (barricade) directly behind the Roach Road Rifle Range targets would be expected to contain concentrated metal from the expended bullets.

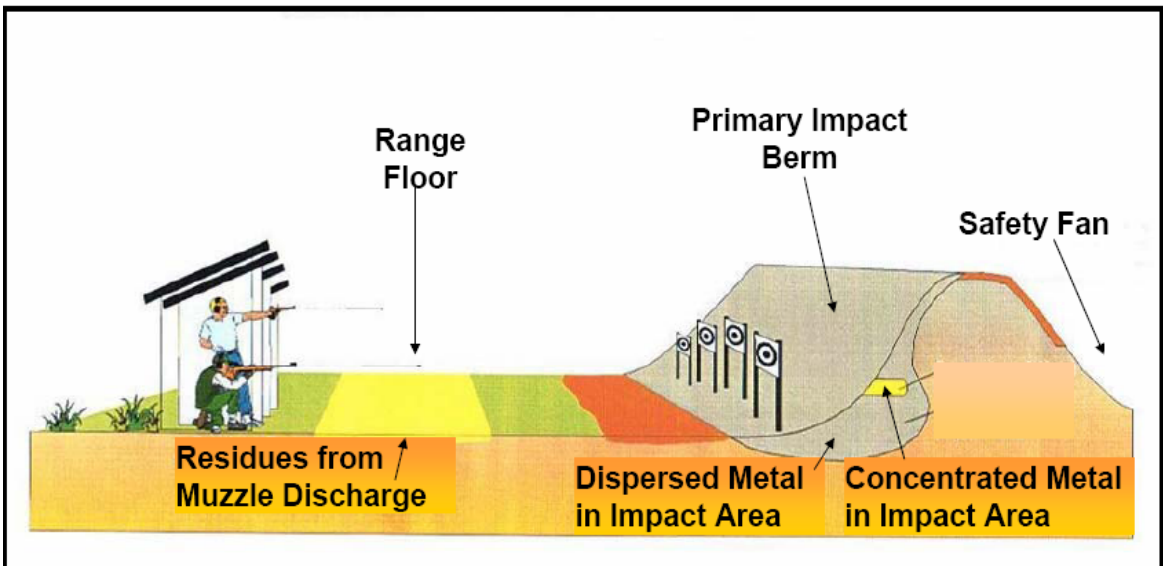


Figure 10.3-11: The Typical Distribution of Residue and Expended Ammunition on a Small Arms Training Range is Similar to That Expected on the Ground Surface and Within the Barricade Soil at the Roach Road Rifle Range.

Based on these observations, there should be a general area for the accumulation of expended small arms ammunition on the Roach Road Rifle Range. Those bullets aimed below the range targets would tend to accumulate in the target butt soil below the targets. Those fired bullets that successfully hit the targets most likely continued on a trajectory that would continue through the targets and into the hillside barricade (impact berm). Range fire that overshot the targets would impact the soil above and behind the targets, but most likely within the surficial soil of the impact berm. The areas to be inspected and assessed during the SI are shown on Figure 17-3 in Worksheet 17.

10.3.3 Munitions and Munitions-Related Materials Associated with the Site

This section describes the munitions or munitions-related materials known or suspected to be at the range. This includes both MEC and non-hazardous munitions-related debris (e.g., fragmentation, base plates, inert mortar fins).

The Roach Road Rifle Range was used for small arms training and historical maps and documents state that rifles and pistols were used at the range. Mr. Gordon Miller, a former EOD employee of NSF-IH, also stated that pistols were used at the range. Specific ordnance types used at the range are not known.

Lead bullets/fragments are suspected within the target butts. NG from unburned propellants and lead from primers is suspected at the firing points. No munitions-related debris items were identified at the Roach Road Rifle Range. No bullets or metallic debris were identified in the shovel tests conducted for the Phase I Cultural Resources Survey of Stump Neck Annex and Supplemental Architectural Investigations (reference).

Based on the information obtained during the data collection process, the Roach Road Rifle Range is not suspected to contain CWM-filled munitions, electrically fuzed munitions, or DU1associated munitions.

10.3.4 MEC Presence

The entire range has been subdivided and categorized into one of three levels of MEC presence including known MEC areas, suspected MEC areas, and areas where no evidence exists to indicate that MEC are known or suspected to occur. These MEC categories are discussed below.

Known MEC Areas

There are no readily apparent MEC areas associated with the Roach Road Rifle Range because small arms do not pose an explosive hazard.

Suspected MEC Areas

There are no suspected MEC areas expected at this range because small arms do not pose an explosive hazard.

Areas Not Suspected to Contain MEC

Small arms are not MEC; therefore, there are no areas at the Roach Road Rifle Range suspected to contain MEC.

10.3.5 Ordnance Penetration Estimates

For small arms ranges, the ITRC has prepared a document titled, “Characterization and Remediation of Soils at Closed Small Arms Firing Ranges”, dated January 2003, to provide information on the general layout of small arms ranges, as well as information on areas that may be impacted with MC and/or MEC as a result of range use and the characteristics of the munitions used. According to the January 2003 ITRC guidance document titled "Characterization and Remediation of Soils at Closed Small Arms Firing

Ranges", the penetration depth of small arms on the range floor is 1 foot or less. The document states that rounds that impact the range floor typically had a flat trajectory that fell short of or missed the target, or resulted from ricochet, and these fragments are usually found within the top 6 inches of soil. MC at the RRR Range is expected to be concentrated in the target butts.

10.3.6 Munitions Constituents

For small arms ammunition, the primary MC of concern is lead from shot. Other associated MC less likely to be of concern may include antimony, arsenic, copper, nickel, and lead styphnate/lead azide. These MC are not consumed when the munitions items function as they are designed. Therefore, these MC may exist at the Roach Road Rifle Range. NG from unburned propellants and lead from primers is suspected at the firing points. There was no record found of environmental sampling at the range.

10.3.7 Contaminant Migration Routes

Contaminants at the Roach Road Rifle Range may potentially migrate within soil, groundwater, surface water and sediment. The Roach Road Rifle Range's proximity to Chicamuxen Creek and its unnamed tributaries provides possible migration routes to surface water. Storm water discharges to surface water via overland flow. Groundwater in the shallow water table aquifer also likely flows towards Chicamuxen Creek; therefore, MC leaching from soil into shallow groundwater may migrate to surface water. Sediments can act as contaminant repositories, and sediment mixing and dredging can act as migration routes to surface water. MC in surface soil and sediment may migrate via plant/animal uptake. Direct human or biota contact with surficial and subsurface soil is possible if the soil is disturbed. Based on a review of hydrogeological data, it is unlikely that MC in shallow groundwater would migrate to the deeper aquifers used as water supplies. However, shallow groundwater is still considered a potential exposure medium.

10.3.8 Receptors

Potential human receptors include authorized Navy personnel (military and civilian), visitors, contractors, maintenance workers, recreational users, and trespassers. Plant and animal biota are also potential receptors. Examples of ecological receptors include deer, wild turkey, and waterfowl.

Buildings Near/Within Site

There are no buildings currently located at the Roach Road Rifle Range. Firing stands, targets, and walkways were present at the site when it was active. The firing stands may be the ones observed at the

former Small Arms (pistol) Range on Rum Point Road during the SI site walk and are currently used for storage purposes.

Utilities On/Near Site

There are no electrical, telephone, sanitary sewer, or potable water lines at the Roach Road Rifle Range.

10.3.9 Land Use

There is no readily available information on land use prior to 1963. From 1963 until 1986, the range was maintained as a rifle range. The site is currently undeveloped and is adjacent to an area used for storage of dirt, gravel, telephone poles, and other road construction supplies. According to installation personnel, there are no planned changes to activities at the Roach Road Rifle Range. According to the 2003-2004 Stump Neck Annex Hunting Map, the Roach Road Rifle Range is located within an upland hunting area.

10.3.10 Access Controls/Restrictions

No public access is authorized at NSF-IH. Signs, partial fencing, locked/secured gates, login book/office check-in, and vehicle security patrols are used to control the entire facility. There are no access control features specific to the Roach Road Rifle Range. According to the 2003-2004 Stump Neck Annex Hunting Map, hunting is permitted within the Roach Road Rifle Range.

10.3.11 Conceptual Site Model

A general description of the CSM exposure pathway analysis is included in Section 10.3.7. For the purpose of this SAP, only MC associated with the Roach Road Rifle Range is considered in the CSM exposure pathway analysis.

For the Roach Road Rifle Range, historical and visual evidence indicate that MEC are not present. Therefore, no complete exposure pathways exist for MEC and no exposure pathway analysis was completed for MEC. Soil impacted by MC represents a primary potential source medium, as illustrated in the CSM (Map 10.3-2). Potential receptors include both human (Navy personnel, contractor/visitors, and trespassers) and ecological receptors (biota) that may contact MC in soil. A potentially complete exposure pathway exists for surface soil through ingestion, inhalation, and dermal contact for both human and ecological receptors. Soil also represents an exposure medium when considering plant/animal uptake for biota (including game such as deer and wild turkey) and human receptors consuming the affected biota (e.g., hunted game). Precipitation infiltration may provide for contaminant mobility into

subsurface soil and into the shallow or surficial groundwater aquifer, which is assumed to be connected to nearby surface water bodies. Subsurface soil represents an exposure medium when considering potential future construction or ground disturbances by Navy personnel, contractors/visitors, and biota. Runoff/erosion impacting surface water/sediment also presents a potential exposure medium to human receptors and biota. Although confining layers are expected to prevent the migration of MC to the lower aquifers used for water supplies, potentially complete pathways exist for MC in shallow groundwater for human receptors. The MC exposure pathway analysis for the Roach Road Rifle Range is presented on Figure 10.3-12.

10.3.12 Problem Definition Summary

The following is a summary of the problem definition which is detailed in Worksheet #10.3:

- Environmental contamination may exist at the Roach Road Rifle Range (0.3 acre) because the facility was an active range from 1963 through 1986. Specific small arms ammunition types and materials used at the Roach Rifle Range most likely included small arms and pistol ammunition (9mm, .45-cal, and .50-cal). Expended bullets are suspected along the range floor and in the earthen embankment to the west behind the former range targets (i.e., the primary impact berm).

- MC consisting of metals (primarily lead and, to a lesser extent, antimony, arsenic, copper, tin, and zinc) and NG may be present in site soil (particularly surface soil [the 0- to 6-inch interval]) and surface water and sediment of drainageways leading from the range to the adjoining unnamed tributaries, wetlands, and Chicamuxen Creek. Lead is assumed to be the primary metal MC of concern because it is the primary constituent in the spent munitions and because of its documented toxicity to both human and ecological receptors. It is anticipated that other metals contamination will be spatially correlated with lead concentrations. MC, if present in surface soil, may have migrated to subsurface soil and subsequently infiltrated to underlying groundwater. These constituents may have also migrated to the aforementioned adjoining surface water bodies via surface soil runoff or discharge of contaminated shallow groundwater to surface water. However, the nature and extent of the potential environmental contamination at the Roach Road rifle Range is not known at this time (no environmental sampling has been conducted to date).

- The CSM (detailed in Worksheet #10.3) indicates that potentially complete exposure pathways exist for both human and ecological receptors under both current and hypothetical future land uses. However, potential human and ecological risks have not been characterized for receptors exposed to

the potentially contaminated environmental media. Unacceptable levels of human and/or ecological risk may exist.

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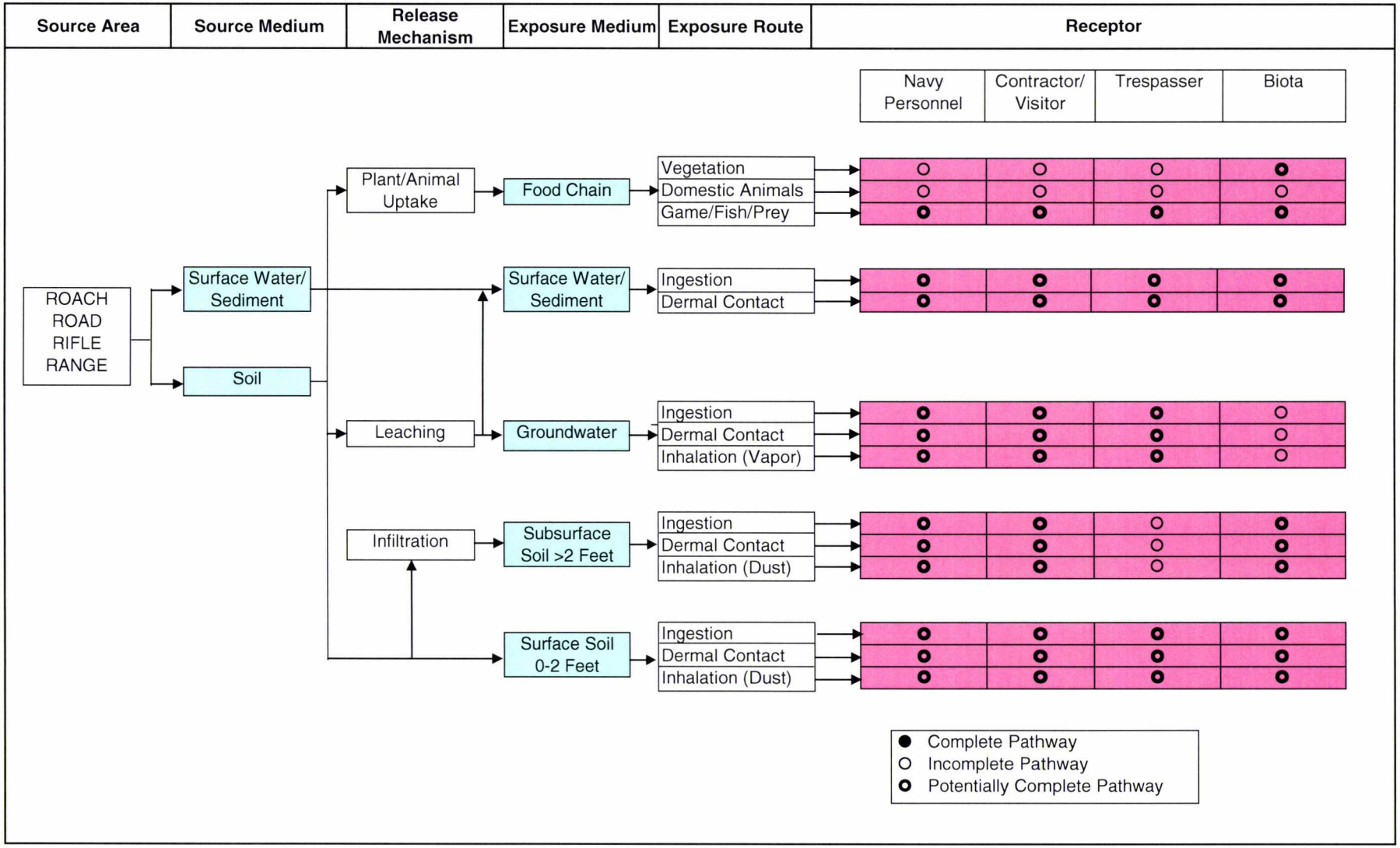
FIGURE 10.3-12

ROACH ROAD RIFLE RANGE
MC EXPOSURE PATHWAY ANALYSIS
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND



SAP WORKSHEET #10.4:
Problem Definition/Conceptual Site Model for Rum Point Skeet Range (Site UXO-16)

10.4 RUM POINT SKEET RANGE

10.4.1 History and Site Description

The Rum Point Skeet Range was constructed and became operational in 1991 as part of the Morale, Welfare, and Recreation program. The area has two skeet ranges side by side and separated by a wooden fence (Map 10.4-1). A 30-foot by 60-foot modular administrative building was constructed in the observation area directly south of the shooting points. An August 1990 range plan drawing outlines the construction plans for the skeet and trap houses, concrete pads and walkways, new access road and parking lot, and new clearing limits. According to installation personnel, this range was used exclusively as a recreational facility. The Potomac River Gun Club used and maintained the range as a private combination skeet, trap and three-dimensional archery range. During this time, the Administration Building (#2153) became the gun club's headquarters. The skeet range was closed in 2001.

The former Rum Point Skeet Range covers approximately 33.45 acres in the northeastern section of the Stump Neck Annex. This acreage covers a 900-foot arc from the firing points on the southern end of the range to the northern edge of the range. This distance complies with the skeet range safety requirements as stated in the 1958 U.S. Naval Aeronautical Shore Facilities Programming Guide. According to the 1999 Environmental Management Plan produced by the Potomac River Gun Club (ref), an estimated 85,000 targets were thrown per year for practice and competition shooting. Only shotguns were used at the range. Approximately 5,300 pounds of lead were deposited at the skeet range each year. From this estimation, roughly 53,000 pounds of lead shot fell within the shot fall zone at the range during its 10 years of operation.

Topography

Section 10-3.2 provides a general description of topography for the Stump Neck Annex. The topography of the former Rum Point Skeet Range is gently sloping from southeast to northwest. Over 94 percent of the range is located on upland terrain. The cleared area of the range is fairly flat. West of the cleared area, the land has a gradual downward slope toward the wetland area and beyond. Approximately 90 percent of the Rum Point Skeet Range is located at or above an elevation of 30 feet above msl. Based on the topography of the area and the complete vegetative ground cover, severe erosion is not considered to be a problem.

Geology

Section 10-3.3 provides a description of the geology of Indian Head and the Stump Neck Annex. There is no range-specific geologic information for the Rum Point Skeet Range because no wells are located within the boundaries and no sampling has been performed.

Soil and Vegetation Types

Section 10-3.4 provides a description of the soil and vegetation types at Indian Head and the Stump Neck Annex. According to the Soil Survey of Charles County, the surface soil at the Rum Point Skeet Range consists of a thick series of silty clays. The subsurface soil is a combination of Sassafras sandy loams with 0 to 5 percent slopes and moderate erodability. These soils form on upland terrains that are nearly level to moderately sloping. They are well-drained soils with moderate permeability. During construction of the range in 1991, activities affecting the soil included earth movement and excavation to level the firing pad area. Grasses and shrubs have now taken root directly within the disturbed surface soil, thus limiting possible erosion. The vegetation on the range is characterized by open fields with maintained grass surrounded by hardwood forests and wetlands. High grasses of the family Graminea and genus festuca, such as Kentucky Fescue 31, were planted by the Potomac River Gun Club within specific zones to dissuade birds and waterfowl from grazing or nesting on the range. There is a 100-yard transition zone between the grasses and the tree line containing a mix of high scrubs, bushes, and tall grasses. The trees are approximately 50 to 75 feet tall. The wetland area is classified by the installation as Palustrine forested broad-leaved deciduous wetland. Figure 10.4-1 illustrates the vegetation on the range.

Figure 10.4-1: Grass Surrounding the Concrete Firing Pad and Bordering Hardwood Forest.

Hydrology

Section 10-3.5 provides a description of hydrology at Indian Head and the Stump Neck Annex. There are two small unnamed tributaries of the Rum Point Skeet Range and a wetland area on the western portion of the range. Surface water runoff occurs in the direction of the wetlands. The Potomac River Gun Club planted tall Fescue grass along the shot fall zone to minimize overland flow. The range was also graded

so that surface runoff is directed away from the shot fall zone to the western edge of the range. The wetlands and tributaries eventually drain into Mattawoman Creek.

Hydrogeology

Section 10-3.6 provides a description of hydrogeology for Indian Head and the Stump Neck Annex. There are no wells located on the Rum Point Skeet Range; however, there is a monitoring well adjacent to the southern side of the range. The well is not used for drinking water, and there are no available data. The closest potable well to the Rum Point Skeet Range is a private well located on Rum Point, approximately 1,200 feet north (downgradient) of the range along Rum Point Road. The well is considered to be private due to the low population density. This well is tested quarterly or monthly if used. The well was installed in 1978 at an elevation of 4 feet above msl with a screened interval of 231 to 246 feet below msl, within the lower Patapsco aquifer, and has an estimated flow rate of 15 gallons per minute. The static water level is 18 feet below msl.

Cultural and Natural Resources

General cultural and natural resource information for the Stump Neck Annex is provided in Section 10.3.7. The range is located in a species protected area and contains a wetland area on the western side. The 1996 Phase I Cultural Resources Survey and Supplemental Architectural Investigations identified three sites that partially overlap the Rum Point Skeet Range.

Site 18CH391: This site was previously identified in a 1966 historic survey. Artifacts recovered from this site were from the Late Archaic through Late Woodland periods. The size and nature of the site suggest that it was once a base camp or village. Many of the prehistoric materials were from the Ap horizon. It was recommended for a Phase II evaluation. The site overlaps the northeastern portion of the Rum Point Skeet Range, and its location is provided on Figure 10.4-2.

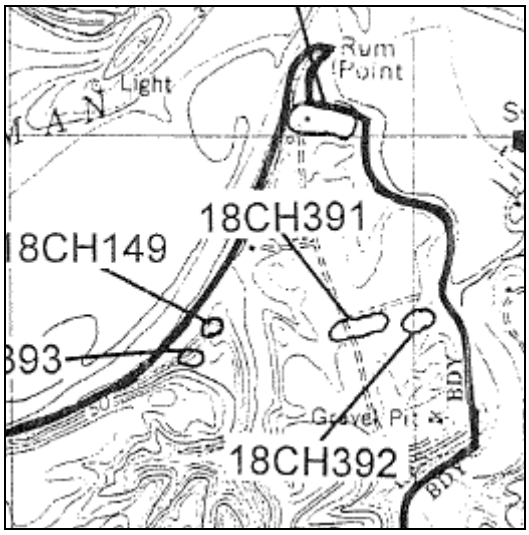


Figure 10.4-2: Location of Site 18CH391.

Site 18CH628: This small Late Woodland site contained a wide variety of artifact classes including debitage, flake tools, ceramics, and fire-cracked rock. The artifacts provide evidence that the site was a resource procurement site or a briefly occupied camp. Cores and primary debitage were located in the southeastern portion of the site. Ceramic artifacts were located in the northwestern portion of the site. Recovered historic artifacts were most likely from field scatter. The site was recommended for a Phase II evaluation. This site overlaps the eastern side of the Rum Point Skeet Range (Figure 10.4-3).

Site 18CH630: A diverse collection of prehistoric artifacts including debitage, a flake tool, a core, ceramics, and fire-cracked rock fragments were recovered at this small Late Woodland site. The artifacts were uncovered in unplowed Sassafrass Silt Loam soils. A Phase II evaluation of the site was recommended. The entire site is overlain by the northwestern section of the Rum Point Skeet Range (Figure 10.4-3).

Endangered and Special Status Species

As discussed in Section 10.3.8, endangered and special status species are reported to exist at the Stump Neck Annex, but none are known to inhabit the Rum Point Skeet Range. The former Rum Point Skeet Range is located within an eagle protection area and in a designated species protection area.

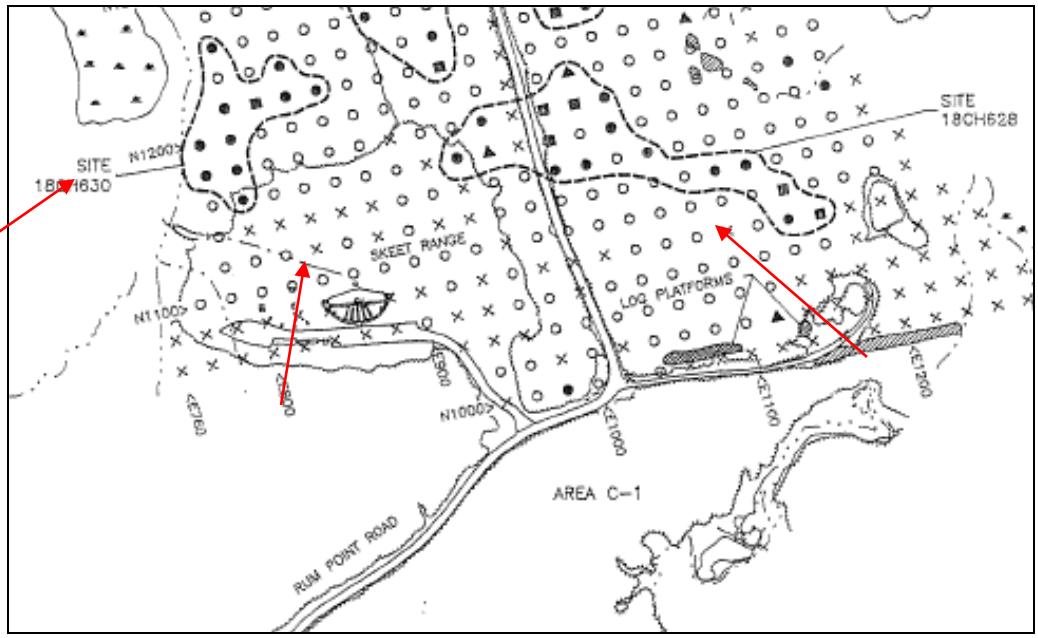


Figure 10.4-3: Location of the Rum Point Skeet Range and Sites 18CH628 and 18CH630.

10.4.2 Visual Survey Observations and Results

Figure 10.4-4: Concrete Pad and Firing Lines Visible on the Southern Edge of the Range

A visual survey of the Rum Point Skeet Range was conducted on June 25, 2003. Malcolm Pirnie personnel, along with NSF-IH Environmental Office personnel, walked the tree line surrounding the range and several transects through the middle of the range. The range is semicircular in shape and accessed from the south at the end of Skeet Range Way. A wooden fence divides the range from the roadway and the small parking area, which are lined with streetlights. Building 2153RP is located on the southern side of Skeet Range Way. The building appeared to be in working condition and in use. The range boundaries overlap Rum Point Road and a small area of land directly east of the road. This road and the cleared portion of the range are separated by forest. The western portion of the range is transected by wetlands. The range appears to be well maintained, and the grass is mowed. The cleared area of the range is approximately 6 acres. The semicircular concrete pads and firing lines for both ranges are still

clearly visible on the range, as illustrated on Figure 10.4-4. There are five concrete walkways on each range. The target houses for the ranges remain. Each range has its own trap house and its own low or high house and shares the one combination high/low house located in the center of the two ranges. There is a 34-foot-long, 8-foot-high wooden protection fence behind the combination house that separates the two ranges. Firing occurred from the concrete pads towards the northern edge of the range.

Clay targets (White AA flyers) were found along the northeastern tree line of the range; an example is shown on Figure 10.4-5. Storage boxes/trailers labeled C45, C12, C13, and C44 were located at the end of Skeet Range Way along the southwestern range perimeter. Four empty 55-gallon steel drums were found next to the southwestern side of the range. One empty shotgun shell casing was identified next to the drums.

Figure 10.4-5: Clay Target (White AA Flyer) Found at the Rum Point Skeet Range.

A series of site walks were conducted with the SI planning project team during the late fall and winter of 2007. An initial site walk of all of the Stump Neck Annex SI MRP sites was completed on September 18, 2007, by Ralph Basinski (Tetra Tech), Joe Rail (Navy RPM), and Shawn Jorgensen (Indian Head Environmental Department) as part of the project kick-off meeting. A subsequent site walk was conducted on November 8 to 10, 2007, and included a larger contingent of the project team: Joe Rail (Navy RPM), Shawn Jorgensen (Indian Head Environmental Department), and several Tetra Tech personnel (Ralph Basinski, Ralph Brooks, Jim Coffman, George Latulippe, and Rick Barringer). Each MRP site was walked, and multiple photographs were taken to document the site conditions observed during the site visit (Figures 10.4-6, 10.4-7, 10.4-8, 10.4-10, 10.4-11, and 10.4-13). The site walks were

performed in conjunction with technical meetings at the facility that reviewed the proposed approaches for the MC fieldwork programs.

Figure 10.4-6: View of the Rum Point Skeet Range Looking South to General Firing Line Area.

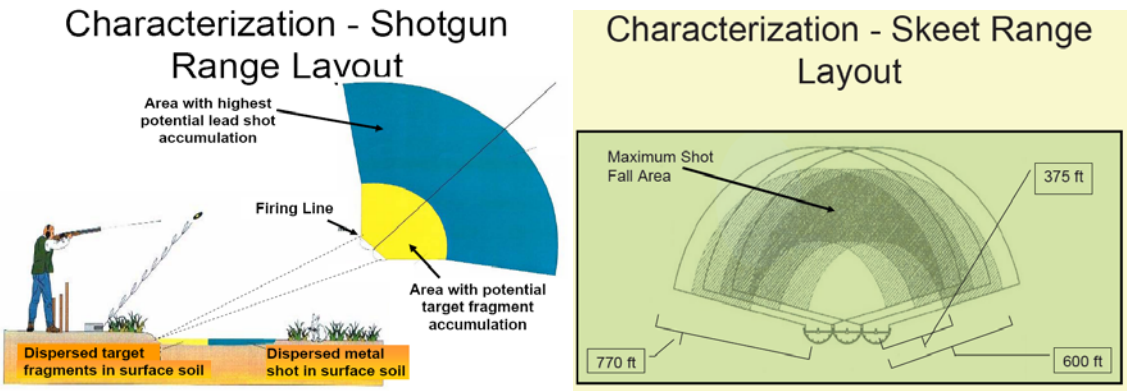
An expanded site walk/meeting was conducted on December 18 and 19, 2007, and included previously identified Navy and Tetra Tech personnel, as well as representatives from the MDE (Curtis Detore) and USEPA Region 3 (Dennis Orenshaw). As before, MRP sites were walked and photographed to record the conditions observed on the sites. Project quality objectives for small arms ranges were discussed with the meeting attendees during December 19, 2007.

Figure 10.4-7: View of the Rum Point Skeet Trap Range Looking East in the Vicinity of the Former Range Skeet Houses and Firing Locations.

Figure 10.4-8: View of the Rum Point Skeet Range in the Vicinity of the Former Range Firing Locations.

As related to the Rum Point Skeet Range, the following observations were made based on the conditions at the site. As indicated in the maps provided in the PA Report (Malcolm Pirnie, 2005), much of the area within the boundary of the Rum Point Skeet Range drains to the west via a shallow surface depression. This surface depression receives surface water runoff from the range then conveys the water and sediment into an unnamed tributary and wetland that discharges into Mattawoman Creek. Dissolved metals and/or soil/sediment particulates are most likely transported within this drainageway during precipitation events to Mattawoman Creek and may eventually discharge into the nearby Potomac River and beyond the Stump Neck Annex installation boundary. For purposes of the SI, the MRP site boundaries for the Rum Point Skeet Range address only the areas within the Stump Neck Annex installation boundary.

Figures 10.4-9a and 10.4-9b present the expected distributions of range residues on the ground surface at the Rum Point Skeet Range. A graded pattern is observed in skeet range residue (target fragments, shotgun shell pellets, etc.) based primarily on the mass, velocity, and general aerodynamic properties of the fired items and the resulting target fragments (Figure 10.4-9a). The zone of maximum shot fall begins approximately 375 feet from the firing point (Figure 10.4-9b).



Figures 10.4-9a and 10.4-9b: Views of typical skeet ranges showing distribution of range residues and areas of maximum shot fall on the range surface.

During the SI site walk, target fragments were observed on the ground surface (Figures 10.4-10 and 10.4-11). The observed target fragments consisted of small pieces of the clay targets that most likely had been hit by shotgun fired pellets, shattered in flight, and fell to the surface in small shards (Figure 10.4-10), and larger fragments that continued on a modified trajectory and fell to the ground further from the firing point (Figure 10.4-11) and closer to the tree line on the range.

Figure 10.4-10: Target Debris Observed on the Ground Surface at the Rum Point Skeet Range during SI Site Walk.

Figure 10.4-11: Larger Target Debris Fragments Observed on the Ground Surface at the Rum Point Skeet Range during SI Site Walk.

The detection of these types of range debris on the ground surface in a general gradation from the firing point on the Rum Point Skeet Range is consistent with anticipated deposition points for skeet range debris as illustrated in Figure 10.4-12.

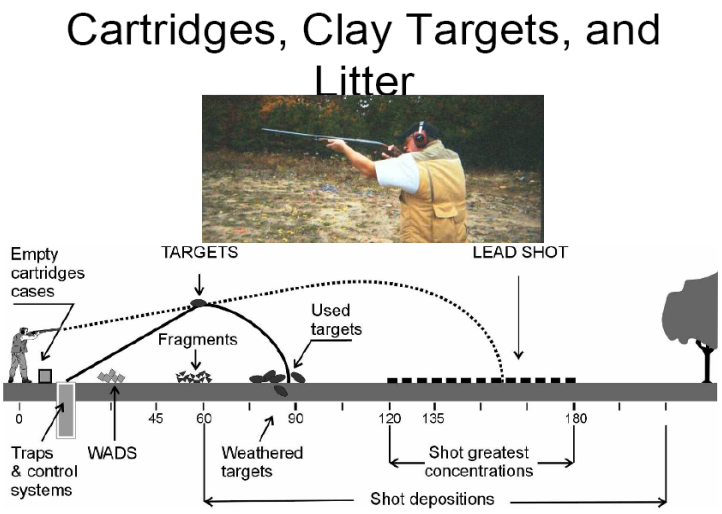


Figure 10.4-12: A Side View of a Typical Trap/Skeet Range Showing the Distribution of Range Residue (Shotgun Cartridge Wads, Target Fragments, Used Targets, and Shot Pellets) Located on the Ground Surface.

As illustrated on Figure 10.4-12, the clay pigeon targets were launched from a control system by means of a mechanical device. The target would pass across the field of view, downrange of the individual on the firing line, and the shotgun was aimed and shot to impact and break the clay target with the fired metal pellets. Shotgun wads ejected during firing would land near the firing point on the range surface (Figure 10.4-13).

Expended shotgun shell pellets missing their intended targets would continue undisturbed on a standard trajectory to the north toward the forested portions of the Rum Point Skeet Range. Intact targets would land on the ground surface. Targets successfully hit with shot on the range would fragment and fall to the ground surface. The much denser shot pellets, after they impacted the clay targets, would continue on a slightly revised trajectory and fall to the ground downrange.

Figure 10.4-13: Shotgun Cartridge Wads with Some Target Fragment Debris Observed on the Ground Surface (near Firing Line) at the Rum Point Skeet Range during SI Site Walk.

Map 10.4-2 depicts the zone of maximum shot fall accumulation, which is presumed to occur near the tree line on the Rum Point Skeet Range and away from the firing point. A shallow surface depression located northwest of the firing points receives surface water runoff from the range. Transport of dissolved metals and soil/sediment particulates occurs within this drainageway during precipitation events. Sampling efforts for the SI will include sampling of the surface sediment (and water if present) in this drainageway to evaluate potential impacts to surface water quality and to ascertain the transport mechanisms for contaminant migration (dissolved versus particulate transport) for MCs in range soil and sediment.

10.4.3 Munitions and Munitions-Related Materials Associated with the Site

This section describes the munitions or munitions-related materials known or suspected to be at the range. This includes both MEC and non-hazardous munitions-related debris (e.g., fragmentation, base plates, inert mortar fins).

The Rum Point Skeet Range was used as a recreational small arms range. According to the installation and documents from the Potomac River Gun Club, only shotguns were used at the range.

Based on the information obtained during the data collection process, the Rum Point Skeet Range is not suspected to contain CWM-filled munitions, electrically fuzed munitions, or DU-associated munitions.

10.4.4 MEC Presence

The entire range has been subdivided and categorized into one of three levels of MEC presence including known MEC areas, suspected MEC areas, and areas where no evidence exists to indicate that MEC are known or suspected to occur. The MEC categories are discussed below.

Known MEC Areas

There are no readily apparent MEC areas associated with this range because small arms do not pose an explosive hazard.

Suspected MEC Areas

There are no suspected MEC areas expected at this range because small arms do not pose an explosive hazard.

Areas Not Suspected to Contain MEC

Small arms are not explosive; therefore, the entire range is not suspected to contain MEC.

10.4.5 Ordnance Penetration Estimates

The depth to which munitions penetrate below the ground surface depends on many factors, including the type of soil, angle of impact, size of the munition, velocity at impact, and range-specific environmental conditions.

For small arms ranges, the ITRC has prepared a document titled, “Characterization and Remediation of Soils at Closed Small Arms Firing Ranges”, dated January 2003, to provide information on the general layout of small arms ranges, as well as information on areas that may be impacted with MC and/or MEC as a result of range use and the characteristics of the munitions used. According to the ITRC guidance, the penetration depth of small arms on the range floor is 1 foot or less. The document states that rounds that impact the range floor typically had a flat trajectory that fell short of or missed the target, or resulted from ricochet, and these fragments are usually found within the top 6 inches of soil. For trap and skeet ranges, the ammunition is dispersed as pellets over a small area in the direction of fire. According to the

1958 Programming Guide, the minimum safe range from a skeet/trap range is 900 feet. Pellets dispersed from a shotgun would be deposited on the ground surface and not significantly penetrate the ground unless disturbed.

10.4.6 Munitions Constituents

The potential for lead contamination exists over the entire 900-foot arc covered by the Rum Point Skeet Range. According to the ITRC guidance, the positions of the shooters and the angles at which the skeet targets are thrown create a fan-shaped shot fall zone. Figure 10.4-14 provides the typical shot fall layout of a skeet range. Depending on the shot angle, wind, and blocking effect of trees, the typical shotfall zone extends up to 680 feet. As illustrated on the figure, the overlapping fans create an area of maximum shot fall positioned approximately 375 to 600 feet from the firing point. The maximum shot fall zone is the area in which the highest concentrations of lead and debris are expected.

At the Rum Point Skeet Range, the cleared area of the range extends approximately 400 feet from the firing point. Thus, the theoretical maximum shot fall zone would extend past the tree line. The actual shot fall zone is an area slightly less than the theoretical shot fall zone due to the blocking effect of the trees. The theoretical and actual shot fall zones at the Rum Point Skeet Range as provided by the Potomac River Gun Club are illustrated on Figure 10.4-15.

The potential for lead contamination is greatest along the actual shot fall zone located at the tree line. There is a concern that the lead shot is embedded within the trees or possibly within the wetland area located on the western edge of the range. There was no record found of any excavations or environmental sampling on the range. Other MC at the range may include elevated concentrations metals such as antimony, copper, zinc, and arsenic from bullets, fragments, and bullet jackets. The only munitions-related debris items located on range were clay targets. These targets normally contain approximately 32 percent petroleum pitches (Chemical Abstract Service [CAS] 68334-31-6, CAS 68187-58-6) and 67 percent dolomitic limestone (CAS 16389-88-1). Petroleum pitch contains PAHs, some of which are classified as carcinogens. The targets are not considered an explosive or fire hazard. Several of these targets were found at the range during the visual survey. NG from unburned propellants and lead from primers is suspected at the firing range.

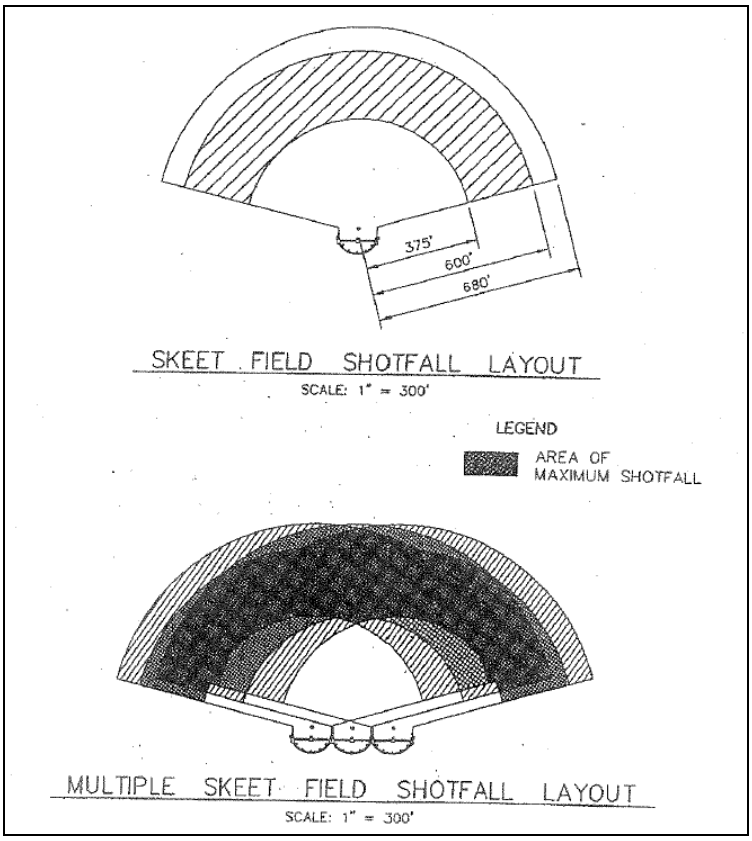


Figure 10.4-14: Typical Shot Fall Layout for a Skeet Range.

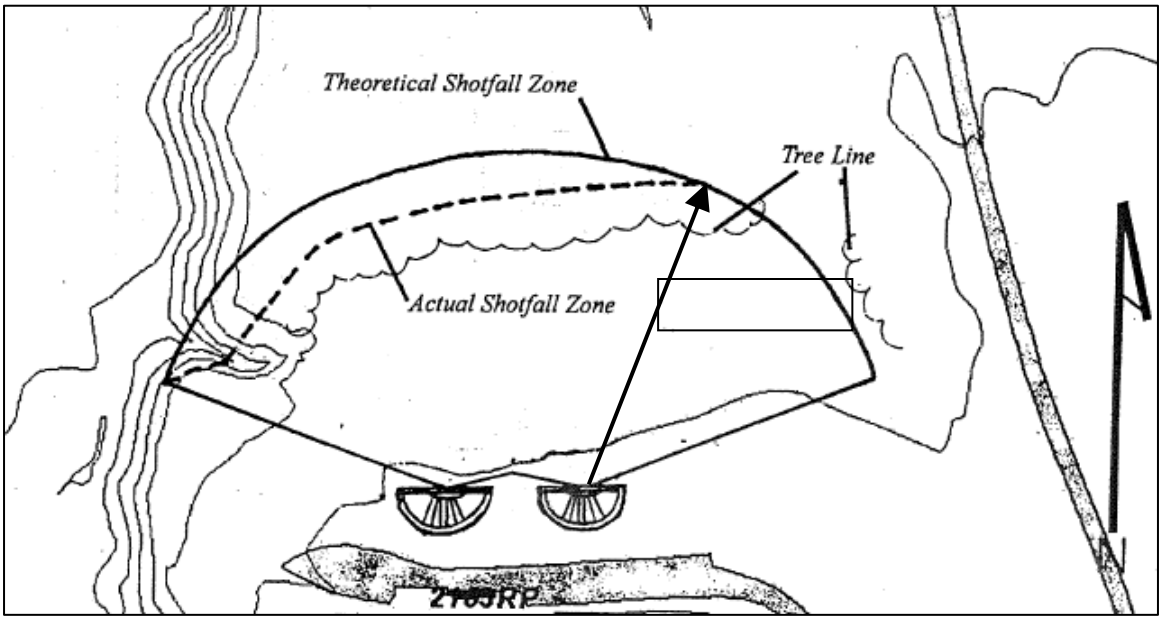


Figure 10.4-15: Location of Theoretical and Actual Shot Fall Zones on the Rum Point Skeet Range.

10.4.7 Contaminant Migration Routes

Contaminants at Rum Point Skeet Range may potentially migrate in soil, groundwater, surface water, and sediment. Direct human or biota contact with surficial and subsurface soil is possible if the soil is disturbed. The Rum Point Skeet Range's unnamed tributaries, wetlands, and proximity to Mattawoman Creek provide possible migration routes to surface water. The majority of the range area is sloped toward the wetland area and Mattawoman Creek. Storm water discharges to surface water via overland flow. Groundwater in the shallow water table aquifer also likely flows towards Mattawoman Creek; therefore, MC leaching from soil into shallow groundwater may migrate to surface water. Sediments can act as contaminant repositories, and sediment mixing and dredging can act as migration routes to surface water. MC in surface soil and sediment may migrate via plant/animal uptake. Based on a review of hydrogeological data, it is unlikely that MC in shallow groundwater would migrate to the deeper aquifers used as water supplies. However, shallow groundwater is still considered a potential exposure medium.

10.4.8 Receptors

Potential human receptors include authorized Navy personnel (military and civilian), visitors, contractors, maintenance workers, recreational users, and trespassers. Plant and animal biota are also potential receptors. Examples of ecological receptors include deer, wild turkey, and waterfowl.

Buildings Near/Within Site

Building 2153 is located on Skeet Range Way on the southern side of the former range. The Potomac River Gun Club used the building while the range was operational. A wooden fence divides the building from the firing area. The other structures on range include storage boxes/trailers designated C12, C13, C45, and C44 that are positioned at the end of Skeet Range Way. The low, high, and trap houses once used for the skeet range are still intact. The two trap houses are numbered 2169RP and 2163RP, the high house is 2170RP, the one combination house is Building 2164RP, and the low house is Building 2162. There are no other installation structures on or near the range.

Utilities On/Near Site

Building 2153, the former Potomac River Gun Club Headquarters, has water and electricity. Streetlights are located along Skeet Range Way. No overhead power lines are visible. A 1990 construction map of the range outlines installation of a ¾-inch conduit including ground wires to be installed 18 inches below the surface. The wires are shown connecting the low, high, and trap houses to an unidentified trailer on the range, which is assumed to be Building 2153.

10.4.9 Land Use

There is no readily available information on the land use prior to 1991. From 1991 until 2001, the area was maintained as a skeet and trap range. The range was primarily used and maintained by the Potomac River Gun Club. The club was operational at NSF-IH for approximately 15 years at another location on the Stump Neck Annex prior to its move to the Rum Point Skeet Range in 1991. The club volunteered to relocate due to environmentally sensitive issues at its previous location. Building 2153 was used as the club's headquarters.

10.4.10 Access Controls/Restrictions

A locked gate at the east end of Skeet Range Way limits access to the range; however, the Rum Point Skeet Range is not within the Stump Neck secured perimeter fence. A wooden split rail fence runs along the southern and eastern edges of the Rum Point Skeet Range. Skeet Range Way leads directly to the southern edge of the range. The area is surrounded by forests on the northern and eastern sides. Wetlands are located in the western portion of the site.

According to the 2003-2004 Stump Neck Annex Hunting Map, the former skeet range is located within an eagle protection area as designated by the installation. Hunting is permitted within this region by permission.

The wetlands located on the western edge of the range are protected under Executive Order 11990, which prohibits construction in a wetland area unless there is no practicable alternative and all possible measures are taken to minimize the environmental impacts. Wetlands are also protected under Section 404 of the Clean Water Act, which requires a permit to be obtained from the USACE before any work in a wetland can begin. The wetland on the former skeet range is classified as Palustrine forested broad-leaved deciduous wetland.

According to the Soil Survey of Charles County, many of the soil types at Stump Neck and Indian Head have load-bearing capacity limitations as a result of high water tables and extreme erodability conditions. Based on this information, the installation has limited the construction and use of septic systems in specific areas of concern. The former Rum Point Skeet Range is located in a region that has a seasonal high water table and overlaps an area containing hydric soils. The range overlaps two restriction areas: one in which septic systems are prohibited and the other in which a waiver is required for septic systems.

10.4.11 Conceptual Site Model

A general description of the CSM exposure pathway analysis is included in Section 10.4.7. For the purpose of this SAP, only MC associated with the Rum Pont Skeet Range is considered in the CSM exposure pathway analysis.

For the Rum Point Skeet Range, historical and visual evidence indicates that MEC are not present. Therefore, no complete exposure pathways exist for MEC, and no exposure pathway analysis was completed for MEC. Soil and surface water/sediment impacted by MC represents a primary potential source medium, as illustrated in the CSM (Map 10.4-3). Exposures to surface soil, subsurface soil, and surface water/sediment containing MC may present potentially complete exposure pathways for human and ecological receptors. The MC exposure pathway analysis for the Rum Point Skeet Range is presented on Figure 10.4-16. All human and ecological receptors have potentially complete exposure pathways for direct contact with MC in surface soil, which includes dermal contact, ingestion, and inhalation (dust). Runoff, discharges, and/or erosion may transport MC from surface soil to surface water/sediment. Because the area is partially located on a wetland, there are also potentially complete pathways for all human and ecological receptors of surface water/sediment. The food chain also represents an exposure medium when considering plant/animal uptake for biota (including game such as deer and wild turkey). Hunting is permitted at the site during certain periods of the year by permit only, creating potentially complete pathways from the food chain to all human receptors if hunted game are consumed. Precipitation infiltration may provide for lead and PAH mobility into subsurface soil and to the surficial groundwater aquifer. Potentially complete exposure pathways exist for MC in subsurface soil (direct contact, ingestion, and inhalation during intrusive work activities) for all human and ecological receptors with the exception of trespassers. It is not anticipated that trespassers would come in contact with subsurface soils. Although confining layers are expected to prevent the migration of MC to the lower aquifers used for water supplies, potentially complete pathways exist for MC in shallow groundwater for human receptors.

10.4.12 Problem Definition Summary

The following is a summary of the problem definition that is detailed in Worksheet #10.4:

- Environmental contamination may exist at the Rum Point Skeet Range (33.45 acres) because the facility was active for 10 years (1991 to 2001) as a skeet range (limited to shotguns). Specific small arms ammunition types and materials used at the Rum Point Skeet Range may have included .410-, .28-, .20-, and 12-guage shells, and clay targets. Based on the anticipated annual site usage,

53,000 pounds of lead and 196,800 pounds of clay target fragments may have accumulated in soil over the 10 years of operations. Lead shot may also be imbedded in trees along the tree line within the shot fall zone for the range. Much of the area within the boundary of the range drains to the west via a shallow surface depression that conveys surface water and sediment into a wetland and unnamed tributaries that discharge into Mattawoman Creek.

- MC consisting of metals (primarily lead and, to a lesser extent, antimony, arsenic, copper, tin, and zinc), NG, and PAHs (from pitch tar used in the clay pigeon targets) may be present in site soil (particularly surface soil [the 0-to 6-inch interval]) and surface water and sediment of the adjoining tributaries, wetland, and Mattawoman Creek as a result of the military training activities. Lead is assumed to be the primary metal MC of concern in soil because it is the primary constituent of the spent munitions and because of its documented toxicity to human and ecological receptors. It is anticipated that other metals contamination will be spatially correlated with lead concentrations. MC in surface soil may have migrated to subsurface soil and subsequently infiltrated to underlying groundwater. These constituents may have also migrated to adjoining surface water and sediment via surface soil run off or the discharge of contaminated shallow groundwater to the surface water of the adjoining tributaries, wetland, and Mattawoman Creek. However, the nature and extent of the potential environmental contamination at the Rum Point Skeet Range is not known at this time (no environmental sampling has been conducted to date).

- The CSM (detailed in Worksheet #10.4) indicates that potentially complete exposure pathways exist for both human and ecological receptors under both current and hypothetical future land uses. However, potential human and ecological risks have not been characterized for receptors exposed to the potentially contaminated environmental media. Unacceptable levels of human and/or ecological risk may exist.

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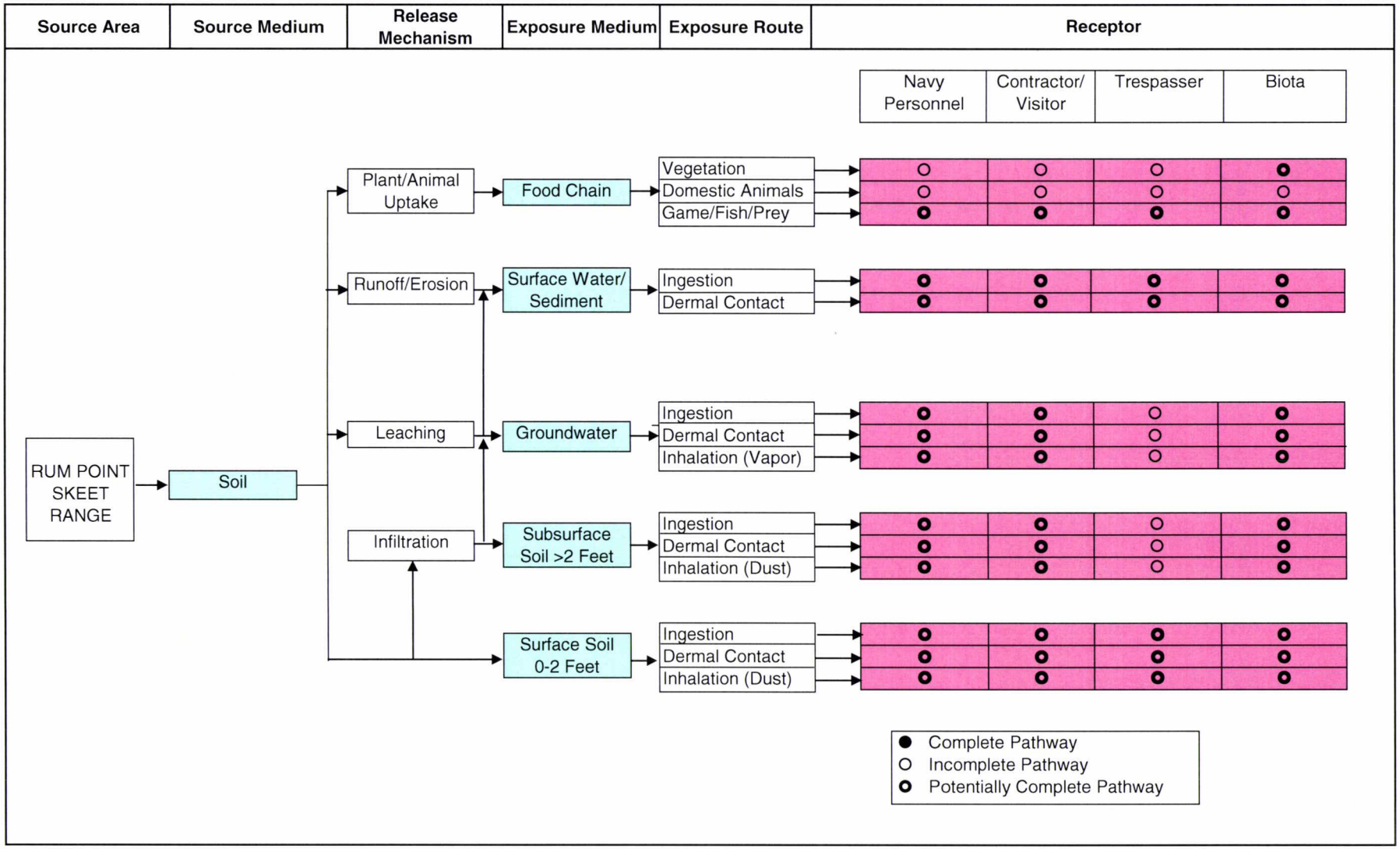
FIGURE 10.4-16

RUM POINT SKEET RANGE
MC EXPOSURE PATHWAY ANALYSIS
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND



SAP WORKSHEET #10.5:
Problem Definition/Conceptual Site Model for Small Arms (Pistol) Range (Site UXO-17)

10.5 SMALL ARMS (PISTOL) RANGE

10.5.1 History and Site Description

The Small Arms Range is approximately 2.41 acres in area and is located at the eastern perimeter of the Stump Neck peninsula. The SDZ, which extends east of the Small Arms Range, covers 382.12 acres. Navy personnel used this range for training and qualifying activities from the mid-1980s until 1991. Rounds were fired into the side of a hill located on the range. The Small Arms Range was closed in August 1991 due to its proximity to General Smallwood State Park, which borders the range on the target side, presenting the potential for stray rounds to enter the park. Subsequently, the range was used for bow qualification, the exact dates of which are unknown.

The Small Arms Range is also known as the Old Pistol Range. When used as a pistol range, the range had three firing lines with north-south orientation, two concrete walkways with east-west orientation, and a stone walkway located in the northern portion of the range (Map 10.5-1). According to personnel interviews, silver-contaminated soil from the X-ray Facility (IR Site #5, Building 731 on the Main Installation) was buried and covered with a clay layer at the Small Arms Range after the range was closed. The buried soil is in two earthen mounds located near the center of the Small Arms Range and is not considered part of the range (see Figure 10.5-1). Currently the Small Arms Range is an open field.

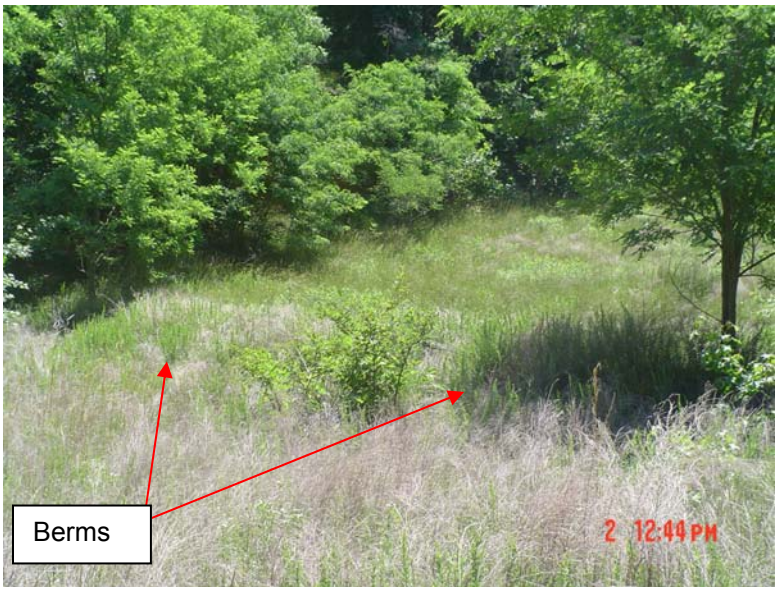


Figure 10.5-1: Silver Contaminated Soil Mounds

In 1992, this range was identified as SWMU #29: Pistol Range and added to the IR Program at the Stump Neck Annex. In January 2002, a desktop audit was performed and concluded that insufficient information was available; therefore, the Small Arms Range still remains an AOC pending investigation.

Topography

Section 10-3.2 provides a general description of topography for the Stump Neck Annex. The topography of the Small Arms Range can be described as relatively flat with a hill on the eastern side, bordering General Smallwood State Park. The highest point on the Small Arms Range is in the northeastern corner at an elevation of approximately 100 feet above msl.

Geology

Section 10-3.3 provides a description of the geology of Indian Head and the Stump Neck Annex, which is applicable to the Small Arms Range. There is no range-specific geological information for the Small Arms Range.

Soil and Vegetation Types

Section 10-3.4 provides a description of the soil and vegetation types at Indian Head and the Stump Neck Annex. According to the Soil Survey of Charles County, the predominant soil types at the Small Arms Range are silty loams and gravelly sandy loam. Specifically, the soils consist of moderately eroded,

Beltsville silt loam with 2 to 5 percent slopes and a moderately eroded Aura gravelly loam with 10 to 15 percent slope. The Small Arms Range is located within a hardwood forest area.

Hydrology

Section 10-3.5 provides a description of hydrology at Indian Head and the Stump Neck Annex. The closest water body to the Small Arms Range is an unnamed tributary along the western boundary of the range. Surface water runoff likely follows topography at the range towards the unnamed tributary. There are no known wetlands in the area.

Hydrogeology

Section 10-3.6 provides a description of hydrogeology for Indian Head and the Stump Neck Annex, which includes the Small Arms Range. It is assumed that shallow groundwater flow from the Small Arms Range follows topography towards the unnamed tributary and is connected to the area's dominant surface water bodies (Mattawoman Creek and Potomac River).

Cultural and Natural Resources

General cultural and natural resources for NSF-IH are provided in Section 10-3.7. According to the 1996 Phase I Cultural Resources Survey of Stump Neck Annex, there are no archeological/cultural sites within the Small Arms Range. Several shovel test pits were dug on the site, and all were negative for artifacts.

Endangered and Special Status Species

As discussed in Section 10-3.8, endangered species are reported to exist at the Stump Neck Annex. These species may be present at the Small Arms Range.

10.5.2 Visual Survey Observations and Results

A visual reconnaissance of the Small Arms Range was conducted on June 25, 2003. Malcolm Pirnie personnel walked the site from the southwestern corner of the range towards the northwestern corner. The Small Arms Range currently consists of an open field. Two berms are located in the central portion of the Small Arms Range. According to range personnel, silver-contaminated soil from the X-ray Facility was buried on site in approximately 1992. The majority of the range is covered by thick vegetation including tall grasses and hardwood species. Evidence of the former range was observed, including three firing lines and target boards. Concrete walkways, a small wooden shack, and wooden stands were

also observed at the Small Arms Range (see Figures 10.5-2 and 10.5-3). The wooden stands were likely used as tree stands to shoot at the targets. However, it is not known if the tree stands were used for pistol shooting, bow shooting, or both.

There were no physical indications of MEC observed during the inspection of the Small Arms Range. Because only small arms were used, MEC is not anticipated at the Small Arms Range. Although no munitions and/or ordnance items were observed on the surface of the areas walked during the survey, the possibility that MC may remain beneath the surface cannot be ruled out based on use of the site as a small arms range.

Figure 10.5-2: Wooden Shack Observed at Northwestern End of Small Arms Range.

Figure 10.5-3: Wooden Stands Observed Behind the Shack at the Small Arms Range.

A series of site walks were conducted with the SI planning project team during the late fall and winter of 2007. An initial site walk of all of the Stump Neck Annex SI MRP sites was completed on September 18, 2007, by Ralph Basinski (Tetra Tech), Joe Rail (Navy RPM), and Shawn Jorgensen (Indian Head Environmental Department) as part of the project kick-off meeting. A subsequent site walk was conducted on November 8 to 10, 2007, and included a larger contingent of the project team: Joe Rail (Navy RPM), Shawn Jorgensen (Indian Head Environmental Department), and several Tetra Tech personnel (Ralph Basinski, Ralph Brooks, Jim Coffman, George Latulippe, and Rick Barringer). Each MRP site was walked, and multiple photographs were taken to document conditions observed during the site walks (Figures 10.5-4 through 10.5-10). The site walks were performed in conjunction with technical meetings at the facility that reviewed the proposed approaches for the MC fieldwork programs.

An expanded site walk/meeting was conducted on December 18 and 19, 2007, and included previously identified Navy and Tetra Tech personnel, as well as representatives from the MDE (Curtis Detore) and

USEPA Region 3 (Dennis Orenshaw). As before, MRP sites were walked and photographed to record the conditions observed on the sites. Project quality objectives for small arms ranges were discussed among the meeting attendees during December 19, 2007.

Figure 10.5-4: General View of Area Conditions at the Small Arms Range as Observed During SI Site Walk, showing the Wooden Shack and the Wooden Firing Stands Depicted in Figures 10.5-2 and 10.5-3.

Figure 10.5-5: Earthen Hillside Slope Backing Former Target Locations on the Small Arms Range as Observed During SI Site Walk.

Figure 10.5-6: View of the Area Vegetation Conditions from the Former Firing Line Area (Range Floor in Foreground) on the Small Arms Range as Observed During SI Site Walk. The Earthen Hillside is in the Background (Downrange) and Nearly Obscured by Tree and Shrub Growth.

During the SI site walks, it became apparent that there had been some slumping of the slope material on the hillside that received the small arms ammunition expended on the range (see Figure 10.5-7). This slumped earthen material also showed erosion rills from surface water overland flow.

During the SI site walks, mounded areas were noted on the southern side of the range floor that appeared to correspond to the buried silver-contaminated soil from the X-ray Facility, as described in Section 10.5.1. The wooden firing stands on the northern side of the range (shown in Figure 10.5-4) may be the wooden stands reportedly used at the former Roach Road Rifle Range, which closed in 1986. The Small Arms Range on Rum Point Road began operations in the mid-1980s and was most likely constructed as a replacement site to host the small arms training and weapons qualification testing that had previously been performed at the Roach Road Rifle Range.

Certain range-related materials such as the concrete bases for the range targets, concrete walkways, and what appear to be rusted iron range target frames (Figure 10.5-8) were observed on the range floor of the Small Arms Range.

Figure 10.5-7: View of Earthen Embankment Behind Former Range Target Locations at the Small Arms Range as Observed During SI Site Walk Showing Evidence of Slumping and Erosion Rills from Overland Flow.

Figure 10.5-8: Discarded Target Frames on the Ground Surface at the Small Arms Range as Observed During SI Site Walk.

The following observations were made based on the conditions at the Small Arms Range. During the SI site walks, it became apparent that the earthen materials in the target impact berm had slumped and were being eroded by overland flow processes. Because the Small Arms Range is located at a high point on the Stump Neck Annex, there is a likelihood that surface water flow and soil/sediment transport could occur to nearby drainages to the north and south of the range, east of Rum Point Road. Both of those drainages lead into property that is beyond the boundary of the Stump Neck Annex and is part of the adjacent General Smallwood State Park. Range operations were discontinued in 1991 for safety purposes because stray rounds from the Small Arms Range had the potential to enter the park, which was downrange of the firing line and behind the range impact berm.

Small arms ranges are generally level and have some sort of impact berm or hillside slope to collect and contain the expended small arms ammunition. Figure 10.5-9 shows an example of an active small arms range that has many similarities to the Small Arms Range on Rum Point Road. The targets are located in front of an earthen impact berm, and there are three concrete firing lines to support small arms training at different distances. The depicted conditions are consistent with the historical aerial photographs as presented in the PA Report (Malcolm Pernie, 2005).

Figure 10.5-9: Operational Small Arms Range Similar to the Small Arms Range on Rum Point Road.

Soil on the hillside behind the Small Arms Range appears to have slumped in some areas since the range ceased operations in 1991. Expended bullets passing through the targets or passing above the targets would continue on a trajectory into the earthen hillside (impact berm) behind the range targets. As shown on Figure 10.5-10, the downrange earthen hillside directly behind the Small Arms Range targets would contain concentrated metal from the expended bullets.

Based on these observations, there should be a primary area for the accumulation of expended small arms ammunition on the Small Arms Range. Bullets aimed below the range targets would be dispersed in the impact berm soil near the toe of the berm slope. Fired bullets that successfully hit the targets most likely continued on a trajectory through the targets and into the hillside impact berm. Range fire that overshot the targets would impact the soil above and behind the targets, but most likely within the surficial soil of the impact berm.

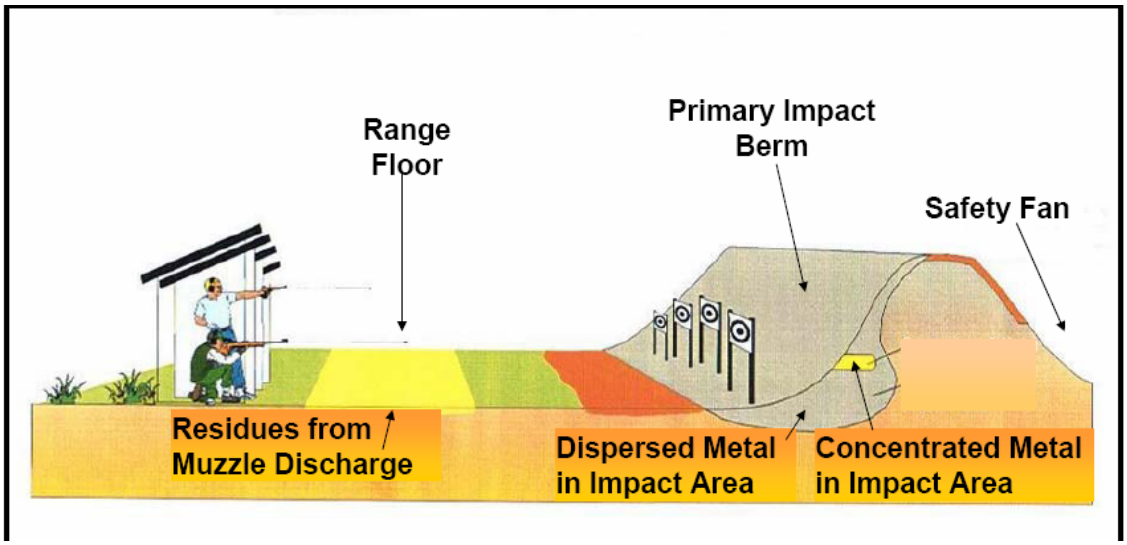


Figure 10.5-10: Typical Distribution of Residue and Expended Ammunition on a Small Arms Training Range is Similar to That Expected on the Ground Surface and within the Target Berm Materials Soil at the Small Arms Range.

10.5.3 Munitions and Munitions-Related Materials Associated with the Site

This section describes the munitions or munitions-related materials known or suspected to be at the range, including the types and estimated maximum penetration depths. This includes both MEC and non-hazardous munitions-related debris (e.g., fragmentation, base plates, inert mortar fins). Potential ordnance concentration areas are presented along with a discussion on the presence of any special consideration ordnance.

According to the Army Technical Manuals on small arms ranges (AR 750-10, TM 9-855), the maximum range for .22-cal ammunition is 4,500 feet, with a muzzle velocity of 1,100 feet per second. The maximum range for .45-cal ammunition is 4,800 feet, with a muzzle velocity of 802 feet per second. The SDZ for a .45-cal pistol range extends downrange from each end of each firing line at a 5 degree angle for 4,800 feet. An additional SDZ, also originating from the end of each firing line, extends downrange at a 25 degree angle for 3,600 feet. An example of a typical SDZ for a pistol range is provided on Figure 10.5-11.

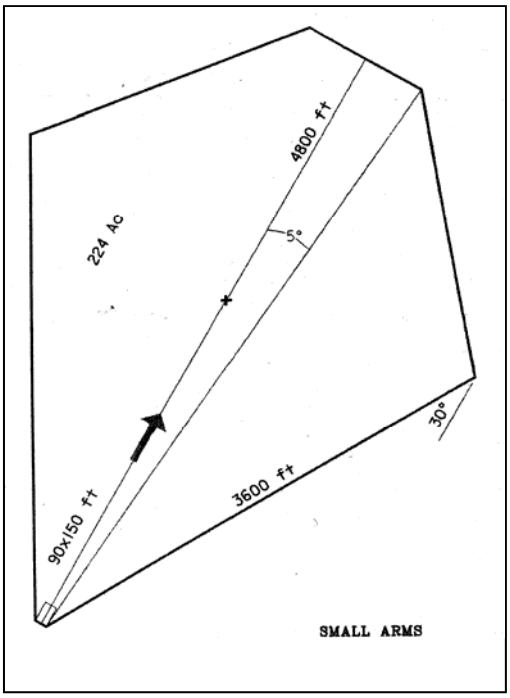


Figure 10.5-11: SDZ for a Typical .45-cal Pistol Range.

According to a 1991 memorandum regarding the Small Arms Range, the SDZ reportedly extends 6,234 feet from the firing line. From this information, ammunition fired at the Small Arms Range likely included .50-cal ammunition, which travels further than the typical .22-cal and .45-cal ammunition. The Stump Neck Annex property line is approximately 900 feet from the firing line. The SDZ extends east of the installation boundary into the General Smallwood State Park.

Detailed records of the types and quantities of small arms ammunition used at the Small Arms Range were not available. However, the following small arms ammunition were likely used at the Small Arms Range: .22-cal, 9-mm, .45-cal, and .50-cal.

Based on the information obtained during the data collection process, the Small Arms Range is not suspected to contain CWM-filled munitions, electrically fuzed munitions, or DU-associated munitions.

10.5.4 MEC Presence

The entire range has been subdivided into one of three levels of MEC presence including known MEC areas, suspected MEC areas, and areas where no evidence exists to indicate that MEC are known or suspected to occur. These MEC categories are discussed below.

Known MEC Areas

Based on historical documents and information obtained during the data collection process, there is no evidence of MEC at the Small Arms Range because only small arms were used. Therefore, there are no known MEC areas.

Suspected MEC Areas

Based on historical documents and information obtained during the data collection process, there is no evidence of MEC at the Small Arms Range because only small arms were used. Therefore, there are no suspected MEC areas.

Areas Not Suspected to Contain MEC

Based on historical documents and information obtained during the data collection process, there is no evidence of MEC at the Small Arms Range because only small arms were used. Therefore, the entire Small Arms Range is not suspected to contain MEC.

10.5.5 Ordnance Penetration Estimates

The depth to which munitions penetrate below the ground surface depends on many factors, including the type of soil, angle of impact, size of the munitions, velocity at impact, and range-specific environmental conditions.

For small arms ranges, the ITRC has prepared a document titled, “Characterization and Remediation of Soils at Closed Small Arms Firing Ranges” (January 2003), to provide information on the general layout of small arms ranges, as well as information on areas that may be impacted with MC and/or MEC as a result of range use and the characteristics of the munitions used. According to the ITRC guidance, the penetration depth of small arms on the range floor is 1 foot or less. The document states that rounds that impact the range floor are typically at a flat trajectory that fell short of or missed the target or those resulted from ricochet, and these fragments are usually found within the top 6 inches of soil. Penetration depths within the side of the hill may vary depending on the soil type and other conditions but are expected to be up to 1 foot or more.

10.5.6 Munitions Constituents

For small arms, the primary MC of concern is lead from bullets. Other associated MC less likely to be of concern may include antimony, arsenic, copper, magnesium, nickel, strontium, tin, zinc, and lead styphnate/lead azide. These items are not consumed when the munitions items function as they are designed. NG from unburned propellants and lead from primers is suspected at the firing points. Therefore, the potential exists for these constituents to remain in surface soil at the Small Arms Range.

10.5.7 Contaminant Migration Routes

Contaminants at the Small Arms Range may potentially migrate within soil, groundwater, surface water and sediment. Direct human or biota contact with surficial soil is considered the most likely exposure scenario. An unnamed tributary is located near the western boundary of the Small Arms Range and represents a primary source medium for MC. MC could potentially migrate from surface soil to subsurface soil or groundwater. Based on a review of hydrogeological data, it is unlikely that MC in shallow groundwater would migrate to the deeper aquifers that are used as water supplies. However, shallow groundwater is still considered a potential exposure medium. MC in surface soils may also migrate via plant/animal uptake.

10.5.8 Receptors

Potential human receptors include authorized Navy personnel (military and civilian), visitors, contractors, maintenance workers, recreational users, and trespassers. Plant and animal biota are also potential receptors. Examples of ecological receptors include deer, wild turkey, and waterfowl.

Buildings Near/Within Site

A wooden shack, which possibly was used for storage of targets and ammunition, is located in the northwestern portion of the Small Arms Range. Structures located near the Small Arms Range include facilities in General Smallwood State Park, located approximately 800 feet to the southwest of the range. There are also installation buildings located approximately 1,200 feet west and north of the Small Arms Range.

Utilities On/Near Site

Based on the Stump Neck Geographic Information Systems (GIS) utility mapping, no utilities exist at the Small Arms Range.

10.5.9 Land Use

The Small Arms Range was used for pistol training and qualifying activities by the facility's Security Department until 1991. Subsequently, the range was used for bow qualification. It is not known when bow qualification was discontinued at the Small Arms Range; however, the Small Arms Range is currently not used. The range is covered with high grass and surrounded by trees on the northern edge (Figure 10.5-12). Open fields, trees, grass, and an access road surround the range. The area to the east and south of the Small Arms Range is General Smallwood State Park. Rum Point Road runs along the western border of the range. The Rum Point Skeet Range is north of the Small Arms Range.

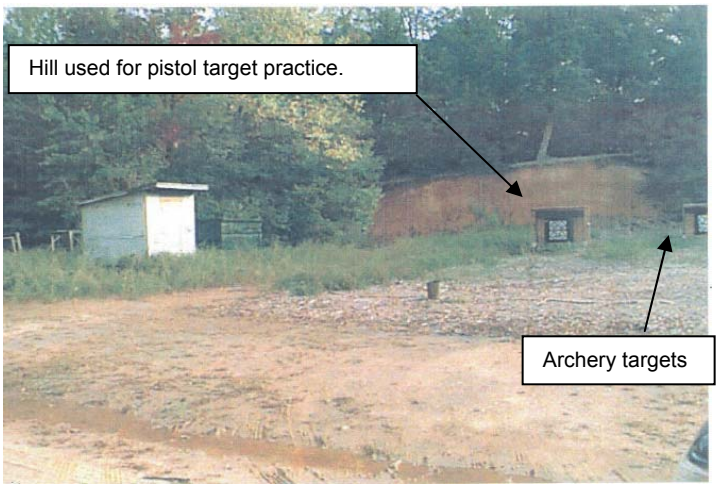


Figure 10.5-12: Hill used for Target Practice and Archery Targets.

10.5.10 Access Controls/Restrictions

No public access is authorized at NSF-IH. Signs, partial fencing, locked/secured gates, login book/office check-in, and vehicle security patrols are used to control the entire facility. The Small Arms Range is located outside of the complex along Rum Point Road. A locked gate prevents access to the Small Arms Range; however, the area is not guarded. There are no known land use/development restrictions for the range.

10.5.11 Conceptual Site Model

A general description of the CSM exposure pathway analysis is included in Section 10.5.7. For the purpose of this SAP, only MC associated with the Small Arms Range is considered in the CSM exposure pathway analysis.

For the Small Arms Range, historical and visual evidence indicate that MEC are not present. Therefore, no complete exposure pathways exist for MEC, and no exposure pathway analysis was completed for MEC. MC may be present; therefore, potential MC exposure pathways do exist. As illustrated on Map 10.5-2, soil and surface water/sediment impacted by MC represent primary source media. The MC

exposure pathway analysis for the Small Arms Range is presented on Figure 10.5-13. All human and ecological receptors have potentially complete exposure pathways for direct contact with MC in surface soil, which includes dermal contact, ingestion, and inhalation (dust). Runoff, discharges, and/or erosion may transport the MC from surface soil to surface water/sediment. There is a small tributary on the western edge of the Small Arms Range, so potentially complete pathways exist for all human and ecological receptors of surface water/sediment. The Small Arms Range is located within an upland hunting area. Human and ecological receptors have potentially complete pathways by ingesting game/prey that previously consumed contaminated vegetation or prey. Precipitation infiltration and leaching may provide for lead and copper mobility into subsurface soil and shallow groundwater. Potentially complete exposure pathways exist for MC in subsurface soil (direct contact, ingestion and inhalation during intrusive work activities) for all human and ecological receptors with the exception of trespassers. It is not anticipated that trespassers would come in contact with subsurface soil. Although confining layers are expected to prevent the migration of MC to the lower aquifers used for water supplies, potentially complete pathways exist for MC in shallow groundwater for human receptors.

10.5.12 Problem Definition Summary

The following is a summary of the problem definition which is detailed in Worksheet #10.5:

- Environmental contamination may exist at the Small Arms (Pistol) Range (2.41 acres) because the facility was an active range from the mid-1980s through 1991. Specific small arms ammunition types and materials used at the range most likely included small arms pistol ammunition and possibly rifle ammunition (9mm, .45-cal, .22-cal, and .50-cal). Expended ammunition is suspected along the range floor immediately in front of the hillside, in the earthen hillside embankment to the east, and behind the former range targets (i.e., the primary impact berm). There appears to be some limited slumping (slope failure) along this hillside such that uncontaminated soil at the head of the slope may now cover potentially contaminated soil at the toe of the slope.

- MC consisting of metals (primarily lead and, to a lesser extent, antimony, arsenic, copper, tin, and zinc) and NG may be present in site soil (particularly surface soil [the 0-to 6-inch interval]) and surface water and sediment of drainageways leading to an unnamed tributary creek along the western boundary of the range and from the range to property that is beyond the boundary of the Stump Neck Annex (i.e., into General Smallwood State Park). Lead is assumed to be the primary metal MC of concern because it is the primary constituent in the spent munitions and because of its documented toxicity to both human and ecological receptors. It is anticipated that other metals contamination will be spatially correlated with lead concentrations. MC in surface soil may have

migrated to subsurface soil and subsequently infiltrated to underlying groundwater. These constituents may have also migrated to the aforementioned adjoining surface water bodies via surface soil runoff or discharge of contaminated shallow groundwater to the surface water. However, the nature and extent of the potential environmental contamination is not known at this time (no environmental sampling has been conducted at the Small Arms (Pistol) Range to date).

- The CSM (detailed in Worksheet #10.5) indicates that potentially complete exposure pathways exist for both human and ecological receptors under both current and hypothetical future land uses. However, potential human and ecological risks have not been characterized for receptors exposed to the potentially contaminated environmental media. Unacceptable levels of human and/or ecological risk may exist.

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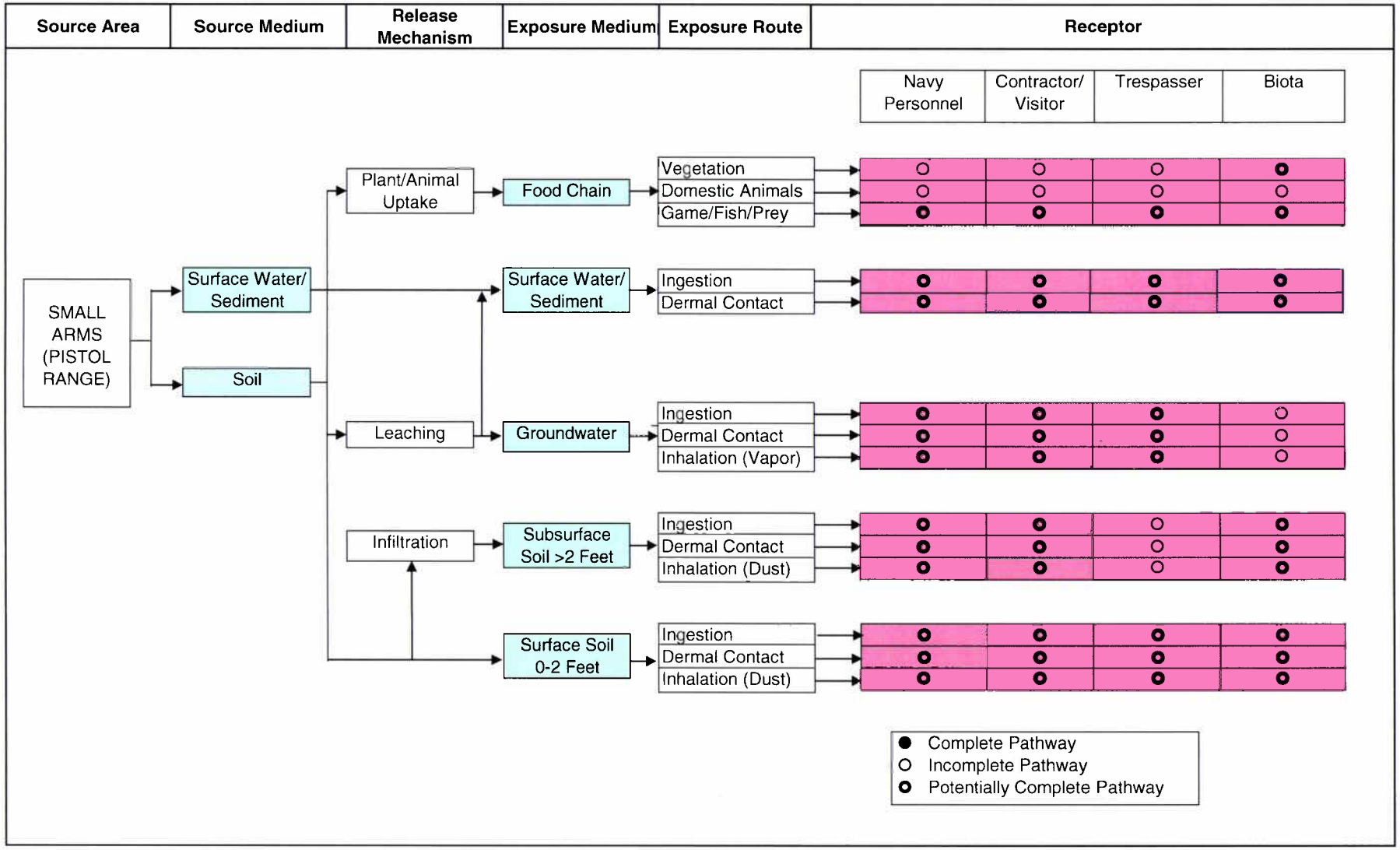
FIGURE 10.5-13

SMALL ARMS (PISTOL RANGE)
MC EXPOSURE PATHWAY ANALYSIS
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND



SAP Worksheet #11.1 - Project Quality Objectives/Systematic Planning Process Statements for The Marine Rifle Range

PROBLEM DEFINITION (STEP 1)

Worksheet 10.1 (Section 10.1.12) contains the problem definition for the Marine Rifle Range.

IDENTIFY THE GOALS OF THE STUDY (STEP 2)

The primary goals of this investigation of the Marine Rifle Range are as follows:

1. Collect adequate data to delineate the horizontal and vertical extent of soil and possible drainageway/creek sediment contamination. If the data collected in the initial round of sampling are adequate, stop delineation; otherwise, continue sampling (i.e., “step-out” vertically and/or horizontally as necessary).

2. Determine whether MC (specifically antimony, arsenic, lead, copper, tin, and zinc) are present in the study area soil and drainageway/creek sediment and also if NG and lead are present in the soil in the area of the firing lines at concentrations that represent unacceptable human health or ecological risk. If either of these risks are unacceptable, evaluate the need to proceed to a Feasibility Study (FS); otherwise, recommend No Further Action (NFA).

3. Determine whether MC (specifically NG, antimony, arsenic, lead, copper, tin, and zinc) are present in study area soil at concentrations that represent a threat to groundwater quality. If MC concentrations in soil do represent a threat to groundwater quality, recommend an environmental investigation of groundwater contamination; otherwise, do not initiate a groundwater investigation on the basis of soil contamination levels.

4. Update the CSM using field reconnaissance survey data and initial hazard and risk screening results.

All data collected for this project are expected to be transferable to support the following actions:

1. Update the Navy cost-to-complete (CTC) estimate
2. Complete the site prioritization protocol

IDENTIFY INFORMATION INPUTS (STEP 3)

Data required for making decisions include the following:

1. A comprehensive listing of the relevant, medium-specific Risk-Based Screening Levels (RBSLs) and potential chemical-specific Applicable or Relevant and Appropriate Requirements (ARARs) for the MC of concern (specifically NG, antimony, arsenic, lead, copper, tin, and zinc). Screening levels based on USEPA Region 3 Risk-Based Concentrations (RBCs), the USEPA action level for lead in soil assuming a residential land use scenario (400 mg/kg), USEPA Soil Screening Levels (SSLs), USEPA Region 3 BTAG Sediment Screening Benchmarks, USEPA Ecological SSLs, USEPA Region 3 BTAG SSLs, and USEPA Region 3 Freshwater Screening Benchmarks.

2. Laboratory analytical data characterizing concentrations of NG and the six MC metals of interest in the soil (surface soil [0 to 1 foot bgs] and subsurface soil [greater than 1 foot bgs], as necessary) and drainage-way/creek sediment samples (0 to 6 inches bgs). Soil and sediment samples will be analyzed for NG using Appendix A of SW-846 Method 8330B and the specified MC metals using SW-846 Method 6020A. MC concentrations in soil and sediment will be compared to site screening levels and background concentrations. These data are necessary to conduct human health and ecological risk screening assessments. Laboratory quantitation limits for the proposed analytical methods should not exceed the aforementioned screening levels and ARARs. Practical quantitation limits should not exceed risk-based project action levels (i.e., levels associated with a $1x10^{-4}$ cumulative cancer risk level, cumulative hazard index of 1, or USEPA action level for lead [400 mg/kg]). Based on knowledge of lead concentrations in similar soils, native soil and sediment lead concentrations are expected to be less than 50 mg/kg. Because of this, contaminated areas (e.g., with lead concentrations exceeding 400 mg/kg) are expected to be well delineated, with steep lead concentration gradients between clean and contaminated areas.

3. Because lead is the primary contaminant, and other metals are expected to be collocated with lead, lead can be used as a marker for contamination. For delineation of lead concentrations, the use of both field and fixed-base laboratory analyses is planned. This will provide a high degree of spatial coverage for relatively low cost. XRF will be used for lead analysis in the field. An XRF field action level of 300 mg/kg will be used as the discriminator between contaminated and uncontaminated locations. The 300 mg/kg level corresponds to the USEPA action level of 400 mg/kg reduced by a 25 percent margin of error to minimize the potential that contaminated areas go undetected. The margin of error was selected based on recent experiences with field XRF analyses at other small arms ranges.

4. Laboratory analytical data characterizing soil and sediment pH, total organic carbon (TOC), and cation exchange capacity (CEC) characteristics. These data are necessary for the evaluation of MC environmental fate and transport.

5. Background soil data suitable for site soil-versus-background soil data comparisons for the six metals of interest for use in site versus background comparisons.

6. Field identification/classification of soil and sediment types (i.e., lithology and Unified Soil Classification System [USCS] for grain size, color, plasticity, etc.). Worksheet #21 contains relevant SOPs.

7. Physical characterization of the major surface water drainage pathways (e.g., number, width, depth, physical characteristics of sediments, etc.). Worksheet #21 contains relevant SOPs.

8. Field investigation specifications (e.g., SOPs for sample collection).

9. QA/QC specifications and the results of the QA/QC reviews (e.g., data validation) conducted throughout the investigation. Specifications are provided in Worksheets #'s 19 through 37.

10. Performance criteria (correlation coefficient) for the correlation of lead XRF data versus fixed-base laboratory data for lead. The correlation coefficient shall be greater than or equal to 0.65 and less than or equal to 1.00. These correlations will be used to translate field XRF values into equivalent laboratory lead concentrations. Correlation analyses are not anticipated to be generated for the five other metals.

Tetra Tech will collect the field samples, conduct the field XRF lead analysis, prepare selected samples for the fixed-base laboratory, and prepare the report in which data are interpreted and presented in tabular and graphical formats.

Data will become part of the Administrative Record (AR) for NSF-IH after approval of the report by USEPA Region 3 and the State of Maryland.

DEFINE THE BOUNDARIES OF THE STUDY (STEP 4)

The current site investigation is limited to the evaluation of soil and drainageway sediment at the Marine Rifle Range. An investigation of the groundwater underlying the site and the surface water of adjoining surface water bodies is beyond the scope of this investigation but may be required in the future based, in part, on the outcome of this investigation. A visual depiction of the range is provided on Map 10.1-1. Establishing the boundaries of contamination will require that both contaminated and non-contaminated soil and drainageway sediment be sampled (i.e., the perimeter of the impacted area must be established). The following items address the horizontal and vertical boundaries as well as the temporal boundaries for the study:

1. The populations of interest are the soil and sediment that may have been contaminated directly by site operations or by subsequent migration of contaminants.

2. The initial horizontal study boundary will encompass the area that, based on historical information, is most likely to have been impacted by site activities (firing lines, the target embankments, and the hillside), as shown on Map 10.1-2. Lateral expansion of this horizontal study boundary may be necessary if MC concentrations detected in the samples collected along this boundary exceed screening levels (see Step 5 Decision Rules). The following sub-areas are within the study boundary:

   - The firing lines. An area at the firing points where unburned powders and lead from primers would have accumulated. NG may have been deposited from unburned propellant.

   - The range floor immediately in front of the target embankments, which may be contaminated with stray bullet fragments. Contamination in this area is anticipated to be marginal compared to the primary impact areas.

   - The embankments below the targets. Bullets that undershoot the targets would be embedded in these embankments.

   - The hillside behind the embankments. Bullets that hit (passed through) or over shot the targets would be embedded in the hillside.

   - The "hot spot" zones. For purposes of risk screening, the site may be further divided horizontally if the analytical data suggest that a distinct “hot spot” zone(s) is present at the site and these “hot spots” are large enough to define an exposure unit for a receptor.

3. The initial vertical study boundary will be limited to 1 foot bgs because the CSM indicates that penetration of soil and sediment by MC is not generally expected to be deeper than this. Vertical expansion of this study boundary may be necessary if MC concentrations detected in the samples collected along this boundary exceed screening levels (see Step 5, Decision Rules). The direct contact soil risk screening assessment will be based on concentrations to a maximum depth of 10 feet bgs. The exposure unit represented by the exposure point concentration will be the entire volume of contaminated soil within the lateral extent of the exposure unit divided as necessary to allow separate evaluations of surface (0 to 1 foot bgs), subsurface soil (1 to 10 feet bgs), or all soil (0 to 10 feet bgs). Each embankment and the hillside will be defined as separate exposure units.

4. The temporal boundary is not a significant consideration in this study because the MC concentrations (NG and metals) are anticipated to be relatively unchanged (stable) over the course of time needed to conduct the environmental investigations and into the foreseeable future.

DEVELOP THE ANALYTIC APPROACH (STEP 5)

The decision rules for this investigation are as follows:

1. If MC concentrations in all soil and drainageway sediment samples in the initial round of sampling are less than screening or background levels, then recommend NFA; otherwise, advance to Rule 2.

2. If the XRF-measured concentrations of lead in any sample on the perimeter (vertical or horizontal) of the sampled area exceeds 300 mg/kg, “step-out” (vertically and/or horizontally) as necessary for XRF lead analysis of additional soil until the results of the XRF analysis are less than 300 mg/kg. Advance to Rule 3.

3. If any of the fixed-base laboratory concentrations for NG, antimony, arsenic, lead, copper, tin, and zinc in the initial round perimeter samples exceed screening and background levels, use professional judgment to determine the degree to which “step-out” samples (vertical or horizontal) are necessary to define the vertical and/or horizontal extent of contamination in perimeter soil or drainageway sediment. Factors considered will include the specific contaminants, metal, the magnitude of the exceedance(s), their spatial distribution, and the overall risk level. If “step-out” samples are deemed necessary by the Project Team, collect samples as necessary as part of a follow-up remedial investigation (RI) and then advance to Rules 4, 5, and 6; otherwise, advance to Rules 4, 5, and 6.

4. If MC concentrations in the receptor exposure units defined for soil or drainageway sediment represent unacceptable human health risk, proceed to an FS; otherwise, recommend NFA from a human health perspective. Unacceptable human health risk will be generally defined as cancer risk estimates exceeding $1x10^{-4}$, non-cancer risk estimates (i.e., hazard indices) exceeding 1 (on a target-organ specific basis), or receptor blood-lead levels that exceed the current USEPA benchmark (i.e., a receptor shall have no more than a 5 percent probability of blood-lead level exceeding 10 µg/dL).

5. For ecological risk screening, the maximum detected MC concentrations in surface soil and sediment will be compared to screening concentrations to determine if a contaminant is a contaminant of potential concern (COPC). Average concentrations (arithmetic means) of surface soil data or sediment data will be used in food-chain modeling. If risks for defined receptor exposure units are determined to be "unacceptable" based on an evaluation of several lines of evidence (e.g., number of exceedances of screening criteria, magnitude of the exceedances of screening criteria, spatial distribution of data, home range, background concentrations, etc.), the project team will determine the need to conduct a Baseline Ecological Risk Assessment (BERA) or consider the potential risks with respect to remedial actions. Any BERA would be conducted as part of a future RI.

6. If MC concentrations in any soil sample exceed both background levels and USEPA SSLs for groundwater protection recommend an evaluation of the need for an investigation of groundwater contamination; otherwise, do not recommend evaluating the need for a groundwater investigation be initiated on the basis of soil/sediment concentrations.

Note:

A) MC are defined specifically for this site as NG, antimony, arsenic, lead, copper, tin, and zinc.

B) Screening levels for the investigation include screening levels based on USEPA Region 3 RBCs, USEPA SSLs, the USEPA action level for lead in soil assuming a residential land use scenario (400 mg/kg), USEPA Region 3 BTAG Sediment Screening Benchmarks, USEPA Ecological SSLs, USEPA Region 3 BTAG SSLs, and USEPA Region 3 Freshwater Screening Benchmarks.

C) Evaluation criteria for "stepping-out" based on fixed-base laboratory data are as follows: Ideally, and in most cases, "step-out" samples should be collected until the perimeter sample concentrations are less than screening or background concentrations. However, professional judgment should be used to determine if such samples are needed in all cases. Professional judgment considerations include:

- The degree to which MC concentrations exceed screening or background levels. The need to collect additional samples should be carefully considered when results, for example, are within 5 percent of screening or background levels. The need to collect additional data will increase with increased number of exceedances and increased contaminant concentrations.

- The degree to which further delineation can be more efficiently and economically collected as part of follow-up RI/FS investigations (i.e., the additional sampling is unlikely to impact the decision to take or not take some type of remedial action but may be necessary to define the limits of the action).

- Considerations of the impact of risk estimations if some contaminant concentrations are not completely delineated.

D) The results of the initial round of sampling and any professional judgments made regarding the need for "step-out" samples will be reviewed with the project team.

E) Define "generally": The National Contingency Plan (NCP) discusses a generally acceptable risk range of $1x10^{-4}$ to $1x10^{-6}$. However, based on guidance presented in USEPA Risk Assessment Guidance for Superfund (RAGS) Part D, risks slightly greater than $1x10^{-4}$, may be considered acceptable (i.e., protective) if justified based on site-specific conditions including any uncertainties about the nature and extent of contamination and associated risks (USEPA, December 2001).

F) A Baseline Ecological Risk Assessment (BERA) (and the collection of data to support such an assessment) is beyond the scope of this investigation and would require development of a "follow-up" UFP-SAP and associated decision rules.

SPECIFY PERFORMANCE OR ACCEPTANCE CRITERIA (STEP 6)

The Visual Sample Plan (VSP) software tool was used to determine the number of soil samples necessary assuming that lead was the primary contaminant of concern. The following key assumptions and performance/acceptance criteria were used to determine the number of soil samples:

- For purposes of statistical analysis, the "null hypothesis" is that the site was "dirty" (i.e., the mean lead concentration at the site exceeds the action level selected for statistical analysis).

- S (Sigma) = the estimated standard deviations for areas assumed to be relatively uncontaminated (clean), contaminated (dirty), and the transition zone were set at 100 mg/kg, 8,330 mg/kg, and 830 mg/kg, respectively.

- Delta = the width of the gray region, which was set equal to 150 for clean areas, 14,600 for contaminated areas, 1,600 for the transition zone.

- Alpha = the tolerance for concluding that the site is "clean" when the site is actually "dirty". If an incorrect decision was to be made, the team prefers to incorrectly take action to remediate a clean site rather than to fail to take action at a dirty site. Making an incorrect decision becomes less tolerable as the site becomes increasingly contaminated or increasingly clean relative to the action level. Therefore, at a true mean concentration infinitesimally greater than the action level, the consequence of a decision error is not as great as, for example, if the true mean concentration was two times the action level. The tolerance for incorrectly concluding that the site mean is less than two times the action level when the true mean is greater than the action level was set at 15 percent.

- Beta = the tolerance for concluding that the site is "dirty" when the site is actually "clean" was also established considering the tolerance for incorrectly concluding that the site mean is greater than the action level when it is actually infinitesimally less than 0.9 times the action level was set to 25 percent. This Beta value is greater than the Alpha value because there is more tolerance for this type of error than for the error of not taking action when a site is dirty.

- The lower bound of the gray region (LBGR) presented in terms of a percentage of the action level: 90 percent.

- The action level of 400 mg/kg for lead which was selected for statistical analysis.

The results of the statistical analysis presented in Appendix D are that a minimum of five soil samples should be collected across each of the designated area(s) to be sampled and for which a decision must be made. The sampling design presented in Step 7 is based on this recommended number of samples and samples necessary to determine the boundaries of contamination and potential remedial areas. All new analytical data collected per the Step 7 sampling design should meet the QA criteria established in Worksheet #'s 19 through 37 and the prescribed detection limit requirements for the MC constituents of concern.

DEVELOP THE PLAN FOR OBTAINING DATA (STEP 7)

The proposed SI field data collection program for the Marine Rifle Range area is presented, to a large extent, on Map 17-1. The program assumes, based on the CSM, that the following potential depositional areas exist downrange of the firing line and to a lesser extent at the firing lines (see Map 10.1-2):

- An area at the firing points where unburned powders and lead from primers would have acuumulated.

- The range floor immediately in front of the embankments, which may be contaminated with stray bullet fragments. Contamination in this area is anticipated to be marginal versus the primary impact areas.

- The hillside, located behind the target embankments. Bullets that hit (passed through) or over shoot the targets would be embedded in the soil in the hillside.

The sampling design is roughly a grid design consisting of samples spaced along transect lines running the length of each target embankment and along the hillside behind the embankments to ensure that the decision units are well represented spatially. Contamination may also be present at the firing lines. A site walk-over will be conducted by the FOL to identify any obvious areas of fragment/lead shot accumulation. The MC sampling and analytical program for soil and drainage-way sediment samples is as follows:

- Soil
  - Thirty surface soil samples will be initially collected from the three transect lines established for each target berm area, and an additional five samples will be collected on the range floor immediately in front of each target berm. Forty-five surface soil samples will be collected from the hillside behind the target berms. All sample locations are shown on Map 17-1. Additional, discretionary surface soil samples may be collected based on visual observation of accumulated spent munitions in the area to be sampled. Emphasis will be placed on collecting samples required to delineate contaminated areas. Additional “step-out” samples will be collected at the boundaries of the area(s) to be sampled if lead concentrations greater than the field action level of 300 mg/kg are detected based on the results of the XRF field screening analysis of the initial surface soil samples. Samples will be collected using hand augers.

  - Subsurface soil samples (starting at 1 foot bgs) will also be collected at all surface soil locations but will only be subject to field XRF analysis if their respective surface soil sample lead concentrations exceed 300 mg/kg. If the subsurface sample XRF lead concentration also

exceeds 300 mg/kg, additional subsurface soil samples will be collected vertically in 1-foot intervals until lead concentrations in subsurface soil do not exceed 300 mg/kg or until collection of samples via a hand auger becomes impractical.

- Two sets of firing lines, one for each target butt, were located at 100-meter increments to a maximum of 1,000 meters at this range. Composite surface soil samples will be collected within the undeveloped areas at each 100 meter incremental firing line for NG and lead analysis using hand augers.

- Drainage-way Sediment
  - Prior to sampling, a site "walk over" will be conducted by the FOL to identify any obvious surface water drainage pathways potentially leading to adjoining surface water bodies. Discretionary drainage-way sediment samples will be collected along these pathways. Locations will be targeted for sampling based on proximity to surface soil locations with elevated lead concentrations, on the accumulation of sediment along the pathway with emphasis placed on sampling sediment traps, and on the potential for the pathway to lead to an adjoining surface water body with emphasis placed on those drainages leading to surface water bodies.

- Analytical Program
  - All soil and drainage-way sediment samples will be analyzed for lead using XRF field screening analysis. A minimum of 20 percent of these samples (but no fewer than 20 samples) will also be analyzed for the MC metals of concern at a fixed-base laboratory. The samples sent to the fixed-base laboratory will be selected to reflect the range of lead concentrations detected in the soil at the site using XRF field screening, with the majority having field XRF lead concentrations between 250 and 550 mg/kg. A correlation study (field screening versus fixed-base laboratory concentrations) may be conducted to support data interpretation for the investigation. The samples will be analyzed using SW-846 Method 6020A.

  - Four of the samples selected for submittal to the fixed-base laboratory will also undergo analysis for pH, TOC, and CEC. The samples will be biased toward locations with evidence of contamination (visual observations of spent munitions/shot and the results of XRF field screening).

  - The firing line samples will be composite samples analyzed for NG using Appendix A of SW-846 Method 8330B and lead using SW-846 Method 6020A at the fixed-base laboratory.

SAP Worksheet #11.2 -- Project Quality Objectives/Systematic Planning Process Statements for the Old Skeet and Trap Range

PROBLEM DEFINITION (STEP 1)

Worksheet 10.2 (Section 10.1.12) contains the Problem Definition.

IDENTIFY THE GOALS OF THE STUDY (STEP 2)

The primary goals of this investigation of the Old Skeet and Trap Range are as follows:

1. Collect adequate data to delineate the horizontal and vertical extent of contamination in soil, drainageway sediment, Potomac River shoreline sediment, and Potomac River sediment within the fall out pattern. If the data collected in the initial round of sampling are adequate, stop delineation; otherwise, continue sampling (i.e., “step-out” vertically and/or horizontally as necessary).

2. Determine whether MC (specifically antimony, arsenic, lead, copper, tin, zinc, and PAHs) are present in study area soil, drainageway sediment, the shoreline sediments, and Potomac River sediment and also if NG and lead are present in the sol in the area of the firing line at concentrations that represent unacceptable human health or ecological risk. If either of these risks are unacceptable, evaluate the need to proceed to an FS; otherwise, recommend NFA.

3. Determine whether MC (specifically NG, antimony, arsenic, lead, copper, tin, zinc, and PAHs) are present in study area soil at concentrations that represent a threat to groundwater quality. If MC concentrations in soil represent a threat to groundwater quality, recommend an environmental investigation of groundwater contamination; otherwise, do not initiate a groundwater investigation on the basis of soil contamination levels.

4. Update the CSM using field reconnaissance survey data and initial hazard and risk screening results.

All data collected for this project are expected to be transferable to support the following actions:

1. Update the Navy CTC estimate
2. Complete the site prioritization protocol

IDENTIFY INFORMATION INPUTS (STEP 3)

Data required for making the decisions which include the following:

1. A comprehensive listing of the relevant, medium-specific RBSLs and potential chemical-specific ARARs for the MC of concern (specifically NG, antimony, arsenic, lead, copper, tin, zinc, and PAHs). Screening levels based on USEPA Region 3 RBCs, the USEPA action level for lead in soil assuming a residential land use scenario (400 mg/kg), USEPA SSLs, USEPA Region 3 BTAG Sediment Screening Benchmarks, USEPA Ecological SSLs, USEPA Region 3 BTAG SSLs, and USEPA Region 3 Freshwater Screening Benchmarks.

2. Laboratory analytical data characterizing concentrations of the NG, the six MC metals of interest and PAHs in soil [surface soil (0 to 1 foot bgs) and subsurface soil (greater than 1 foot bgs), as necessary], drainageway/river shoreline sediment, and river sediment samples (0 to 6 inches). Soil and sediment samples will be analyzed for NG using Appendix A of SW-846 Method 8330B, MC metals using SW-846 Method 6020A and for PAHs using SW-846 Method 8270C. MC concentrations in soil and sediment will be compared to site screening levels and background concentrations. These data are necessary to conduct the human health and ecological risk screening assessments. Laboratory quantitation limits for the proposed analytical methods should not exceed the aforementioned screening levels and ARARs. Practical quantitation limits should not exceed risk-based project action levels (i.e., levels associated with a $1x10^{-4}$ cumulative cancer risk level, a cumulative hazard index of 1, or the USEPA action level for lead [400 mg/kg]). Based on knowledge of lead concentrations in similar soils, native soil and sediment lead concentrations are expected to be less than 50 mg/kg. Because of this, contaminated areas (e.g., with lead concentrations exceeding 400 mg/kg) are expected to be well delineated, with steep lead concentration gradients between clean and contaminated areas.

3. Because lead is the primary contaminant and other metals are expected to be collocated with lead, lead can be used as a marker for contamination. For delineation of lead contamination, the use of both field and fixed-base laboratory analyses is anticipated. This will provide a high degree of spatial coverage for relatively low cost. XRF analysis will be used for lead analysis in the field. An XRF field action level of 300 mg/kg will be used as the discriminator between contaminated and uncontaminated locations. The 300 mg/kg level corresponds to the USEPA action level of 400 mg/kg reduced by a 25 percent margin of error to minimize the potential that contaminated areas go undetected. The margin of error was selected based on recent experiences with field XRF analyses at other small arms ranges.

4. Laboratory analytical data characterizing soil and sediment pH, TOC, and CEC characteristics. These data are necessary for the evaluation of MC environmental fate and transport.

5. Background soil data suitable for site soil-versus-background soil data comparisons for the six metals of interest for use in site versus background comparisons.

6. Field identification/classification of soil and sediment types (i.e., lithology and USCS soil classification for grain size, color, plasticity, etc.). Worksheet #21 contains relevant SOPs.

7. Physical characterization of the major surface water drainage pathways (e.g., number, width, depth, physical characteristics of sediments, etc.). Worksheet #21 contains relevant SOPs.

8. Field investigation specifications (e.g., SOPs for sample collection).

9. QA/QC specifications and the results of the QA/QC reviews (e.g., data validation) conducted throughout the investigation. Specifications are provided in Worksheet #'s 19 through 37.

10. Performance criteria (correlation coefficients) for correlation of the lead XRF data versus fixed-base laboratory data for lead: The correlation coefficient shall be greater than or equal to 0.65 and less than or equal to 1.00. These correlations will be used to translate field XRF values into the equivalent laboratory lead concentration. Correlation analyses are not anticipated to be generated for the five other metals.

Tetra Tech will collect the field samples, conduct the field XRF lead analysis, prepare selected samples for the fixed-base laboratory, and prepare the report in which data are interpreted and presented in tabular and graphical formats.

Data will become part of the AR for NSF-IH after approval of the report by USEPA Region 3 and the State of Maryland.

DEFINE THE BOUNDARIES OF THE STUDY (STEP 4)

The current site investigation is limited to the evaluation of soil, drainageway/river shoreline sediment, and Potomac River sediment at the Old Skeet and Trap Range. As noted above, an investigation of the groundwater underlying the site is beyond the scope of this investigation, but may be required in the

future based, in part, on the outcome of this investigation. A visual depiction of the range is provided on Map 10.2-1. Establishing the nature and extent of contamination will require that both contaminated and non-contaminated soil, drainageway/river shoreline sediment, and Potomac River sediment be sampled (i.e., the perimeters of the impacted areas must be established). The following items address the horizontal and vertical boundaries as well as the temporal boundaries for the study:

1. The population of interest includes soil and sediment that may have been contaminated directly by site operations or by subsequent migration of contaminations.

2. The initial horizontal study boundary will encompass the area that, based on historical information, is most likely to have been impacted by site activities and is shown on Map 10.2-2. Lateral expansion of this horizontal study boundary may be necessary if MC concentrations detected in the samples collected along this boundary exceed screening levels (see Step 5, Decision Rules). The study boundary will include the following subzones:

    - The firing line. An area at the firing point where unburned powders and lead from primers would have accumulated. NG may have been deposited from unburned propellant.

    - An area downrange of the firing line where clay target fragments (i.e., fragments of targets actually hit by shot) would have accumulated (spent lead shot would also be present). This area likely includes the river shoreline and river adjoining the site;

    - An area downrange of the clay target fragments where “used/weathered targets” (not hit by shotgun shells [i.e., missed targets]) would have accumulated. As shown on Map 10.2-3, many of these used targets may have fallen into the Potomac River;

    - An area downrange of the firing line, clay target fragments, and used/weathered target fragments where the greatest concentrations of lead shot would have accumulated.

    - The "hot spot" zones. For purposes of risk assessment, the site may be further divided horizontally if the analytical data suggest that a distinct “hot spot” zone(s) is present at the site and these “hot spots” are large enough to define an exposure unit for a receptor.

3. The initial vertical study boundary will be limited to 1 foot bgs for soil and 6 inches for sediment because the CSM indicates that penetration of soil and sediment by MC is not generally expected to

be deeper than this. Vertical expansion of this vertical study boundary may be necessary if MC concentrations detected in the samples collected along this boundary exceed screening levels (see Step 5, Decision Rules). The direct contact soil risk screening assessment will be based on concentrations to a maximum depth of 10 feet bgs for soil. The exposure unit represented by the exposure point concentration will be the entire volume of contaminated soil divided as necessary to allow separate evaluations of surface (0 to 1 foot bgs), subsurface soil (1 to 10 feet bgs), or all soil (0 to 10 feet bgs).

4. The temporal boundary is not a significant consideration in this study because MC concentrations (NG, metals and PAHs) are anticipated to be relatively unchanged (stable) over the course of time needed to conduct the environmental investigations.

DEVELOP THE ANALYTIC APPROACH (STEP 5)

Decision rules for this investigation are as follows:

1. If MC concentrations in all soil and drainageway/river shoreline and river sediment samples in the initial round of sampling are less than screening or background levels, then recommend NFA; otherwise, advance to Rule 2.

2. If the XRF-measured concentrations of lead in any sample on the perimeter (vertical or horizontal) of the sampled area exceeds 300 mg/kg, “step-out” (vertically and/or horizontally) as necessary for XRF lead analysis of additional soil until the results of the XRF analysis are less than 300 mg/kg. Advance to Rule 3.

3. If any of the fixed-base laboratory concentrations for NG, antimony, arsenic, lead, copper, tin, zinc, or PAHs in the initial round perimeter samples exceed screening or background levels, use professional judgment to determine the degree to which “step-out” samples (vertical or horizontal) are necessary to define the vertical and/or horizontal extent of contamination in perimeter soil or drainageway/river shoreline sediment. Factors considered will include the specific contaminants, the magnitude of the exceedance, spatial distribution, and overall risk levels. If “step-out” samples are deemed necessary by the Project team, collect samples as necessary as part of a follow-up RI and then advance to Rules 4, 5, and 6; otherwise, advance to Rules 4, 5, and 6.

4. If MC concentrations in the receptor exposure units defined for soil or drainageway/river shoreline sediment represent unacceptable human health risk, proceed to an FS; otherwise, recommend NFA

from a human health perspective. Unacceptable human health risk will be generally defined as cancer risk estimates exceeding 1E-04, non-cancer risk estimates (i.e., hazard indices) exceeding 1 (on a target-organ specific basis), or receptor blood-lead levels that exceed the current USEPA benchmark (i.e., a receptor shall have no more than a 5 percent probability of blood lead levels exceeding 10 µg/dL).

5. For ecological risk assessments, the maximum detected MC concentrations in surface soil and sediment will be compared to screening concentrations to determine if a contaminant is a COPC. Average concentrations (arithmetic means) of surface soil data and sediment data will be used in food-chain modeling. If risks for defined receptor exposure units are determined to be "unacceptable" based on an evaluation of several lines of evidence (e.g., number of exceedances of screening criteria, magnitude of the exceedances of screening criteria, spatial distribution of data, home range, background concentrations, etc.), the project team will determine the need to conduct a BERA or consider the potential risks with respect to remedial actions. Any BERA would be conducted as part of a future RI.

6. If MC concentrations in any soil or drainageway, shoreline, or river sediment sample, exceed both background levels and USEPA SSLs for groundwater protection, recommend an evaluation of the need for an investigation of groundwater contamination; otherwise, do not recommend evaluating the need for that a groundwater investigation be initiated on the basis of soil/sediment concentrations.

Note:

A) MC are defined specifically for this site as NG, antimony, arsenic, lead, copper, tin, zinc, and PAHs.

B) Screening levels for the investigation: Screening levels are based on USEPA Region 3 RBCs, USEPA SSLs, the USEPA action level for lead in soil assuming a residential land use scenario (400 mg/kg), USEPA Region 3 BTAG Sediment Screening Benchmarks, USEPA Ecological SSLs, USEPA Region 3 BTAG SSLs, and USEPA Region 3 Freshwater Screening Benchmarks.

C) Evaluation criteria for "stepping out" based on fixed-base laboratory data are as follows: Ideally, and in most cases, "step-out" samples should be collected until the perimeter sample concentrations are less than screening or background concentrations. However, professional judgment should be used to determine if such samples are needed in all cases. Professional judgment considerations include:

- The degree to which MC concentrations exceed screening or background levels. The need to collect additional samples should be carefully considered when results, for example, are within 5 percent of screening or background levels. The need to collect additional data will increase with increased number of exceedances and increased contaminant concentrations.

- The degree to which further delineation can be more efficiently and economically collected as part of follow-up RI/FS investigations (i.e., the additional sampling is unlikely to impact the decision to take or not take some type of remedial action but may be necessary to define the limits of the action).

- Considerations of the impact of risk estimations if some contaminant concentrations are not completely delineated.

D) The results of the initial round of sampling and any professional judgments made regarding the need for "step-out" samples will be reviewed with the project team.

E) Define "generally": The NCP discusses a generally acceptable risk range of $1x10^{-4}$ to $1x10^{-6}$. However, based on guidance presented in USEPA RAGS Part D, risks slightly greater than $1x10^{-4}$, may be considered acceptable (i.e., protective) if justified based on site-specific conditions including any uncertainties about the nature and extent of contamination and associated risks (USEPA, December 2001).

F) A BERA (and the collection of data to support such an assessment) is beyond the scope of this investigation and would require development of a "follow-up" UFP-SAP and associated decision rules.

SPECIFY PERFORMANCE OR ACCEPTANCE CRITERIA (STEP 6)

The VSP software tool was used to determine the number of soil samples necessary assuming that lead was the primary contaminant of concern. The following key assumptions and performance/acceptance criteria were used to determine the number of soil samples:

- For purposes of statistical analysis, the "null hypothesis" is that the site was "dirty" (i.e., the mean lead concentration at the site exceeds the action level selected for statistical analysis).

- S (Sigma) = the estimated standard deviations for areas assumed to be relatively uncontaminated (clean), contaminated (dirty), and the transition zone were set at 100 mg/kg, 8,330 mg/kg, and 830 mg/kg, respectively.

- Delta = the width of the gray region, which was set equal to 150 for clean areas, 14,600 for contaminated areas, 1,600 for the transition zone.

- Alpha = the tolerance for concluding that the site is "clean" when the site is actually "dirty". If an incorrect decision was to be made, the team prefers to incorrectly take action to remediate a clean site rather than to fail to take action at a dirty site. Making an incorrect decision becomes less tolerable as the site becomes increasingly contaminated or increasingly clean relative to the action level. Therefore, at a true mean concentration infinitesimally greater than the action level, the consequence of a decision error is not as great as, for example, if the true mean concentration was two times the action level. The tolerance for incorrectly concluding that the site mean is less than two times the action level when the true mean is greater than the action level was set at 15 percent.

- Beta = the tolerance for concluding that the site is "dirty" when the site is actually "clean" was also established considering the tolerance for incorrectly concluding that the site mean is greater than the action level when it is actually infinitesimally less than 0.9 times the action level was set to 25 percent. This Beta value is greater than the Alpha value because there is more tolerance for this type of error than for the error of not taking action when a site is dirty.

- The LBGR presented in terms of a percentage of the action level: 90 percent.

- The action level of 400 mg/kg for lead which was selected for statistical analysis.

The results of the statistical analysis presented in Appendix D are that a minimum of five soil samples should be collected across each of the designated area(s) to be sampled and for which a decision must be made. The sampling design presented in Step 7 is based on this recommended number of samples and samples necessary to determine the boundaries of contamination and potential remedial areas. All new analytical data collected per the Step 7 sampling design should meet the QA criteria established in Worksheet #'s 19 through 37 and the prescribed detection limit requirements for the MC constituents of concern.

DEVELOP THE PLAN FOR OBTAINING DATA (STEP 7)

The proposed SI field data collection program for the Old Skeet and Trap Range area is presented, to a large extent, on Map 17-2. The program assumes, based on the CSM, that the following potential depositional areas exist downrange of the firing line and to a lesser extent at the firing line:

- An area at the firing points where unburned powders and lead from primers would have accumulated.

- An area where clay "target fragments" (i.e., fragments of targets actually hit by shot) would have accumulated (spent lead shot would also be present). This area likely includes the river shoreline and river adjoining the site.

- An area where used/weathered targets (not hit by shotgun shells [i.e., missed targets]) would have accumulated. As shown on Map 10.2-3, many of these used targets may have fallen into the Potomac River.

- An area where the clay target fragments, and the used/weathered target fragments where the greatest concentrations of lead shot would accumulate.

The primary shot/skeet fragment accumulation area for the Old Skeet and Trap Range includes areas along the Potomac River shoreline and into the Potomac River. Much of the maximum lead shot fall accumulation zone is actually in the Potomac River (as noted above, an area "out of scope" for the current investigation). As noted in Step 4, the horizontal and vertical boundaries of the soil/sediment drainageway/river shoreline areas to be sampled will encompass all areas significantly impacted by site activities.

The sampling design consists of samples spaced along transect lines extending from the shooters' positions to the maximum shot fall point in the Potomac River to ensure that the decision units are well represented spatially. Contamination may also be present at the firing line. A site walk-over will be conducted by the FOL to identify any obvious areas of fragmented/lead shot accumulation. The MC sampling and analytical program for soil, drainageway, shoreline, and river sediment samples is as follows:

- Soil
  - Fifty-six surface soil samples (0 to 1 foot bgs) will be initially collected from the transect lines established within the area to be sampled, as shown on Map 17.2. Additionally, a site "walkover"

will be conducted by the FOL to identify any obvious areas of fragment/lead shot accumulation. Additional discretionary surface soil samples may be collected based on visual observation of accumulated fragments or shot in the area to be sampled. Emphasis will be placed on collecting samples required to delineate contaminated areas. Additional "step-out" samples will also be collected at the boundaries of the areas to be sampled if lead concentrations greater than the field action level of 300 mg/kg are detected based on the results of the XRF field screening analysis of the initial surface soil samples.

- Subsurface soil samples (starting at 1 foot bgs) will also be collected at all surface soil locations but will only be subject to field XRF analysis if their respective surface soil sample lead concentrations exceed 300 mg/kg. If the subsurface sample XRF lead concentration also exceeds 300 mg/kg, additional subsurface soil samples will then be collected vertically in 1-foot intervals until lead concentrations in subsurface soil do not exceed 300 mg/kg or until collection of samples via a hand auger becomes impractical.

- Two composite surface soil samples will be collected from the area at the firing line for NG and lead analysis using hand augers.

- Drainage-way and River Shoreline Sediment
  - Prior to sampling, a site "walk over" will be conducted by the FOL to identify any obvious surface water drainage pathways (e.g., the swale area in the eastern portion of the range) potentially leading to adjoining surface water bodies such as the Potomac River. Discretionary drainageway sediment samples will be collected from each of these pathways. Locations will be targeted for sampling based on proximity to surface soil locations with elevated lead concentrations, on the accumulation of sediments along the pathway with emphasis placed on sampling sediment traps, and on the potential for the pathway to lead to a surface water body such as the Potomac River. Additionally, nine sediment samples will be collected along the shoreline as shown on Map 17.2.

  - Twenty-five sediment samples will be collected from within the Potomac River at a depth of 0 to 6 inches along three transect lines spaced between the shore line and the maximum shot fall line.

- Analytical Program
  - All soil drainageway/shoreline and river sediment samples will be analyzed for lead using XRF field screening analysis. A minimum of 20 of these samples (but no fewer than 20 samples) will also be analyzed for the MC metals of concern at a fixed-base laboratory. The samples sent to the fixed-base laboratory will be selected to reflect the range of lead concentrations detected in

the soil/sediment at the site using XRF field screening, with the majority having field XRF lead concentrations between 250 and 550 mg/kg. A correlation study (field screening versus fixed-base laboratory concentrations) may be conducted to support data interpretation for the investigation. The samples will be analyzed for metals using SW-846 Method 6020A and for PAHs using SW-846 Method 8270C.

- The two firing line samples will be composite samples analyzed for NG using Appendix A of SW-846 Method 8330B and lead using SW-846 Method 6020A at the fixed-base laboratory.

- Sediment samples and soil samples collected along the transect lines within the downrange area potentially contaminated with spent targets and target fragments (and selected for MC metals analysis at a fixed-base laboratory) and any discretionary samples collected from those areas will also be analyzed for PAHs. The samples will be analyzed for metals using SW-846 Method 6020A and for PAHs using SW-846 Method 8270C.

- Four of the samples selected for submittal to the fixed-base laboratory will also undergo analysis for pH, TOC, and CEC. The samples will be biased toward locations with evidence of contamination (visual observations of fragments/shot and the results of field screening).

SAP Worksheet #11.3 -- Project Quality Objectives/Systematic Planning Process Statements for the Roach Road Rifle Range

PROBLEM DEFINITION (STEP 1)

Worksheet 10.3 (Section 10.3.12) contains the Problem Definition for the Roach Road Rifle Range.

IDENTIFY THE GOALS OF THE STUDY (STEP 2)

The primary goals of this investigation of the Roach Road Rifle Range are as follows:

1. Collect adequate data to delineate the horizontal and vertical extent of soil and drainageway/creek sediment contamination. If the data collected in the initial round of sampling are adequate, stop delineation; otherwise, continue sampling (i.e., “step-out” vertically and/or horizontally as necessary).

2. Determine whether MC (specifically antimony, arsenic, lead, copper, tin, and zinc) are present in study area soil and drainageway/creek sediment and also if NG and lead are present in the soil in the area of the firing line at concentrations that represent unacceptable human health or ecological risk. If either of these risks are unacceptable, evaluate the need to proceed to a FS; otherwise, recommend NFA.

3. Determine whether MC (specifically NG, antimony, arsenic, lead, copper, tin, and zinc) are present in study area soil at concentrations that represent a threat to groundwater quality. If MC concentrations in soil do represent a threat to groundwater quality, recommend an environmental investigation of groundwater contamination; otherwise, do not initiate a groundwater investigation on the basis of soil contamination levels.

4. Update the CSM using field reconnaissance survey data and initial hazard and risk screening results.

All data collected for this project are expected to be transferable to support the following actions:

1. Update the Navy CTC estimate
2. Complete the site prioritization protocol

IDENTIFY INFORMATION INPUTS (STEP 3)

Data required for making decisions include the following:

1. A comprehensive listing of the relevant, medium-specific RBSLs and potential chemical-specific ARARs for the MC of concern (specifically NG, antimony, arsenic, lead, copper, tin, and zinc). Screening levels based on USEPA Region 3 RBCs, the USEPA action level for lead in soil assuming a residential land use scenario (400 mg/kg), USEPA SSLs, USEPA Region 3 BTAG Sediment Screening Benchmarks, USEPA Ecological SSLs, USEPA Region 3 BTAG SSLs, and USEPA Region 3 Freshwater Screening Benchmarks.

2. Laboratory analytical data characterizing concentrations of the NG and the six MC metals of interest in soil [surface soil (0 to 1 foot bgs) and subsurface soil (greater than 1 foot bgs), as necessary] and drainageway/creek sediment samples (0 to 6 inches bgs). Soil and sediment samples will be analyzed for NG using Appendix A of SW-846 Method 8330B and specified MC metals using SW-846 Method 6020A. MC concentrations in soil and sediment will be compared to site screening levels and background concentrations. These data are necessary to conduct the human health and ecological risk screening assessments. Laboratory quantitation limits for the proposed analytical methods should not exceed the aforementioned screening levels and ARARs. Practical quantitation limits should not exceed risk-based project action levels (i.e., levels associated with a $1x10^{-4}$ cumulative cancer risk level, a cumulative hazard index of 1, or the USEPA action level for lead [400 mg/kg]). Based on knowledge of lead concentrations in similar soils, native soil and sediment lead concentrations are expected to be less than 50 mg/kg. Because of this, contaminated areas (e.g., with concentrations exceeding 400 mg/kg) are expected to be well delineated, with steep lead concentration gradients between clean and contaminated areas.

3. Because lead is the primary contaminant and other metals are expected to be collocated with lead, lead can be used as a marker for contamination. For delineation of lead contamination, the use of both field and laboratory analyses is anticipated. This will provide a high degree of spatial coverage for relatively low cost. XRF analysis will be used for lead analysis in the field. An XRF field action level of 300 mg/kg will be used as the discriminator between contaminated and uncontaminated locations. The 300 mg/kg corresponds to the USEPA action level of 400 mg/kg reduced by a 25 percent margin of error to minimize the potential that contaminated areas go undetected. The margin of error was selected based on recent experiences with field XRF analyses at other small arms ranges.

4. Laboratory analytical data characterizing soil and sediment pH, TOC, and CEC characteristics. These data are necessary for the evaluation of MC environmental fate and transport.

5. Background soil data suitable for site soil-versus-background soil data comparisons for the six metals of interest for use in site versus background comparisons.

6. Field identification/classification of soil and sediment types (i.e., lithology and USCS soil classification for grain size, color, plasticity, etc.). Worksheet #21 contains relevant SOPs.

7. Physical characterization of the major surface water drainage pathways (e.g., number, width, depth, physical characteristics of sediments, etc.). Worksheet #21 contains relevant SOPs.

8. Field investigation specifications (e.g., SOPs for sample collection).

9. QA/QC specifications and the results of QA/QC reviews (e.g., data validation) conducted throughout the investigation. Specifications are provided in Worksheet #'s 19 through 37.

10. Performance criteria (correlation coefficients) for correlation of the lead XRF data to fixed-base laboratory data for lead. The correlation coefficient shall be greater than or equal to 0.65 and less than or equal to 1.00. These correlations will be used to translate field XRF values into equivalent laboratory lead concentrations. Correlation analyses are not anticipated to be generated for the five other metals.

Tetra Tech will collect the field samples, conduct the field XRF lead analysis, prepare selected samples for the fixed-base laboratory, and prepare the report in which data are interpreted and presented in tabular and graphical formats.

Data will become part of the AR for NSF-IH after approval of the report by USEPA Region 3 and the State of Maryland.

DEFINE THE BOUNDARIES OF THE STUDY (STEP 4)

The current site investigation is limited to the evaluation of soil and drainageway sediments at the Roach Road Rifle Range. An investigation of groundwater underlying the site and the surface water of adjoining surface water bodies is beyond the scope of this investigation but may be required in the future based, in part, on the outcome of this investigation. A visual depiction of the range is provided on Map 10.3.1. Establishing the boundaries of contamination will require that both contaminated and non-contaminated soil and drainageway sediment be sampled (i.e., the perimeter of the impacted area must be established).

The following items address the horizontal and vertical boundaries as well as the temporal boundaries for the study:

1. The populations of interest include soil and sediment that may have been contaminated directly by site operations or by subsequent migration of contaminants.

2. The initial horizontal study boundary will encompass the area that, based on historical information, is most likely have been impacted by site activities (the impact hillside) as shown on Map 10.3.2. Lateral expansion of this horizontal study boundary may be necessary if MC concentrations detected in the samples collected along this boundary exceed screening levels (see Step 5, Decision Rules). The following sub-zones are within the study boundary:

   - The firing lines. An area at the firing point where unburned powders and lead from primers would have accumulated. NG may have been deposited form unburned propellant.

   - The study boundary will include the range floor immediately in front of the hillside, which may be contaminated with residues from stray bullet fragments. Contamination in this area is anticipated to be marginal compared the primary impact areas.

   - The primary impact berm (i.e., hillside) may have two sub-zones. Bullets that were shot below the range targets would likely be dispersed in the lower sections of the hillside, closer to the toe of the embankment (i.e., a dispersed metal impact zone). Bullets that successfully hit the targets most likely would continue through the targets and primarily strike a sub-area of the embankment (i.e., a concentrated metal impact zone) (see Map 10.3.2).

   - For purposes of risk assessment, the site may be divided horizontally if the analytical data suggest that a distinct “hot spot” zone(s) is present at the site and these “hot spots” are large enough to define an exposure unit for a receptor.

3. The initial vertical study boundary will be limited to 1 foot bgs because the CSM indicates that penetration of soil and sediment by bullets is not generally expected to be deeper than this. Vertical expansion of this study boundary may be necessary if MC concentrations detected in the samples collected along this boundary exceed screening levels (see Step 5, Decision Rules). The direct contact soil risk screening assessment will be based on concentrations to a maximum depth of 10 feet bgs. The exposure unit represented by the exposure point concentration will be the entire

volume of contaminated soil within the lateral extent of the exposure unit divided as necessary to allow separate evaluations of surface (0 to 1 foot bgs), subsurface soil (1 to 10 feet bgs), or all soil (0 to 10 feet bgs).

4. The temporal boundary is not a significant consideration in this study because MC concentrations (NG and metals) are anticipated to be relatively unchanged (stable) over the course of time needed to conduct the environmental investigations and into the foreseeable future.

DEVELOP THE ANALYTIC APPROACH (STEP 5)

Decision rules for this investigation are as follows:

1. If MC concentrations in all soil and drainageway sediment samples in the initial round of sampling are less than screening or background levels, recommend NFA; otherwise, advance to Rule 2.

2. If the XRF-measured concentrations of lead in any sample on the perimeter (vertical or horizontal) of the sampled area exceeds 300 mg/kg, “step-out” (vertically and/or horizontally) as necessary for XRF lead analysis of additional soil until the results of the XRF analysis are less than 300 mg/kg. Advance to Rule 3.

3. If any of the fixed-base laboratory concentrations for NG, antimony, arsenic, lead, copper, tin, and zinc in the initial round perimeter samples exceed screening or background levels, use professional judgment to determine the degree to which “step-out” samples (vertical or horizontal) are necessary to define the vertical and/or horizontal extent of contamination in perimeter soil or drainageway sediment. Factors considered will include the specific contaminants, the magnitude and number of the exceedances, their spatial distribution, and overall risk levels. If “step-out” samples are deemed necessary by the Project Team, collect samples as necessary as part of a follow-up RI and then advance to Rules 4, 5, and 6; otherwise, advance to Rules 4, 5, and 6.

4. If MC concentrations in the receptor exposure units defined for soil or drainageway sediment represent unacceptable human health risk, proceed to an FS; otherwise, recommend NFA from a human health perspective. Unacceptable human health risk will be generally defined as cancer risk estimates exceeding 1E-04, non-cancer risk estimates (i.e., hazard indices) exceeding 1 (on a target-organ specific basis), or receptor blood-lead levels that exceed the current USEPA benchmark (i.e., a receptor shall have no more than a 5 percent probability of blood-lead level exceeding 10 µg/dL).

5. For ecological risk assessments, the maximum detected MC concentrations in surface soil and sediment will be compared to screening concentrations to determine if a contaminant is a COPC. Average concentrations (arithmetic means) of surface soil or sediment data will be used in food-chain modeling. If risks for defined receptor exposure units are determined to be "unacceptable" based on an evaluation of several lines of evidence (e.g., number of exceedances of screening criteria, magnitude of the exceedances of screening criteria, spatial distribution of data, home range, background concentrations, etc.), the project team will determine the need to conduct a BERA or consider the potential risks with respect to remedial actions. Any BERA would be conducted as part of a future RI.

6. If MC concentrations in any soil sample exceed both background levels and USEPA SSLs for groundwater protection, recommend an evaluation of the need for an investigation of groundwater contamination; otherwise, do not recommend evaluating the need for a groundwater investigation be initiated on the basis of soil/sediment concentrations.

Note:

A) MC are defined specifically for this site as NG, antimony, arsenic, lead, copper, tin, and zinc.

B) Screening levels for the investigation: Screening levels based on USEPA Region 3 RBCs, USEPA SSLs, USEPA action level for lead in soil assuming a residential land use scenario (400 mg/kg), USEPA Region 3 BTAG Sediment Screening Benchmarks, USEPA Ecological SSLs, USEPA Region 3 BTAG SSLs, and USEPA Region 3 Freshwater Screening Benchmarks.

C) Evaluation criteria for "stepping out" based on fixed-base laboratory data are as follows: Ideally, and in most cases, "step-out" samples should be collected until perimeter sample concentrations are less than screening or background concentrations. However, professional judgment should be used to determine if such samples are needed in all cases. Professional judgment considerations include:

- The degree to which MC concentrations exceed screening or background levels. The need to collect additional samples should be carefully reconsidered when results, for example, are within 5 percent of screening or background levels. The need to collect additional data will increase with increased numbers of exceedances and increased contaminant concentrations.

- The degree to which further delineation can be more efficiently and economically collected as part of follow-up RI/FS investigations (i.e., the additional sampling is unlikely to impact the decision to

take or not take some type of remedial action but may be necessary to define the limits of the action).

- Considerations of the impact of risk estimation if some contaminant concentrations are not completely delineated.

D) The result of the initial round of sampling and any "professional judgments" made regarding the need for "step-out" samples will be reviewed with the project team.

E) Definition of "generally": The NCP discusses a generally acceptable risk range of $1x10^{-4}$ to $1x10^{-6}$. However, based on guidance presented in USEPA RAGS Part D, risks slightly greater than $1x10^{-4}$ may be considered acceptable (i.e., protective) if justified based on site-specific conditions including any uncertainties about the nature and extent of contamination and associated risks (USEPA, December 2001).

F) A BERA (and the collection of data to support such an assessment) is beyond the scope of this investigation and would require development of a "follow-up" UFP-SAP and associated decision rules.

SPECIFY PERFORMANCE OR ACCEPTANCE CRITERIA (STEP 6)

The VSP software tool was used to determine the number of soil samples necessary assuming that lead was the primary contaminant of concern. The following key assumptions and performance/acceptance criteria were used to determine the number of soil samples:

- For purposes of statistical analysis, the "null hypothesis" is that the site was "dirty" (i.e., the mean lead concentration at the site exceeds the action level selected for statistical analysis).

- S (Sigma) = the estimated standard deviations for areas assumed to be relatively uncontaminated (clean), contaminated (dirty), and the transition zone were set at 100 mg/kg, 8,330 mg/kg, and 830 mg/kg, respectively.

- Delta = the width of the gray region, which was set equal to 150 for clean areas, 14,600 for contaminated areas, 1,600 for the transition zone.

- Alpha = the tolerance for concluding that the site is "clean" when the site is actually "dirty". If an incorrect decision was to be made, the team prefers to incorrectly take action to remediate a clean

site rather than to fail to take action at a dirty site. Making an incorrect decision becomes less tolerable as the site becomes increasingly contaminated or increasingly clean relative to the action level. Therefore, at a true mean concentration infinitesimally greater than the action level, the consequence of a decision error is not as great as, for example, if the true mean concentration was two times the action level. The tolerance for incorrectly concluding that the site mean is less than two times the action level when the true mean is greater than the action level was set at 15 percent.

- Beta = the tolerance for concluding that the site is "dirty" when the site is actually "clean" was also established considering the tolerance for incorrectly concluding that the site mean is greater than the action level when it is actually infinitesimally less than 0.9 times the action level was set to 25 percent. This Beta value is greater than the Alpha value because there is more tolerance for this type of error than for the error of not taking action when a site is dirty.

- The LBGR presented in terms of a percentage of the action level: 90 percent.

- The action level of 400 mg/kg for lead which was selected for statistical analysis.

The results of the statistical analysis presented in Appendix D are that a minimum of five soil samples should be collected across each of the designated area(s) to be sampled and for which a decision must be made. The sampling design presented in Step 7 is based on this recommended number of samples and samples necessary to determine the boundaries of contamination and potential remedial areas. All new analytical data collected per the Step 7 sampling design should meet the QA criteria established in Worksheet #'s 19 through 37 and the prescribed detection limit requirements for the MC constituents of concern.

DEVELOP THE PLAN FOR OBTAINING DATA (STEP 7)

The proposed SI field data collection program for the Roach Road Rifle Range area is presented, to a large extent, on Map 17-3 and is outlined below. The program assumes that the following depositional areas exist downrange of the firing line and to a lesser extent at the firing line:

- An area at the firing line where unburned powders and lead from primers would have accumulated.

- The range floor immediately in front of the hillside, which may be contaminated with stray bullet fragments. Contamination in this area is anticipated to be marginal compared to the primary impact areas.

- The primary impact berm (i.e., the hillside). Bullets that were shot below the range targets would likely be dispersed in the lower sections of the embankment, closer to the toe of the embankment (i.e., a dispersed metal impact zone). Bullets that successfully hit the targets most likely would continue through the targets and primarily strike a sub-area of the embankment (i.e., a concentrated metal impact zone.) (see Map 10.3-2).

Range fire that overshoot targets would have impacted the soil above and behind the targets. However, it is assumed that most of the spent munitions would most likely have fallen within the area of the impact berm. As noted in Step 4, the horizontal and vertical boundaries of the soil/sediment drainageway areas to be sampled will encompass all areas significantly impacted by site activities. A complicating factor in any sampling design is the presence of rubble/fill materials near present-day Roach Road. These fill materials may obscure/cover site features including the original floor of the range.

The sampling design consists of samples spaced along transect lines extending the length of the target embankment and on the hillside behind the embankments to ensure that the decision units are well represented spatially. Contamination may also be present at the firing line. A site walk-over will be conducted by the FOL to identify any obvious areas of fragmented/lead shot accumulation. The MC sampling and analytical program for soil and drainage-way sediment samples is as follows:

- Soil
  - Thirty-two surface soil samples will be collected from the transect lines established in locations of the former target areas and the hillside that lies directly behind the former target areas. All sample locations are shown on Map 17-3. Additional, discretionary surface soil samples may be collected based on visual observation of accumulated spent munitions in the area to be sampled. Emphasis will be placed on collecting samples required to delineate contaminated areas. Additional “step-out” samples will also be collected at the boundaries of the area(s) to be sampled if lead concentrations greater than the field action level of 300 mg/kg are detected based on the results of XRF field screening analysis of the initial surface soil samples. Samples will be collected using hand augers.

  - Subsurface soil samples (starting at 1 foot bgs) will also be collected at all surface soil locations but will only be subject to field XRF analysis if their respective surface soil sample lead concentrations exceed 300 mg/kg. If the subsurface sample XRF lead concentration also exceeds 300 mg/kg, additional subsurface soil samples will then be collected vertically in one-foot

intervals until lead concentrations in subsurface soil do not exceed 300 mg/kg or until collection of samples via a hand auger becomes impractical.

- Two composite surface soil samples will be collected from the area at the firing line for NG and lead analysis using hand augers.

- Drainageway Sediment
  - Prior to sampling, a site "walk over" will be conducted by the FOL to identify any obvious surface water drainage pathways potentially leading to adjoining surface water bodies. Discretionary drainageway sediment samples will be collected from each of these pathways. Locations will be targeted for sampling based on proximity to surface soil locations with elevated lead concentrations, on the accumulation of sediments along the pathway with emphasis placed on sampling sediment traps, and on the potential for the pathway to lead to an adjoining surface water body with emphasis placed on those drainages leading to surface water bodies.

- Analytical Program
  - All soil and drainageway sediment samples will be analyzed for lead in the field using XRF field screening analysis. A minimum of 20 percent of these samples (but no fewer than 20 samples) will also be analyzed for the MC metals of concern at a fixed-base laboratory. The samples sent to the fixed-base laboratory will be selected to reflect the range of lead concentrations detected in the soil at the site using XRF field screening, with the majority having field XRF lead concentrations between 250 and 550 mg/kg. A correlation study (field screening versus fixed-base laboratory concentrations) may be conducted to support the data interpretation for the investigation. The samples will be analyzed using SW-846 Method 6020A.

  - Four of the samples selected for submittal to the fixed-base laboratory will also undergo analysis for pH, TOC, and CEC. The samples will be biased toward locations demonstrating evidence of contamination (visual observations of spent munitions/shot and the results of XRF field screening).

  - The two firing line samples will be composite samples analyzed for NG using Appendix A of SW-846 Method 8330B and lead using SW-846 Method 6020A at the fixed-base laboratory.

SAP Worksheet #11. 4 -- Project Quality Objectives/Systematic Planning Process Statements for the Rum Point Skeet Range

PROBLEM DEFINITION (STEP 1)

Worksheet 10.4 (Section 10.4.12) contains the Problem Definition for the Rum Point Skeet Range.

IDENTIFY THE GOALS OF THE STUDY (STEP 2)

The primary goals of this investigation of the Rum Point Skeet Range are as follows:

1. Collect adequate data to delineate the horizontal and vertical extent of soil and drainageway surface water and sediment contamination. If the data collected in the initial round of sampling are adequate, stop delineation; otherwise, continue sampling (i.e., “step-out” vertically and/or horizontally as necessary).

2. Determine whether MC (specifically antimony, arsenic, lead, copper, tin, zinc, and PAHs) are present in study area soil and drainageway surface water and sediment and also NG in the soil in the area of the firing lines at concentrations that represent unacceptable human health or ecological risk. If either risk is unacceptable, evaluate the need to proceed to an FS; otherwise, recommend NFA.

3. Determine whether MC (specifically NG, antimony, arsenic, lead, copper, tin, zinc, and PAHs) are present in study area soil at concentrations that represent a threat to groundwater quality. If MC concentrations in soil do represent a threat to groundwater quality, recommend an environmental investigation of groundwater contamination; otherwise, do not initiate a groundwater investigation on the basis of soil contamination levels.

4. Update the CSM using field reconnaissance survey data and initial hazard and risk screening results.

All data collected for this project are expected to be transferable to support the following actions:

1. Update the Navy CTC estimate
2. Complete the site prioritization protocol

IDENTIFY INFORMATION INPUTS (STEP 3)

Data required for making the decisions include the following:

1. A comprehensive listing of the relevant, medium-specific RBSLs and potential chemical-specific ARARs for the MC of concern (specifically antimony, arsenic, lead, copper, tin, zinc, and PAHs). Screening levels based on USEPA Region 3 RBCs, the USEPA action level for lead in soil assuming a residential land use scenario (400 mg/kg), USEPA SSls, USEPA Region 3 BTAG Sediment Screening Benchmarks, USEPA Ecological SSLs, USEPA Region 3 BTAG SSLs, and USEPA Region 3 Freshwater Screening Benchmarks.

2. Laboratory analytical data characterizing concentrations of NG, the six MC metals of interest and PAHs in soil [surface soil (0 to 1 foot bgs) and subsurface soil (greater than 1 foot bgs), as necessary] and drainageway surface water and sediment samples (0 to 6 inches). Soil, surface water, and sediment samples will be analyzed for NG using Appendix A to SW-846 Method 8330B, MC metals using SW-846 Method 6020A, and for PAHs using SW-846 Method 8270C. MC concentrations in soil, surface water, and sediment will be compared to site screening levels and background concentrations. These data are necessary to conduct the human health and ecological risk screening assessments. Laboratory quantitation limits for the proposed analytical methods should not exceed the aforementioned screening levels and ARARs. Practical quantitation limits should not exceed risk-based project action levels (i.e., levels associated with a $1x10^{-4}$ cumulative cancer risk level, a cumulative hazard index of 1, or the USEPA action level for lead [400 mg/kg]). Based on knowledge of lead concentrations in similar soil, native soil and sediment lead concentrations are expected to be less than 50 mg/kg. Because of this, contaminated areas (e.g., concentrations exceeding 400 mg/kg) are expected to be well delineated, with steep lead concentration gradients between clean and contaminated areas.

3. Because lead is the primary contaminant and other metals are expected to be collocated with lead, lead can be used as a marker for contamination. For delineation of lead contamination, the use of both field and laboratory analyses is anticipated. This will provide a high degree of spatial coverage for relatively low cost. XRF analysis will be used for lead analysis in the field. An XRF field action level of 300 mg/kg will be used as the discriminator between contaminated and uncontaminated locations. The 300 mg/kg corresponds to the USEPA action level of 400 mg/kg reduced by a 25 percent margin of error to minimize the potential that contaminated areas go undetected. The margin of error was selected based on recent experiences with field XRF analyses at other small arms ranges.

4. Laboratory analytical data characterizing soil and sediment pH, TOC, and CEC characteristics. These data are necessary for the evaluation of MC environmental fate and transport.

5. Background soil data suitable for site soil-versus-background soil data comparisons for the six metals of interest for use in site versus background comparisons.

6. Field identification/classification of soil and sediment types (i.e., lithology). Worksheet #21 contains relevant SOPs.

7. Physical characterization of the major surface water drainage pathways (e.g., number, width, depth, physical characteristics of sediment, etc.). Worksheet #21 contains relevant SOPs.

8. Field investigation specifications (e.g., SOPs for sample collection).

9. QA/QC specifications and the results of QA/QC reviews (e.g., data validation) conducted throughout the investigation. Specifications are provided in Worksheet #'s 19 through 37.

10. Performance criteria (correlation coefficients) for correlation of lead XRF data to fixed-base laboratory data for lead. The correlation coefficient shall be greater than or equal to 0.65 and less than or equal to 1.00. These correlations will be used to translate field XRF values into equivalent laboratory lead concentrations. Correlation analyses are not anticipated to be generated for the five other metals.

Tetra Tech will collect the field samples, conduct the field XRF lead analysis, prepare selected samples for the fixed-base laboratory, and prepare the report in which data are interpreted and presented in tabular and graphical formats.

Data will become part of the AR for NSF-IH after approval of the report by USEPA Region 3 and the State of Maryland.

DEFINE THE BOUNDARIES OF THE STUDY (STEP 4)

The current site investigation is limited to the evaluation of soil and drainageway surface water/sediment at the Rum Point Skeet Range. An investigation of groundwater underlying the site, and surface water and sediment of the adjoining Mattawoman Creek is beyond the scope of this investigation, but may be required in the future based, in part, on the outcome of this investigation. A visual depiction of the range is provided on Map 10.4-1. Establishing the nature and extent of contamination will require that both contaminated and non-contaminated soil and drainageway surface water/sediment be sampled (i.e., the

perimeter of the impacted area must be established). The following items address the horizontal and vertical boundaries as well as the temporal boundaries for the study:

1. The populations of interest include soil and sediment that may have been contaminated directly by site operations or by subsequent migration of contaminants.

2. The initial horizontal study boundary will encompass the area that, based on historical information, is most likely to have been impacted by site activities and is shown on Map 10.4-2. Lateral expansion of this horizontal study boundary may be necessary if MC concentrations detected in the samples collected along this boundary exceed screening levels (see Step 5, Decision Rules). The study area has the following sub-zones:

   - The firing lines. An area at the firing points where unburned powders and lead from primers would have accumulated. NG may have been deposited from unburned propellant.

   - An area downrange of the firing line where clay “target fragments” (i.e., fragments of targets actually hit by shotguns) would have accumulated (spent lead shot would also be present in this area).

   - An area downrange of the target fragments where used/weathered targets (not hit by shotgun shells [i.e., missed targets]) would have accumulated; An area downrange of the firing line, the target fragments, and the used/weathered target fragments where the greatest concentrations of lead shot would accumulate (targets and target fragments are unlikely to be within this area) (see Map 10.4-3).

   - For purposes of risk assessment, the site may be further divided horizontally if analytical data suggest that “hot spot” zones are present at the site.

3. The initial vertical study boundary will be limited to 1 foot bgs because the CSM indicates that penetration of soil and sediment by MC is not generally expected to be deeper than this. Vertical expansion of this study boundary may be necessary if MC concentrations detected in the samples collected along this boundary exceed screening levels (see Step 5 Decision Rules). The direct contact soil risk screening assessment will be based on concentrations to a maximum depth of 10 feet bgs. The exposure unit represented by the exposure point concentration will be the entire volume of contaminated soil within the lateral extent of the exposure unit divided as necessary to

allow separate evaluations of surface (0 to 1 foot bgs), subsurface soil (1 to 10 feet bgs), or all soil (0 to 10 feet bgs).

4. The temporal boundary is not a significant consideration in this study because MC concentrations (NG, metals, and PAHs) are anticipated to be relatively unchanged (stable) over the course of time needed to conduct the environmental investigations.

DEVELOP THE ANALYTIC APPROACH (STEP 5)

Decision rules for this investigation include the following:

1. If MC concentrations in all soil and drainageway surface water/sediment samples in the initial round of sampling are less than screening or background levels, recommend NFA; otherwise, advance to Rule 2.

2. If the XRF-measured concentrations of lead in any soil/sediment sample on the perimeter (vertical or horizontal) of the sampled area exceeds 300 mg/kg, “step-out” (vertically and/or horizontally) as necessary for XRF lead analysis of additional soil/sediment until the results of the XRF analysis are less than 300 mg/kg. Advance to Rule 3.

3. If any of the fixed-base laboratory concentrations for NG, antimony, arsenic, lead, copper, tin, zinc, or PAHs in the initial round perimeter soil/sediment samples exceed screening and background levels, use professional judgment to determine the degree to which “step-out” samples (vertical or horizontal) are necessary to define the vertical and/or horizontal extent of contamination in perimeter soil or drainageway sediment. Factors considered will include the specific contaminants, the magnitude of the exceedance, their spatial distribution and the overall risk level. If step-out samples are deemed necessary by the Project Team, collect as necessary during a follow-up RI and then advance to Rules 4, 5, and 6; otherwise, advance to Rules 4, 5, and 6.

4. If MC concentrations in the receptor exposure units defined for soil, surface water, or drainageway sediment represent unacceptable human health risk, proceed to an FS; otherwise, recommend NFA from a human health perspective. Unacceptable human health risk will be generally defined as cancer risk estimates exceeding 1E-04, non-cancer risk estimates (i.e., hazard indices) exceeding 1 (on a target-organ specific basis), or receptor blood-lead levels that exceed the current USEPA benchmark (i.e., a receptor shall have no more than a 5 percent probability of a blood-lead levels exceeding 10 µg/dL).

5. For ecological risk assessments, maximum detected MC concentrations in surface soil, surface water, and sediment will be compared to screening concentrations to determine if a contaminant is a COPC. Average concentrations (arithmetic means) of surface soil data or surface water/sediment data will be used in food-chain modeling. If risks for defined receptor exposure units are determined to be "unacceptable" based on an evaluation of several lines of evidence (e.g., number of exceedances of screening criteria, magnitude of the exceedances of screening criteria, spatial distribution of data, home range, background concentrations, etc.), the project team will determine the need to conduct a BERA or consider the potential risks with respect to remedial actions. Any BERA would be conducted as part of a future RI.

6. If MC concentrations in any soil sample exceed both background levels and USEPA SSLs for groundwater protection, recommend an evaluation of the need for an investigation of groundwater contamination; otherwise, do not recommend evaluating the need for a groundwater investigation be initiated on the basis of soil/sediment concentrations.

Note:

A) MC are defined specifically for this site as NG, antimony, arsenic, lead, copper, tin, zinc, and PAHs.

B) Screening levels for the investigation: Screening levels based on USEPA Region 3 RBCs, the USEPA action level for lead in soil assuming a residential land use scenario (400 mg/kg), USEPA SSLs, USEPA Region 3 BTAG Sediment Screening Benchmarks, USEPA Ecological SSLs, USEPA Region 3 BTAG SSLs, and USEPA Region 3 Freshwater Screening Benchmarks

C) Evaluation criteria for "stepping out" based on fixed-base laboratory data: Ideally, and in most cases, "step-out" samples should be collected until perimeter sample concentrations are less than screening or background concentrations. However, professional judgment should be used to determine if such samples are needed in all cases. Professional judgment considerations include:

- The degree to which MC concentrations exceed screening or background levels. The need to collect additional samples should be carefully considered when results, for example, are within 5 percent of screening or background levels. The need to collect additional data will increase with increased number of exceedances and increased contaminant concentrations.

- The degree to which further delineation can be more efficiently and economically collected as part of follow-up RI/FS investigations (i.e., the additional sampling is unlikely to impact the decision to take or not take some type of remedial action but may be necessary to define the limits of the action).

- Considerations of the impact of risk estimations if some contaminant concentrations are not completely delineated.

D) The results of the initial round of sampling and any professional judgments made regarding the need for "step-out" samples will be reviewed with the project team.

E) Define "generally": The NCP discusses a generally acceptable risk range of $1x10^{-4}$ to $1x10^{-6}$. However, based on guidance presented in USEPA RAGS Part D, risks slightly greater than $1x10^{-4}$, may be considered acceptable (i.e., protective) if justified based on site-specific conditions including any uncertainties about the nature and extent of contamination and associated risks (USEPA, December 2001).

F) A BERA (and the collection of data to support such an assessment) is beyond the scope of this investigation and would require development of a "follow-up" UFP-SAP and associated decision rules.

SPECIFY PERFORMANCE OR ACCEPTANCE CRITERIA (STEP 6)

The VSP software tool was used to determine the number of soil samples necessary assuming that lead was the primary contaminant of concern. The following key assumptions and performance/acceptance criteria were used to determine the number of soil samples:

- For purposes of statistical analysis, the "null hypothesis" is that the site was "dirty" (i.e., the mean lead concentration at the site exceeds the action level selected for statistical analysis).

- S (Sigma) = the estimated standard deviations for areas assumed to be relatively uncontaminated (clean), contaminated (dirty), and the transition zone were set at 100 mg/kg, 8,330 mg/kg, and 830 mg/kg, respectively.

- Delta = the width of the gray region, which was set equal to 150 for clean areas, 14,600 for contaminated areas, 1,600 for the transition zone.

- Alpha = the tolerance for concluding that the site is "clean" when the site is actually "dirty". If an incorrect decision was to be made, the team prefers to incorrectly take action to remediate a clean site rather than to fail to take action at a dirty site. Making an incorrect decision becomes less tolerable as the site becomes increasingly contaminated or increasingly clean relative to the action level. Therefore, at a true mean concentration infinitesimally greater than the action level, the consequence of a decision error is not as great as, for example, if the true mean concentration was two times the action level. The tolerance for incorrectly concluding that the site mean is less than two times the action level when the true mean is greater than the action level was set at 15 percent.

- Beta = the tolerance for concluding that the site is "dirty" when the site is actually "clean" was also established considering the tolerance for incorrectly concluding that the site mean is greater than the action level when it is actually infinitesimally less than 0.9 times the action level was set to 25 percent. This Beta value is greater than the Alpha value because there is more tolerance for this type of error than for the error of not taking action when a site is dirty.

- The LBGR presented in terms of a percentage of the action level: 90 percent.

- The action level of 400 mg/kg for lead which was selected for statistical analysis.

The results of the statistical analysis presented in Appendix D are that a minimum of five soil samples should be collected across each of the designated areas to be sampled and for which a decision must be made. The sampling design presented in Step 7 is based on this recommended number of samples and samples necessary to determine the boundaries of contamination and potential remedial areas. All new analytical data collected per the Step 7 sampling design should meet the QA criteria established in Worksheet #'s 19 through 37 and the prescribed detection limit requirements for the MC constituents of concern.

DEVELOP THE PLAN FOR OBTAINING DATA (STEP 7)

The proposed SI field data collection program for the Rum Point Skeet Range is presented, to a large extent, on Map 17-4. The program assumes, based on the CSM, that the following potential depositional areas exist downrange of the firing line and to a lesser extent at the firing lines:

- An area at the firing lines where unburned powders and lead from primers would have accumulated.

- An area where clay "target fragments" (i.e., fragments of targets actually hit by shotguns) would have accumulated (spent lead shot would also be present in this area).

- An area where used/weathered targets (not hit by shotgun shells [i.e., missed targets]) would have accumulated.

- An area where the greatest concentrations of lead shot would accumulate (targets and target fragments are unlikely to be within this area).

The zone of maximum shot accumulation for the Rum Point Skeet Range is presumed to occur near the tree line at the range. As noted in Step 4, the horizontal and vertical boundaries of the soil/sediment drainageway areas to be sampled will encompass all areas significantly impacted by site activities.

The sampling design consists of samples spaced along transect lines extending from the shooters' positions to the maximum shot fall line to ensure that the decision units are well represented spatially. The heavy tree line encompassing the entire range shot fall out area would have significantly reduced the maximum distance of the lead shot and targets. Contamination may also be present at the firing lines. A site walk-over will be conducted by the FOL to identify any obvious areas of fragment/lead shot accumulation. The MC sampling and analytical program for soil and drainageway sediment samples is as follows:

- Soil
  - Eighty-one surface soil samples (0 to 1 foot bgs) will be initially collected from the transect lines established in the area to be sampled as shown on Map 17-4. Additionally, a site "walk over" will be conducted by the FOL to identify any obvious areas of fragment/lead shot accumulation. Additional discretionary surface soil samples may be collected based on visual observation of accumulated fragments or shot in the area to be sampled. Emphasis will be placed on collecting samples required to delineate contaminated areas. Additional "step-out" samples will also be collected at the boundaries of the area to be sampled if lead concentrations greater than the field action level of 300 mg/kg are detected based on the results of the XRF field screening analysis of the initial surface soil samples.

- Subsurface soil samples (starting at 1 foot bgs) will also be collected at all surface soil locations but will only be subject to field XRF analysis if their respective surface soil sample lead concentrations exceed 300 mg/kg. If the subsurface sample XRF lead concentration also exceeds 300 mg/kg, additional subsurface soil samples will then be collected vertically in 1-foot intervals until lead concentrations in subsurface soil do not exceed 300 mg/kg or until collection of samples via a hand auger becomes impractical.

- Two composite surface soil samples will be collected, one from the area at the left firing point, and one from the area at the right firing point, for NG and lead analysis using hand augers.

- Drainageway Surface Water/Sediment
  - Prior to sampling, a site "walk over" will be conducted by the FOL to identify any obvious surface water drainage pathways potentially leading to adjoining surface water bodies (e.g., the adjoining wetlands and Mattawoman Creek.) Discretionary drainageway surface water/sediment samples will be collected from along these pathways. Locations will be targeted for sampling based on proximity to surface soil locations with elevated lead concentrations, on the accumulation of sediment along the pathway with emphasis placed on sampling sediment traps, and on the potential for the pathway to lead to a surface water body such as the wetlands of Mattawoman Creek.

- Analytical Program
  - All soil and sediment samples will be analyzed for lead using XRF field screening analysis. A minimum of 20 percent of these samples (but no fewer than 20 samples) will also be analyzed for the MC metals of concern at a fixed-base laboratory. The samples sent to the fixed-base laboratory will be selected to reflect the range of lead concentrations detected in the soil/sediment at the site using XRF field screening, with the majority having field XRF lead concentrations between 250 and 550 mg/kg. A correlation study (field screening versus fixed-base laboratory concentrations) may be conducted to support data interpretation for the investigation. The samples will be analyzed for metals using SW-846 Method 6020A.

  - Sediment and soil samples collected along the transect lines in the downrange area potentially contaminated with spent targets and target fragments (and selected for MC metals analysis at a fixed-base laboratory) and any discretionary samples collected from those areas will also be analyzed for PAHs. The samples will be analyzed for metals using SW-846 Method 6020A and for PAHs using SW-846 Method 8270C.

- Four of the samples selected for submittal to the fixed-base laboratory will also undergo analysis for pH, TOC, and CEC. The samples will be biased toward locations with evidence of contamination (visual observations of fragments/shot and the results of field screening).

- The two firing line samples will be composite samples analyzed for NG using Appendix A of SW-846 Method 8330B and lead using SW-846 Method 6020A at the fixed-base laboratory.

SAP Worksheet #11.5 -- Project Quality Objectives/Systematic Planning Process Statements for the Small Arms (Pistol) Range

PROBLEM DEFINITION (STEP 1)

Worksheet 10.5 (Section 10.5.12) contains the Problem Definition for the Small Arms (Pistol) Range.

IDENTIFY THE GOALS OF THE STUDY (STEP 2)

The primary goals of this investigation of the Small Arms (Pistol) Range are as follows:

1. Collect adequate data to delineate the horizontal and vertical extent of soil and drainageway/creek sediment contamination. If the data collected in the initial round of sampling is adequate, stop delineation; otherwise, continue sampling (i.e., “step-out” vertically and/or horizontally as necessary).

2. Determine whether MC (specifically antimony, arsenic, lead, copper, tin, and zinc) are present in study area soil and drainageway/creek sediment and also if NG and lead are present in the soil in the area of the firing lines at concentrations that represent unacceptable human health or ecological risk. If either of these risks are unacceptable, evaluate the need to proceed to an FS; otherwise, recommend NFA.

3. Determine whether MC (specifically NG, antimony, arsenic, lead, copper, tin, and zinc) are present in study area soil at concentrations that represent a threat to groundwater quality. If MC concentrations in soil do represent a threat to groundwater quality, recommend an environmental investigation of groundwater contamination; otherwise, do not initiate a groundwater investigation on the basis of soil contamination levels.

4. Update the CSM using field reconnaissance survey data and initial hazard and risk screening results.

All data collected for this project are expected to be transferable to support the following actions:

1. Update the Navy CTC estimate
2. Complete the site prioritization protocol

IDENTIFY INFORMATION INPUTS (STEP 3)

Data required for making the decisions which include the following:

1. A comprehensive listing of the relevant, medium-specific RSBLs and potential chemical-specific ARARs for the MC of concern (specifically antimony, arsenic, lead, copper, tin, and zinc). (Screening levels based on USEPA Region 3 RBCs, the USEPA action level for lead in soil assuming a residential land use scenario (400 mg/kg), USEPA SSLs, USEPA Region 3 BTAG Sediment Screening Benchmarks, USEPA Ecological SSLs, USEPA Region 3 BTAG SSLs, and USEPA Region 3 Freshwater Screening Benchmarks.

2. Laboratory analytical data characterizing concentrations of NG and the six MC metals of concern in soil [surface soil (0 to 1 foot bgs) and subsurface soil (greater than 1 foot bgs), as necessary] and drainageway/creek sediment samples (0 to 6 inches bgs). Soil and sediment samples will be analyzed for NG using Appendix A of SW-846 Method 8330B and MC metals using SW-846 Method 6020A. MC concentrations in soil and sediment will be compared to site screening levels and background concentrations. These data are necessary to conduct the human health and ecological risk screening assessments. Laboratory quantitation limits for the proposed analytical methods should not exceed the aforementioned screening levels and ARARs. Practical quantitation limits should not exceed risk-based project action levels (i.e., levels associated with a $1x10^{-4}$ cumulative cancer risk level, a cumulative hazard index of 1, or the USEPA action level for lead [400 mg/kg]). Based on knowledge of lead concentrations in similar soils, native soil and sediment lead concentrations are expected to be less than 50 mg/kg. Because of this, contaminated areas (e.g., with concentrations exceeding 400 mg/kg) are expected to be well delineated, with steep lead concentration gradients between clean and contaminated areas.

3. Because lead is the primary contaminant and other metals are expected to be collocated with lead, lead can be used as a marker for contamination. For delineation of lead contamination, the use of both field and laboratory analyses is anticipated. This will provide a high degree of spatial coverage for relatively low cost. XRF analysis will be used for lead analysis in the field. An XRF field action level of 300 mg/kg will be used as the discriminator between contaminated and uncontaminated locations. The 300 mg/kg corresponds to the USEPA action level of 400 mg/kg reduced by a 25 percent margin of error to minimize the potential that contaminated areas go undetected. The margin of error was selected based on recent experiences with field XRF analyses at other small arms ranges.

4. Laboratory analytical data characterizing soil and sediment pH, TOC, and CEC characteristics. These data are necessary for the evaluation of MC environmental fate and transport.

5. Background soil data suitable for site soil-versus-background soil data comparisons for the six metals of interest for use in site versus background comparisons.

6. Field identification/classification of soil and sediment types (i.e., lithology and USCS soil classification for grain size, color, plasticity, etc.). Worksheet #21 provides relevant SOPs.

7. Physical characterization of the major surface water drainage pathways (e.g., number, width, depth, physical characteristics of sediment, etc.). Worksheet #21 provides relevant SOPs.

8. Field investigation specifications (e.g., SOPs for sample collection).

9. QA/QC specifications and the results of the QA/QC reviews (e.g., data validation) conducted throughout the investigation. Specifications are provided in Worksheet #'s 19 through 37.

10. Performance criteria (correlation coefficients) for correlation of the lead XRF data to fixed-base laboratory data for lead. The correlation coefficient shall be greater than or equal to 0.65 and less than or equal to 1.00. These correlations will be used to translate field XRF values into equivalent laboratory lead concentrations. Correlation analyses are not anticipated to be generated for the five other metals.

Tetra Tech will collect the field samples, conduct the field XRF lead analysis, prepare selected samples for the fixed-base laboratory, and prepare the report in which data are interpreted and presented in tabular and graphical formats.

Data will become part of the AR for NSF-IH after approval of the report by USEPA Region 3 and the State of Maryland.

DEFINE THE BOUNDARIES OF THE STUDY (STEP 4)

The current site investigation is limited to the evaluation of soil and drainageway sediment at the Small Arms (Pistol) Range. An investigation of groundwater underlying the site and the surface water of adjoining surface water bodies is beyond the scope of this investigation but may be required in the future based, in part, on the outcome of this investigation. A visual depiction of the range is provided on Map 10.5-1. Establishing the nature and extent of contamination will require that both contaminated and non-contaminated soil and drainageway sediment be sampled (i.e., the perimeter of the impacted area must

be established). The following items address the horizontal and vertical boundaries as well as the temporal boundaries for the study:

1. The populations of interest include those soil and sediment that may have been contaminated directly by site operations or by subsequent migration of contaminants.

2. The initial horizontal study boundary will encompass the area that, based on historical information, is most likely impacted by site activities (the earthen embankment) and is shown on Map 10.5-2. Lateral expansion of this horizontal study boundary may be necessary if MC concentrations detected in the samples collected along this boundary exceed screening levels (see Step 5, Decision Rules). The following sub-zones are within the study boundary:

   - The firing lines: An area at the firing points where unburned powders and lead from primers would have accumulated. NG may have been deposited from unburned propellant.

   - The range floor immediately in front of the hillside which may be contaminated with stray bullet fragments. Contamination in this area is anticipated to be marginal compared to the primary impact areas.

   - The primary impact berm (i.e., the hillside embankment). Bullets that were shot below the range targets would likely to have been dispersed in the lower sections of the embankment, closer to the toe of the embankment (i.e., a dispersed metal impact zone) (see Map 10.5-2).

   - For purposes of risk assessment, the site may be further divided horizontally if the analytical data suggest that a distinct “hot spot” zone(s) is present at the site and these “hot spots” are large enough to define an exposure unit for a receptor.

3. The initial vertical study boundary will be limited to 1 foot bgs, except in the area where sloughed soil is present, because the CSM indicates that penetration of soil sediment by MC is not generally expected to be deeper than this. In the area where sloughed soil is present, samples will be collected to 10 feet bgs by DPT. Vertical expansion of this study boundary may be necessary if MC concentrations detected in the samples collected along this boundary exceed screening levels or background (see Step 5, Decision Rules). The direct contact soil risk screening assessment will be based on concentrations to a maximum depth of 10 feet bgs. The exposure unit represented by the exposure point concentration will be the entire volume of contaminated soil within the lateral extent of

the exposure unit divided as necessary to allow separate evaluations of surface (0 to 1 foot bgs), subsurface soil (1 to 10 feet bgs) or all soil (0 to 10 feet bgs).

4. The temporal boundary is not a significant consideration in this study because MC concentrations (NG and metals) are anticipated to be relatively unchanged (stable) over the course of time needed to conduct the environmental investigations and into the foreseeable future.

DEVELOP THE ANALYTIC APPROACH (STEP 5)

The decision rules for this investigation are as follows:

1. If MC concentrations in all soil and drainageway sediment samples in the initial round of sampling are less than screening or background levels, recommend NFA; otherwise, advance to Rule 2.

2. If the XRF-measured concentrations of lead in any sample on the perimeter (vertical or horizontal) of the sampled area exceeds 300 mg/kg, then “step-out” (vertically and/or horizontally) as necessary for XRF lead analysis of additional soil until the results of the XRF analysis are less than 300 mg/kg. Advance to Rule 3.

3. If any of the fixed-base laboratory concentrations for NG, antimony, arsenic, lead, copper, tin, and zinc in the initial round perimeter samples exceed screening and background levels, use professional judgment to determine the degree to which “step-out” samples (vertical or horizontal) are necessary to define the vertical and/or horizontal extent of contamination in perimeter soil or drainageway sediment. Factors considered will include the specific contaminant, the magnitude of the exceedance, their spatial distribution, and the overall risk level. If “step-out” samples are deemed necessary, collect samples as necessary as part of a follow-up RI and then advance to Rules 4, 5, and 6; otherwise, advance to Rules 4, 5, and 6.

4. If MC concentrations in the receptor exposure units defined for soil or drainageway sediment represent “unacceptable human health risk” proceed to an FS; otherwise, recommend NFA from a human health perspective. “Unacceptable human health risk” will be generally defined as cancer risk estimates exceeding 1E-04, non-cancer risk estimates (i.e., hazard indices) exceeding 1 (on a target-organ specific basis), or receptor blood-lead levels that exceed the current USEPA benchmark (i.e., a receptor shall have no more than a 5 percent probability of a blood-lead level exceeding 10 µg/dL).

5. For ecological risk assessments, maximum detected MC concentrations in surface soil and sediment will be compared to screening concentrations to determine if a contaminant is a COPC. Average concentrations (arithmetic means) of surface soil data or sediment data will be used in food-chain modeling. If risks for defined receptor exposure units are determined to be "unacceptable" based on an evaluation of several lines of evidence (e.g., number of exceedances of screening criteria, magnitude of the exceedances of screening criteria, spatial distribution of data, home range, background concentrations, etc.), the project team will determine the need to conduct a BERA or consider the potential risks with respect to remedial actions. Any BERA would be conducted as part of a future RI.

6. If MC concentrations in any soil sample exceed both background levels and USEPA SSLs for groundwater protection, recommend an evaluation of the need for an investigation of groundwater contamination; otherwise, do not recommend evaluating the need for a groundwater investigation be initiated on the basis of soil/sediment concentrations.

Note:

A) MC are defined specifically for this site as NG, antimony, arsenic, lead, copper, tin, and zinc.

B) Screening levels for the investigation: Screening levels based on USEPA Region 3 RBCs, the USEPA action level for lead in soil assuming a residential land use scenario (400 mg/kg), USEPA SSLs, USEPA Region 3 BTAG Sediment Screening Benchmarks, USEPA Ecological SSLs, USEPA Region 3 BTAG SSLs, and USEPA Region 3 Freshwater Screening Benchmarks.

C) Evaluation criteria for "stepping out" based on fixed-base laboratory data are as follows: Ideally, and in most cases, "step-out" samples should be collected until perimeter sample concentrations are less than screening or background concentrations. However, professional judgment should be used to determine if such samples are needed in all cases. Professional judgment considerations include:

- The degree to which MC concentrations exceed screening or background levels. The need to collect additional samples should be carefully considered when results, for example, are within 5 percent of screening or background levels. The need to collect additional samples will increase with increased number of exceedances and increased contaminant concentrations.

- The degree to which further delineation can be more efficiently and economically collected as part of follow-up RI/FS investigations (i.e., the additional sampling is unlikely to impact the decision to

take or not take some type of remedial action but may be necessary to define the limits of the action.)

- Considerations of the impact of risk estimations if some contaminant concentrations are not completely delineated.

D) The results of the initial round of sampling and any professional judgments made regarding the need for "step-out" samples will be reviewed with the project team.

E) Define "generally": The NCP discusses a generally acceptable risk range of $1x10^{-4}$ to $1x10^{-6}$. However, based on guidance presented in USEPA RAGS Part D, risks slightly greater than $1x10^{-4}$ may be considered acceptable (i.e., protective) if justified based on site-specific conditions including any uncertainties about the nature and extent of contamination and associated risks (USEPA, December 2001).

F) A BERA (and the collection of data to support such an assessment) is beyond the scope of this investigation and would require development of a "follow-up" UFP-SAP and associated decision rules.

SPECIFY PERFORMANCE OR ACCEPTANCE CRITERIA (STEP 6)

The VSP software tool was used to determine the number of soil samples necessary assuming that lead was the primary contaminant of concern. Based on the VSP output, the following key assumptions and performance/acceptance criteria were used to determine the number of soil samples:

- For purposes of statistical analysis, the "null hypothesis" is that the site was "dirty" (i.e., the mean lead concentration at the site exceeds the action level selected for statistical analysis).

- S (Sigma) = the estimated standard deviations for areas assumed to be relatively uncontaminated (clean), contaminated (dirty), and the transition zone were set at 100 mg/kg, 8,330 mg/kg, and 830 mg/kg, respectively.

- Delta = the width of the gray region, which was set equal to 150 for clean areas, 14,600 for contaminated areas, 1,600 for the transition zone.

- Alpha = the tolerance for concluding that the site is "clean" when the site is actually "dirty". If an incorrect decision was to be made, the team prefers to incorrectly take action to remediate a clean

site rather than to fail to take action at a dirty site. Making an incorrect decision becomes less tolerable as the site becomes increasingly contaminated or increasingly clean relative to the action level. Therefore, at a true mean concentration infinitesimally greater than the action level, the consequence of a decision error is not as great as, for example, if the true mean concentration was two times the action level. The tolerance for incorrectly concluding that the site mean is less than two times the action level when the true mean is greater than the action level was set at 15 percent.

- Beta = the tolerance for concluding that the site is "dirty" when the site is actually "clean" was also established considering the tolerance for incorrectly concluding that the site mean is greater than the action level when it is actually infinitesimally less than 0.9 times the action level was set to 25 percent. This Beta value is greater than the Alpha value because there is more tolerance for this type of error than for the error of not taking action when a site is dirty.

- The LBGR presented in terms of a percentage of the action level: 90 percent.

- The action level of 400 mg/kg for lead which was selected for statistical analysis.

The results of the statistical analysis presented in Appendix D are that a minimum of five soil samples should be collected across each of the designated area(s) to be sampled and for which a decision must be made. The sampling design presented in Step 7 is based on this recommended number of samples and samples necessary to determine the boundaries of contamination and potential remedial areas. All new analytical data collected per the Step 7 sampling design should meet the QA criteria established in Worksheet #'s 19 through 37 and the prescribed detection limit requirements for the MC constituents of concern.

DEVELOP THE PLAN FOR OBTAINING DATA (STEP 7)

The proposed SI field data collection program for the Small Arms (Pistol) Range area is presented, to a large extent, on Map 17-5. The program assumes, based on the CSM, that the following potential depositional areas exist downrange of the firing line and to a lesser extent at the firing lines:

- An area at the firing lines where unburned powders and lead from primers would have accumulated.

- The range floor immediately in front of the sloughed soil from the hillside, which may be contaminated with stray bullet fragments. Contamination in this area is anticipated to be marginal compared to the primary impact areas.

- The primary impact berm (i.e., the embankment). Bullets that were shot below the range targets would likely be dispersed in the lower sections of the embankment, closer to the toe of the embankment (i.e., a dispersed metal impact zone). Bullets that successfully hit the targets most likely would continue through the targets and primarily strike a sub-area of the embankment (i.e., a concentrated metal impact zone.) (see Map 10.5-2).

Range fire that overshoot targets would have impacted the soil above and behind the targets. However, it is assumed that most of the spent munitions would most likely have fallen in the area of the impact berm. As noted in Step 4, the horizontal and vertical boundaries of the soil/sediment drainageway areas to be sampled will encompass all areas significantly impacted by site activities.

The sampling design consists of samples spaced along transect lines extending the length of the hillside that falls within the range area to ensure that the decision units are well represented spatially. Contamination may also be present at the firing lines. A site walk-over will be conducted by the FOL to identify any obvious areas of fragmented/lead shot accumulation. The MC sampling and analytical program for soil and drainageway sediment samples is as follows:

- Soil
  - Twenty-one soil samples will be collected from seven locations at three depths using DPT at the base of the hillside where the sloughed material from the hillside has accumulated. Seven surface soil samples will be collected from 0 to 1 foot bgs from the face of the hillside from 5 to 6 feet above ground surface. Six surface soil samples will be collected along a transect line directly in front of the sloughed soil, with at least one sample extending beyond each end of the sloughed soil. All sample locations are shown on Map 17-5. Additional discretionary surface soil samples may be collected based on visual observation of accumulated spent munitions in the area to be sampled. Emphasis will be placed on collecting samples required to delineate contaminated areas. Additional “step-out” samples will also be collected at the boundaries of the area(s) to be sampled if lead concentrations greater than the field action level of 300 mg/kg are detected based on the results of the XRF field screening analysis of the initial surface soil samples. Surface soil samples will be collected using hand augers, and subsurface samples within the sloughed area of the hillside will be collected using DPT.

  - Subsurface soil samples (starting at 1 foot bgs) will also be collected at all surface soil locations along the other two transect lines not in the sloughed soil, but will only be subject to field XRF analysis if their respective surface soil sample lead concentrations exceed 300 mg/kg. If the

subsurface sample XRF concentration also exceeds 300 mg/kg, additional subsurface soil samples will be collected in 1-foot intervals until lead concentrations in the subsurface soil do not exceed 300 mg/kg or until collection of samples via a hand auger becomes impractical.

- Three composite surface soil samples will be collected from areas of each of the three firing lines for NG and lead analysis using hand augers.

- Drainageway Sediment
  - Prior to sampling, a site "walk over" will be conducted by the FOL to identify any obvious surface water drainage pathways potentially leading to adjoining surface water bodies. Discretionary drainageway sediment samples will be collected from each of these pathways. Locations will be targeted for sampling based on proximity to surface soil locations with elevated lead concentrations, on the accumulation of sediment along the pathway with emphasis placed on sampling sediment traps, and on the potential for the pathway to lead to an adjoining surface water body with emphasis placed on those drainages leading to surface water bodies.

- Analytical Program
  - All soil and drainageway sediment samples will be analyzed for lead using XRF field screening analysis. A minimum of 20 percent of these samples (but no fewer than 20 samples) will also be analyzed for the MC metals of concern at a fixed-base laboratory. The samples sent to the fixed-base laboratory will be selected to reflect the range of lead concentrations detected in soil at the site using XRF field screening, with the majority having field XRF lead concentrations between 250 and 550 mg/kg. A correlation study (field screening versus fixed-base laboratory concentrations) may be conducted to support data interpretation for the investigation. The samples will be analyzed for metals using SW-846 Method 6020A.

  - Four of the samples selected for submittal to the fixed-base laboratory will also undergo analysis for pH, TOC, and CEC. The samples will be biased toward locations with evidence of contamination (visual observations of spent munitions/shot and the results of XRF field screening).

  - The three firing line samples will be composite samples analyzed for NG using Appendix A of SW-846 Method 8330B and lead using SW-846 Method 6020A at the fixed-base laboratory.

SAP Worksheet #12.1 -- Measurement Performance Criteria Table Field Quality Control Sample - All Fractions

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Field Blank	All Fractions	One per source water	Bias/Contamination	Detections ≤ QL	S&A
Equipment Rinsate Blanks	All Fractions	One per 20 field samples per matrix per sampling equipment	Bias/Contamination	Detections ≤ QL	S&A
Field Duplicate	All Fractions	One per 10 field samples collected	Precision	Aqueous samples Relative Percent Difference (RPD) of 30; Solid samples RPD of 50	S
Cooler Temperature Blank	All Fractions	One per cooler	Accuracy / Representativeness	Between 2 and 6 ° C	S&A

Miscellaneous parameters (pH, TOC, CEC) are being collected and do not require any field quality control samples except a cooler blank.

SAP Worksheet #12.2 -- Measurement Performance Criteria Table – Soil/Sediment Field Analyses – Lead via Field-Portable X-Ray Fluorescence

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Energy Calibration Checks	Lead	Per manufacturer recommendations	Accuracy/Precision	Per manufacturer recommendations	A
Instrument blank	Lead	One per 20 samples	Bias and Contamination	Detections less than reporting level	A
Method blank	Lead	One per 20 samples	Bias and Contamination	Detections less than reporting level	A
Duplicate Analysis	Lead	One per 20 field samples per matrix or once per day, whichever is more frequent.	Precision	$\leq$ 50% RPD	S&A
Field Duplicate	Lead	One per 20 field samples per matrix or once per day, whichever is more frequent.	Precision	$\leq$ 50% RPD	S&A
Data Completeness check	Lead	Calculated for the data set	Data Completeness	$\geq$ 95% Overall	S&A
Comparability and Sensitivity Check	Lead	Each compound	Sensitivity	Quantitation limits less than the screening levels and ARARs used to determine the Project Quantitation Limits listed in Worksheet #15.	S&A

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

(UFP-QAPP Manual Section 2.7)

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
Site histories, initial CSM	PA Report September 2005	Malcolm Pirnie September 2005	Basis for UFP-SAP site histories and CSMs	NA

The information in the PA Report was used as the basis for this UFP-SAP. In general, site histories and initial CSMs were used. Modifications were made to the Marine Rifle Range and the Roach Road Rifle Range as described below.

Marine Rifle Range

The hillside east of the target berms was added as an impact area for investigation. Bullets passing through, over, or by would have impacted this hillside.

Roach Road Rifle Range

Based on site-walks by Tetra Tech personnel, it was determined the initial layout of the range did not fit the topography of the area. The location of the range was modified slightly, so the face of the hillside would be the backdrop to the target berms. The modification can be seen on Map 10.3-1.

SAP Worksheet #14 -- Summary of Project Tasks

(UFP-QAPP Manual Section 2.8.1)

The Small Arms Range Site Investigation project activities consist of the following tasks:

- Field tasks, including:
  - Mobilization/demobilization and Utility Clearance
  - Drilling and soil sample collection
  - Sediment and surface water sampling
  - Quality control samples
  - Field Instrument calibration
  - Decontamination
  - IDW removal and disposal
  - Global Positioning System
  - XRF lead analysis of soil
- Analytical Tasks
- Data Management
- Assessment and Oversight
- Data Review
- Project Report

These tasks are summarized below. The standard operating procedures (SOPs) and field documents referenced below and in other worksheets are included in Appendices A and B, respectively.

Field Tasks

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

  Equipment requirements will be finalized by the FOL following the acceptance of the UFP-SAP. The FOL will review the scope of work and assemble equipment (e.g., vehicles and sampling, personal protection, and decontamination equipment) to implement and complete the field investigations. This list will be reviewed by the project team and the PM. The FOL will be responsible for receiving and unpacking the equipment and ensuring that all equipment is operable and calibrated. The FOL will be responsible for tracking equipment used in the field. Analytical laboratory services have been

subcontracted and the FOL will be responsible for coordinating associated field activities. The Tetra Tech QAO will be responsible for coordinating the analytical services, and the acquisition and delivery of sample containers to the site.

During mobilization, the FOL will review the roles and responsibilities of each field team member and review the requirements of the various field activities. A series of meetings will be conducted to review the sampling and analytical requirements. Upon mobilization, an on-site meeting will be conducted to review health and safety requirements. The Site Safety Officer (SSO) will be responsible for reviewing the HASP with the field team members and subcontractors. The field team will also be required to attend a hazard control briefing administered by the Environmental Division point of contact (POC) prior to commencing any field work at the Stump Neck Annex. Daily safety briefings by Tetra Tech shall occur for all field personnel.

- **Utility Clearance Tasks:** Prior to commencing any work at the Stump Neck Annex, the Comprehensive Work Approval Process (CWAP) will be followed. The CWAP will identify constraints in the work area, such as the locations of eagle's nests, archeological sites, wetlands, etc. that may affect work at the site and other requirements that must be met prior to commencing work, such as locating underground utilities, etc. Upon completion of the CWAP a Dig Permit will be issued to the utility contractor. The FOL will coordinate with facility personnel and with a Tetra Tech subcontractor for the utility clearance of all soil boring locations. Utilities that are not shown or are incorrectly located shall be marked on the permit and the marked-up permit returned to the Activity POC for inclusion in the Activity GIS.

- **Drilling Tasks:** All soil borings at the Marine Rifle Range (UXO 14), Old Skeet and Trap Range (UXO 15), Rum Point Skeet Range (UXO 16), Small Arms (Pistol) Range (UXO 17), and Roach Road Rifle Range (UXO 25) will be advanced utilizing hand augering techniques in accordance with SOP-05 (Soil Coring and Sampling Using Hand Augering Techniques). Some samples at the Small Arms (Pistol) Range will be collected utilizing DPT in accordance with SOP-06 (Borehole Advancement and Soil Coring and Sampling Using Direct-Push Technology) due to sloughing of the impact hillside face, which may have buried lead contamination at deeper depths. All borings will be logged in accordance with SOP-10 (Borehole and Soil Sample Logging). Methods for recording data are included in each SOP. Field SOPs are included in Appendix A.

- **Sample Collection Tasks:** Surface and subsurface soil samples will be collected in accordance with SOP-05 (Soil Coring and Sampling Using Hand Augering Techniques), SOP-06 (Borehole

Advancement and Soil Coring and Sampling Using Direct-Push Technology), and SOP-14 (Composite Sampling for Soil and Sediment). Sediment samples will be collected in accordance with SOP-08 (Sediment Sampling), and surface water samples will be collected in accordance with SOP-07 (Surface Water Sampling). The sample numbering scheme will be in accordance with SOP-02 (Sample Identification and Nomenclature). Methods for recording data are included in each SOP and SOP-03 (Sample Custody and Documentation of Field Activities). Sample labeling will be in accordance with SOP-01 (Labeling), and sample containers, preservation, packaging, and shipping will be in accordance with SOP-11 (Sample Packaging). Field SOPs are included in Appendix A of this document.

The numbers and types of samples to be collected at each site along with associated analytical programs are presented in Tables 18.1 through 18.5.

- **Quality Control Tasks:** Equipment blanks, field duplicates, matrix spikes, and matrix spike duplicates will be collected as presented in Worksheet #20.

  Initial and continuing calibrations, tuning, reagent blanks, surrogates, duplicates, laboratory control samples, and all other applicable QC for all analytical methods is presented in Worksheet #22.

- **Field Instrument Calibration:** These procedures are described in Worksheet #22.

- **Equipment Decontamination**: All reusable sampling equipment (e.g., stainless steel trowels, etc.) will be decontaminated prior to sampling and between samples, according to the sequence established in SOP-04 (Appendix A).

- **Investigation-Derived Waste Tasks:** It is anticipated that waste materials will be generated during the field investigation. These wastes must be disposed in such a manner that does not contribute to further environmental contamination or pose a threat to public health or safety. SOP-09 located in Appendix A provides information on the handling of investigation-derived waste.

- **Global Positioning System (GPS):** A GPS survey will be used to locate all sampling points.

- **XRF Analysis:** On-site field XRF analysis for lead. Analysis will be performed according to SOP-13. Twenty percent, or a minimum of 20 samples from each site will be sent to the fixed based laboratory for confirmation analysis.

Analytical Tasks - Chemical analysis for Select Metals (antimony, arsenic, copper, lead, tin, and zinc) and PAHs will be performed by the subcontracted laboratory, Katahdin Analytical Services. Nitroglycerin analysis will be performed by the subcontracted laboratory, Analytical Laboratory Services, Inc. Katahdin Analytical Services and Analytical Laboratory Services, Inc. are NFESC approved and NELAP accredited. Analyses will be performed in accordance with the analytical methods identified in Worksheet #30. Katahdin and Analytical Laboratory Services will meet the PQLs specified in Worksheet #15. Katahdin and Analytical Laboratory Services will perform the chemical analyses following laboratory-specific SOPs (Worksheets #19 and #23) developed based on the methods listed in Worksheets #19 and #30. Copies of the SOPs are included in Appendix D on the attached CD.

Data Management

- Project documentation and records
  - Field sample collection and field measurement records are described in Worksheets #27 and #29.
  - Laboratory data package deliverables are described in the analytical specifications.
  - Data assessment documents and records are listed in Worksheet #29.
- Data recording formats are described in Worksheet #27.

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

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

- **Data Tracking**. Data is tracked from its generation to its archiving in the Tetra Tech project-specific files. The Tetra Tech 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 Tetra Tech 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 Tetra Tech.

- **Data Security**. The Tetra Tech project files are restricted to designated personnel only. Records can only be borrowed temporarily from the project file using a sign-out system. The Tetra Tech Data Manager maintains the electronic data files. Access to the data files is restricted to qualified personnel only. File and data backup procedures are routinely performed.

Assessment and Oversight – Refer to Worksheet #32 for assessment findings and corrective actions and Worksheet #33 for QA management reports.

Data Review

- Data verification is described in Worksheet #34.
- Data validation is described in Worksheets #35 and #36.
- Usability assessment is described in Worksheet #37.

Project Report - Draft and Final versions of project reports will be prepared and submitted to the Navy, EPA, and MDEP for review. The reports will include the following sections:

- Executive Summary – a very brief description of the work conducted and the findings.

- Introduction and Background – includes a description of the history of operations and activities at the Site and a summary of any previous investigations and removal actions.

- Description of Field Investigations – includes a summary of the work performed in the approved SAP and any field modifications as documented by the FOL. This section will include maps showing the sampling locations and well installations and tables summarizing the data collected.

- Data quality – includes a summary of quantitative analytical performance indicators such as completeness, precision, bias and sensitivity, as well as qualitative indicators such as representativeness and comparability. Includes a reconciliation of project data with the DQOs and an identification of deviations from this SAP.

  A data usability assessment will be used to identify significant deviations in analytical performance that could affect the ability to meet project objectives. The elements of this review are presented in Worksheet #37.

- Nature and Extent of Contamination – includes the contamination previously found in each medium sampled in relation to the conceptual model of the site. This section will note the removals previously conducted, the contamination addressed and any additional contaminants found during this effort. Detected contaminant concentrations will be tabulated for each medium and depicted on maps.

- Contaminant Fate and Transport – includes a description of the contaminants detected and their behavior in the soil and bedrock, particularly with emphasis on the future migration of these contaminants to any possible exposure areas.

- Summary and Conclusions – this section will summarize the findings, conclude whether delineation of contamination is adequate, and recommend further sampling if needed.

Tetra Tech will respond to comments received on the draft report. Tetra Tech will submit the draft report before any additional sampling begins. The final version of the report will submitted in hardcopy and electronic format to the project stakeholders.

Risk Assessment reports will be generated using this data as a separate effort after finalization of the data report.

SAP Worksheet #15 -- Reference Limits and Evaluation Table

Matrix: Soil/Sediment

Analytical Group: Inorganics – Select Metals (Fixed-Base Laboratory)

Analyte	CAS Number	Project Action Limit [1] (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-Specific Limits [2]	
					Quantitation Limit (mg/kg)	Method Detection Limit (mg/kg)
Antimony	7440-36-0	0.27	USEPA Eco-SSL	0.27	0.10	0.027
Arsenic	7440-38-2	0.39	ORNL – Res Soil	**0.39**	0.50	0.169
Copper	7440-50-8	100	ORNL-Plant	100	1.0	0.538
Lead	7439-92-1	11	USEPA-SSL	**11**	0.10	0.022
Tin	7440-31-5	0.89	R3 BTAG	0.89	0.10	0.063
Zinc	7440-06-66	46	USEPA-SSL	46	1.0	0.223

Matrix: Soil/Sediment

Analytical Group: Inorganics – Lead (Field Data Collected via Field Portable X-Ray Fluorescence Analyzer)

Analyte	CAS Number	Project Action Limit [1] (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Method Detection Limit [4] (mg/kg)
Lead	7439-92-1	11	USEPA-SSL	11	10

Matrix: Soil/Sediment

Analytical Group: Organics – PAHs

Analyte	CAS Number	Project Action Limit [1] (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific [2]	
					Quantitation Limit (mg/kg)	Method Detection Limit (mg/kg)
1-Methylnaphthalene	90-12-0	22	ORNL – Res Soil	22	0.02	0.0017
2-Methylnaphthalene	91-57-6	31	ORNL – Res Soil	31	0.02	0.0028
Acenaphthene	83-32-9	29	USEPA Eco-SSL	29	0.02	0.0015
Acenaphthylene	208-96-8	29[5]	USEPA Eco-SSL	29[5]	0.02	0.0013
Anthracene	120-12-7	29	USEPA Eco-SSL	29	0.02	0.0026
Benzo(a)anthracene	56-55-3	0.15	ORNL - Res Soil	0.15	0.02	0.0025
Benzo(a)pyrene	50-32-8	0.015	ORNL – Res Soil	**0.015**	0.02	0.0021
Benzo(b)fluoranthene	205-99-2	0.15	ORNL – Res Soil	0.15	0.02	0.0022
Benzo(g,h,i)perylene	191-24-2	1.1[6]	USEPA Eco-SSL	1.1[6]	0.02	0.0033
Benzo(k)fluoranthene	207-08-9	1.1	USEPA Eco-SSL	1.1	0.02	0.0019
Chrysene	218-01-9	1.1	USEPA Eco-SSL	1.1	0.02	0.0026
Dibenzo(a,h)anthracene	53-70-3	0.015	ORNL – Res Soil	**0.015**	0.02	0.0038
Fluoranthene	206-44-0	29	USEPA Eco-SSL	29	0.02	0.004
Fluorene	86-73-7	29	USEPA Eco-SSL	29	0.02	0.0016
Indeno(1,2,3-cd)pyrene	193-39-5	0.15	ORNL – Res Soil	0.15	0.02	0.0043
Naphthalene	91-20-3	0.33	MDE Standard (soil)	0.33	0.02	0.0018
Phenanthrene	85-01-8	29[6]	USEPA Eco-SSL	29[6]	0.02	0.0043
Pyrene	129-00-0	1.1	USEPA Eco-SSL	1.1	0.02	0.0069

Matrix: Soil/Sediment

Analytical Group: Organics – Explosives

Analyte	CAS Number	Project Action Limit [1] (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-specific [9]	
					Quantitation Limit (µg/L)	Instrument Detection Limit (MDL) (µg/L)
Nitroglycerin	55-63-0	6.1	ORNL - Res Soil	1.0	1.0	0.25

Matrix: Aqueous

Analytical Group: Inorganics – Select Metals

Analyte	CAS Number	Project Action Limit [3] (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-specific [2]	
					Quantitation Limit (µg/L)	Instrument Detection Limit (MDL) [4] (µg/L)
Antimony	7440-36-0	1.5	ORNL – Res Soil	1.5	1.0	0.12
Arsenic	7440-38-2	0.045	ORNL – Res Soil	**0.045**	5.0	1.47
Copper	7440-50-8	150	ORNL – Res Soil	150	1.0	.28
Lead	7439-92-1	15	USEPA MCL	15	1.0	.11
Tin	7440-31-5	2.2	MDE Cleanup Standard	2.2	1.0	.30
Zinc	7440-06-66	1100	ORNL – Res Tapwater	1100	10	.90

Matrix: Aqueous

Analytical Group: Organics – PAHs

Analyte	CAS Number	Project Action Limit [3] (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-specific [2]	
					Quantitation Limit (µg/L)	Method Detection Limit (µg/L)
1-Methylnaphthalene	90-12-0	2.3	ORNL – Res Tapwater	2.3	0.20	0.027
2-Methylnaphthalene	91-57-6	15	ORNL – Res Tapwater	15	0.20	0.035
Acenaphthene	83-32-9	37	MDE Cleanup Standard	37	0.20	0.028
Acenaphthylene	208-96-8	37[5]	MDE Cleanup Standard	37[5]	0.20	0.018
Anthracene	120-12-7	180	MDE Cleanup Standard	180	0.20	0.035
Benzo(a)anthracene	56-55-3	0.029	ORNL – Res Tapwater	**0.029**[8]	0.20	0.043
Benzo(a)pyrene	50-32-8	0.0029	USEPA MCL	**0.0029**[8]	0.20	0.080
Benzo(b)fluoranthene	205-99-2	0.029	ORNL – Res Tapwater	**0.029**[8]	0.20	0.078
Benzo(g,h,i)perylene	191-24-2	18[6]	MDE Cleanup Standard	18[6]	0.20	0.072
Benzo(k)fluoranthene	207-08-9	0.29	ORNL – Res Tapwater	0.29	0.20	0.051
Chrysene	218-01-9	2.9	ORNL – Res Tapwater	2.9	0.20	0.093
Dibenzo(a,h)anthracene	53-70-3	0.0029	ORNL – Res Tapwater	**0.0029**[8]	0.20	0.11
Fluoranthene	206-44-0	150	ORNL – Res Tapwater	150	0.20	0.071
Fluorene	86-73-7	24	MDE Cleanup Standard	24	0.20	0.033
Indeno(1,2,3-cd)pyrene	193-39-5	0.029	ORNL – Res Tapwater	**0.029**[8]	0.20	0.085
Naphthalene	91-20-3	0.14	ORNL – Res Tapwater	**0.14**	0.20	0.049
Phenanthrene	85-01-8	110[6]	ORNL – Res Tapwater	110[6]	0.20	0.038
Pyrene	129-00-0	18	MDE Cleanup Standard	18	0.20	0.11

Matrix: Aqueous

Analytical Group: Organics – Explosives

Analyte	CAS Number	Project Action Limit [3] (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-specific [9]	
					Quantitation Limit (µg/L)	Method Detection Limit (µg/L)
Nitroglycerin	55-63-0	0.37	ORNL – Res Tapwater	**0.37**	1.5	0.2

Note: Shaded cells indicate that the project quantitation limit goal is less than the method detection limit.
Bolded cells indicate that the project quantitation limit goal is less than the laboratory quantitation limit.

Abbreviations:
µg/L = micrograms per liter
mg/kg = milligrams per kilograms
MDE Cleanup Standard = State of Maryland, Department of the Environment, Cleanup Standards for Soil and Groundwater, August 2001, Interim Final Guidance (Update No. 1).
MDE Standard (soil) = State of Maryland, Department of the Environment, Cleanup Standards for Soil to Groundwater, August 2001, Interim Final Guidance (Update No. 1).
NA = Not available
ORNL – Res Soil = Oak Ridge National Laboratory Residential Soil Cleanup Standards (USEPA Regional Screening Levels for Chemical Contaminants at Superfund Sites, July 2008).
ORNL – Res Tapwater = Oak Ridge National Laboratory Residential Tapwater Cleanup Standards (USEPA Regional Screening Levels for Chemical Contaminants at Superfund Sites, July 2008).
R3 BTAG = USEPA Region 3 Biology Technical Advisory Group (BTAG) screening levels (soil).
USEPA MCL = USEPA Maximum Contaminant Level, Safe Drinking Water Act, 2006.
USEPA Eco-SSL = USEPA 2005. Guidance for Developing Ecological Soil Screening Levels. Office of Solid Waste and Emergency and Response. OSWER Directive 92857-55. February.

Footnotes:

1 USEPA Regional Screening Levels for Chemical Contaminants at Superfund Sites. The noncarcinogenic values (denoted with an "N" flag on Worksheet #15B) are the RBC divided by 10 to correspond to a target hazard quotient of 0.1. Carcinogenic values represent an incremental cancer risk of 1.0E-06 (carcinogens denoted with a "C" flag on Worksheet #15B) (USEPA Regional, July 2008).

2 The specific laboratory that will be performing work for this project is Katahdin Analytical Services, Inc.

3 Project Action Limits are tap water standards from USEPA Regional Screening Levels for Chemical Contaminants at Superfund Sites. (USEPA Regional, July 7, 2008).

4 Estimated IDL based on SW-846 Method 6010C.

5 The value for acenaphthene has been used as a surrogate for acenaphthylene.

6 The value for pyrene has been used as a surrogate for benzo(g,h,i)perylene and phenanthrene.

7 The laboratory QL for arsenic is greater than the PAL, which is based on 0.1 of the USEPA Regional Screening Levels for tap water. However, the QL is less than the USEPA MCL (15 µg/L) and the Maryland generic cleanup standard of 50 µg/L. For purposes of COPC selection, one half of the laboratory PQL will be used.

8 The laboratory PQL is greater than the PAL, which is based on the USEPA Residential Screening Values for Tap Water. For purposes of COPC selection, one half of the laboratory PQL will be used.

9 The specific laboratory that will be performing nitroglycerin analysis for this project is Analtyical Laboratory Services, Inc.

SAP Worksheet #16 -- Project Schedule / Timeline Table

(UFP-QAPP Manual Section 2.8.2)

Activity	Organization	Start Date(1)	End Date(1)
Prepare Rough Draft SI Work Plan and Appendices	Tetra Tech	10/5/07	3/21/08
Submit Rough Draft SI Work Plan and Appendices	Tetra Tech		3/21/08
Navy Review	Navy	3/24/08	4/10/08
Prepare Draft SI Work Plan and Appendices	Tetra Tech	4/21/08	9/3/08
Submit Draft SI Work Plan and Appendices	Tetra Tech	9/3/08	
Regulator Review	USEPA and MDE	October 08	November 08
Receive Comments/Comment Resolution	Tetra Tech	November 08	December 08
Prepare Final SI Work Plan and Appendices	Tetra Tech	January 09	April 09
Submit Final SI Work Plan and Appendices	Tetra Tech	April 09	
Mobilization and Field Investigation	Tetra Tech	April 09	May 09
Complete Field Investigation and Demobilization	Tetra Tech	May 09	June 09
Laboratory Analysis	Katahdin Analytical Services, Inc.	May 09	July 09
Data Validation	Tetra Tech	July 09	August 09
Database Entry	Tetra Tech	July 09	August 09
Prepare Rough Draft SI Report	Tetra Tech	June 09	December 09
Submit Rough Draft SI Report	Tetra Tech	December 09	
Navy Review	Navy	January 10	February 10
Prepare Draft SI Report	Tetra Tech	February 10	March 10
Submit Draft SI Report	Tetra Tech	March 10	
Regulator Review	USEPA and MDE	March 10	April 10
Receive Comments/Comment Resolution	Tetra Tech	April 10	May 10
Prepare Final SI Report	Tetra Tech	May 10	June 10
Submit Final SI Report	Tetra Tech	July 10	

(1) Actual date shown where applicable, otherwise anticipated month shown.

SAP Worksheet #17 – Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)

This section describes sampling locations, methods, and rationales for the sampling activities to be conducted in support of the field investigations at the five small arms ranges located at the NSF in Indian Head, Maryland. All referenced field SOPs are presented in Appendix A. The majority of proposed soil samples will be collected via hand auger in accordance with SOP-05 and SOP-15, except at the Small Arms (Pistol) Range where proposed soil samples collected in sloughed soil will be via DPT in accordance with SOP-06. The rationale for utilizing DPT at the Small Arms (Pistol) Range is presented below. All surface soil samples will be collected from 0 to 12 inches bgs.

All proposed sediment samples will be collected from 0-to 6-inches in accordance with SOP-08. Proposed sediment samples within Mattawoman Creek will be collected from a boat specifically equipped for this type of sampling and in accordance with SOP-14.

Prior to any field activities, the Tetra Tech FOL will ensure that all field personnel read and understand this UFP-SAP and the associated HASP, and ensure that all non-health and safety-related equipment is available and operational, and the SSO will ensure that all health and safety-related equipment is available and operational.

The sampling objective is to determine specific metals concentrations in soil and sediment as a result of potential leaching of contaminants from lead shot and bullets. And, to a lesser extent, nitroglycerin in soil at the firing lines. All field visual observations including physical observation of lead shot, bullets, and pieces of clay targets will be recorded on sample log sheets. Any bullets, bullet fragments, or lead shot observed in a sample will be removed so as not to bias lead results. Any encounters with metallic objects or other objects that indicate a potential contaminant source or hazard will be reported to the FOL and SSO, and appropriate actions will be taken as specified in this UFP-SAP and associated HASP.

Although MEC are not expected within any of the five investigation sites, if MEC is observed in any boring sample or near any work area, work must be halted. The presence of MEC must be communicated to the FOL, and the FOL will then communicate with the NSF Indian Head Point of Contact (POC) so that appropriate action may be taken. If obvious soil staining is observed in any boring, the staining will be described in the boring log, and additional samples may be collected at the discretion of the FOL or site geologist to determine the nature and possibly the extent of associated site-related contamination.

Soil Sampling Strategy

The chosen sampling strategy employs a transect line sample design to target those areas most likely to be contaminated based on the CSMs presented in Worksheet #10, plus nearby areas that will help to bound the contamination. Composite samples will be collected at the firing lines. The data collected under this conservative strategy are expected to represent concentrations greater than those which human or ecological receptors would actually be exposed. The strategy, therefore, ensures that a potential environmental problem is not overlooked. The sampler will attempt to ensure good spatial coverage of the targeted contamination areas when selecting sampling locations to validate the sampling design and CSMs; areas outside of those expected to be contaminated will also be sampled. If the CSMs are correct, these additional locations will exhibit lesser, and perhaps even non-detectable, concentrations of MC than are found in the targeted contamination areas. Additional sampling strategies can be found under the discussions for each individual site below. All soil borings will be logged to determine lithology. Details regarding soil sampling equipment and procedures are included in Worksheet #14 and SOP-05 and -06 contained in Appendix A.

All soil and sediment samples collected will undergo screening in the field utilizing XRF. Additional sample locations may be added if XRF field screening results greater than the field action level of 300 mg/kg for lead are exhibited in the samples collected from the periphery of the sample collection areas depicted on the maps located under Worksheet #17. The actual sample locations may vary from the proposed locations based on field conditions provided they remain in the estimated range areas as depicted on sample location maps 17.1 through 17.5. Prior to collection of the XRF samples, a site walkover of each site will be conducted to assess whether any lead shot or bullets are visible on the surface or in the very near surface soil at various areas throughout the site. The FOL will use his/her professional judgment and inspection of the site to determine which areas are most likely to be contaminated and adjust the proposed sample locations accordingly.

The total numbers of soil and sediment analyses for each analyte group are tabulated in Worksheet #'s 18 and 20. Soil QA/QC samples will be collected at the frequencies listed in Worksheet #20. Worksheet #19 presents a summary of the sample analyses, container types and volumes, preservation requirements, and holding times for the samples to be collected.

For all sample locations, except along the firing lines within each area, to save time in the field, one surface soil sample (0 to 12 inches bgs) and one subsurface soil sample (12 to 24 inches bgs) will be initially collected at each proposed sample location. Each will be placed in a separate Ziploc plastic baggie, which will then be marked with the sample location ID, depth, date, and time. The samples will

then be transferred back to the field office where the surface soil sample will undergo XRF screening in accordance with SOP-13. Three separate XRF measurements will be made for each sample, and care will be taken to mix the sample in the plastic bag between measurements. The average of the three readings will be computed and recorded as the concentration for the sample. Based on the initial lead XRF concentrations exceeding 300 mg/kg, the FOL will use his judgement in determining the need to collect an additional sample or if it is necessary to "step out" from the sample location to determine the horizontal extent of contamination at that particular location. The subsurface soil sample will be held at the field office and only screened with the XRF if the associated surface soil sample has an average lead XRF concentration greater than the field action level of 300 mg/kg. The results of the field XRF analyses will be the basis for determining which samples will be sent to the fixed-base laboratory for analysis. The FOL will attempt to choose samples representing the range of concentrations observed in the field with the majority of samples in the 400 to 500 mg/kg range. All samples selected for fixed-base laboratory analyses will be prepared and analyzed according to the normal laboratory protocol as identified on Worksheet #30. At the end of the field event(s), any unused portions of the soil samples will be returned to the sample location from which it was collected.

Actual sample locations may vary from the proposed locations based on field conditions observed during an initial site walkover by the FOL. The FOL will use his professional judgment and inspection of the site to determine which areas are most likely to be contaminated and bias the sampling towards those areas. The professional judgment will take into account the topography of the site and the FOL's understanding of site operations. GPS measurements will be taken at each sample location which will allow for future repeatable investigations or guide in any remedial action.

Upon validation, laboratory lead concentrations will be compared to field XRF lead concentrations through a correlation plot. The field data will be plotted against the laboratory data, and a linear least-squares fit will be applied to generate an estimate of the "best" line through the data. If the data set exhibits more than one region where there appears to be a linear fit to the data but the regions have different slopes, the data may be subdivided into separate regions for fitting. No more than three separate regions of the correlation will be fit in this manner, and as few regions as possible will be used to adequately represent a linear fit. If a correlation coefficient (commonly denoted as "*r*") of 0.65 or greater is obtained for a region, the fit will be considered adequate to translate field XRF results to the equivalent laboratory lead concentrations with confidence. A correlation coefficient less than 0.65 will be considered unacceptable for making this translation. An ideal correlation coefficient would be 1.0.

The use of subdivisions of the overall concentrations range to represent a best linear fit will be most important for concentrations in the range of about 250 to 550 mg/kg because the 400 mg/kg risk screening value for lead is within this range.

Assuming that a satisfactory correlation coefficient is obtained, the field XRF data will be translated into equivalent laboratory concentrations, which will then be used for estimating the spatial distribution and magnitude of lead contamination in surface soil. The 250 to 550 mg/kg concentration range will be the most critical range for these translations because it spans the 400 mg/kg concentration representing the RBSL for lead. If the correlation coefficient is less than 0.65 for any region, several courses of action are possible. One course of action may include fixed-base laboratory analysis of additional temperature-controlled samples that will be stored onsite. Because the 250 to 550 mg/kg range is most critical for decision making, a poor correlation at concentrations less than 1,000 mg/kg will be considered more serious than a poor correlation obtained for greater concentrations.

The field XRF for lead and laboratory data for metals and PAHS are intended to be used to establish the nature and extent of contamination and potential environmental mobility. Soil properties such as pH, TOC, and CEC are principal determinants of environmental mobility. Therefore, samples from each of the five sites will be analyzed for these parameters.

At the firing lines, NG and lead are the only contaminants of concern. Beyond the firing lines, lead is the major contaminant of concern at the five sites. The other contaminants (arsenic, antimony, tin, copper, and zinc) at all sites and PAHs (skeet and trap ranges) are expected to be associated with lead. Field XRF data for lead will be used as an indication of potential contamination. Correlations between field XRF results for lead and fixed-base laboratory data will be established.

All sample locations will be marked with a wooden stake or brightly colored pin flag indicating the sample location. Coordinates will be determined by GPS at each individual sample location which will allow for future repeatable investigations or guide in any remedial action. All sample location markers will be removed prior to the final demobilization.

Sampling activities at the following ranges are described in this UFP-SAP:

- UXO 14 – Marine Rifle Range
- UXO 15 – Old Skeet and Trap Range
- UXO 25 – Roach Road Rifle Range

- UXO 16 – Rum Point Skeet Range
- UXO 17 – Small Arms (Pistol) Range

Marine Rifle Range (UXO 14)

Proposed sample locations for the Marine Rifle Range are presented on Map 17-1. Two sets of firing lines, one for each target butt, were located at 100-meter increments to a maximum of 1,000 meters at this range. Composite surface soil samples will be collected within the undeveloped areas at each 100 meter incremental firing line. The exact number of discrete soil cores required for each composite soil sample will be determined in the field by the FOL or designee. The sampling strategy for the impact areas is described below.

Target Berms: Thirty-five soil samples are proposed for collection from each of the two target berms and analyzed for lead in the field using XRF equipment. Thirty of the samples will be collected along three transect lines along the length of each berm. Samples will be evenly spaced along the 250-foot length of the berms. The first transect line will be placed at the base of the target berm where contamination is least likely to occur. The second transect line will be placed approximately at the mid-height of the berm, and the third transect line will be placed along the top of the target berm, which has the highest probability of contamination. The remaining five samples will be evenly spaced in front of each target berm along the range floor where no contamination would be expected to be found.

Hillside Impact Area: Forty-five soil samples are proposed for collection from three transect lines extending along the length of the hillside. Samples will be evenly spaced along the 600-foot length of the hillside. Since the targets were positioned approximately 15 feet above ground surface on the target berms, the first transect line on the hillside will be placed approximately 10 to 12 feet above ground surface on the hillside where impact is least likely to have occurred. The second transect line will be placed approximately 15 to 20 feet above ground surface where the highest probability of impact was likely to occur, and the third transect line will be placed approximately 25 to 30 feet above the ground surface where impact was less likely to have occurred.

Based on field observations and conditions, up to 10 discretionary soil and sediment samples will be collected at the Marine Rifle Range. The FOL will determine if a drainage pathway exists from the target berms leading to the marsh area located east of each berm, and if so, the discretionary samples may be collected in these areas.

All samples collected, except for the firing line samples, will be analyzed in the field for lead utilizing portable XRF equipment. A minimum of 20 percent of these samples (but no fewer than 20) will then be selected for fixed-base laboratory analysis. Samples selected for submittal to the fixed-base laboratory will be analyzed for specific metals as presented in Worksheet #18.1. The composite firing line samples will be submitted to the fixed-base laboratory for NG and lead analysis. Four of the samples will be selected for pH, TOC, and CEC analysis. Five field duplicate samples are proposed for collection at the Marine Rifle Range.

Old Skeet and Trap Range (UXO 15)

Proposed sample locations for the Old Skeet and Trap Range are presented on Map 17-2. Two composite samples will be collected in the firing line area. The exact number of discrete soil cores required for each composite soil sample will be determined in the field by the FOL or designee. The sampling strategy is described below.

The Old Skeet and Trap Range encompasses both land and water. The proposed sediment samples will be collected along the shoreline and in the open waters of Mattawoman Creek.

Fifty-six soil samples are proposed for collection at the Old Skeet and Trap Range. Samples will be spaced approximately 50 feet apart along 11 transect lines starting at the shooters' positions and ending at the shoreline of Mattawoman Creek. Eight of these samples will be collected in the area directly in front of the shooters' positions in an area that would be assumed to be clean.

Thirty-four sediment samples are proposed for collection at the Old Skeet and Trap Range. Nine of those samples will be collected along the shoreline that falls within the shooting arc of the range. These sediment samples will be collected during low tide. The remaining 25 sediment samples will be collected within Mattawoman Creek utilizing a boat specifically equipped for the task (SOP-15). The samples will be collected at various locations within the shot fall arc to the maximum shot distance of approximately 770 feet. Proposed sample locations for the Old Skeet and Trap Range are presented on Map 17-2.

Based on field observations and conditions, up to 10 discretionary soil and sediment samples will be collected at the Old Skeet and Trap Range.

All samples collected, except for the firing line samples, will be analyzed in the field for lead utilizing portable XRF equipment. A minimum of 20 percent of these samples (but no fewer than 20) will then be selected for fixed-base laboratory analysis. Samples selected for submittal to the fixed-base laboratory

will be analyzed for specific metals and PAHs as presented in Worksheet #18.2. The two composite firing line samples will be submitted to the fixed-base laboratory for NG and lead analysis. Four of the samples will be selected for pH, TOC, and CEC analysis. Five field duplicate samples are proposed for collection at the Old Skeet and Trap Range.

Roach Road Rifle Range (UXO 25)

The Roach Road Rifle Range consists of one primary impact area consisting of the former target area and the hillside located directly behind the intended targets. It is believed that the intended target areas no longer exist on site. A secondary area within the Rifle Range consists of the range floor. Proposed sample locations for the Roach Road Rifle Range are presented on Map 17-3. Two composite samples will be collected in the firing line area. The exact number of discrete soil cores required for each composite soil sample will be determined in the field by the FOL or designee. Except for the firing line samples, all soil and sediment samples selected for submittal to the fixed-base laboratory will be analyzed for specified metals as indicated in Worksheet #18.3. The sampling strategies for the impact area and range floor are described below.

Impact Hillside and Former Target Berms: Thirty-two soil samples are proposed for collection from the area where the former target berms were located and from the impact hillside that is directly behind the former target areas. These samples will be analyzed for lead in the field using XRF equipment. Eighteen of the samples will be collected along three transect lines along the length of the impact hillside. Samples will be spaced approximately 10 feet apart along the 65-foot length of the hillside. The first transect line will be placed at the base of the hillside where contamination is least likely expected to occur. The second transect line will be placed approximately 8 to 10 feet above the base of the hillside, and the third transect line will be placed approximately 14 to 16 feet above the base of the hillside. The remaining seven samples will be evenly placed in the areas where the former target berms are believed to have been located.

Based on field observations and conditions, up to 10 discretionary soil and sediment samples will be collected at the Roach Road Rifle Range.

All samples collected, except for the firing line samples, will be analyzed in the field for lead utilizing portable XRF equipment. A minimum of 20 percent of these samples (but no fewer than 20) will then be selected for fixed-base laboratory analysis. Samples selected for submittal to the fixed-base laboratory will be analyzed for specific metals as presented in Worksheet #18.3. The two composite firing line samples will be submitted to the fixed-base laboratory for NG and lead analysis. Four of the samples will

be selected for pH, TOC, and CEC analysis. Two field duplicate samples are proposed for collection at the Roach Road Rifle Range.

Rum Point Skeet Range (UXO 16)

Proposed sample locations for the Rum Point Skeet Range are presented on Map 17-4. Two composite samples will be collected, one from the left firing line area and one from the right firing line area. The exact number of discrete soil cores required for each composite soil sample will be determined in the field by the FOL or designee. The sampling strategy is described below.

Eighty-two soil samples are proposed for collection at the Rum Point Skeet Range. Samples will be spaced approximately 50 feet apart along 11 transect lines starting at the shooters' positions. The Rum Point Skeet Range consists of open field and heavily forested areas. The tree line begins approximately 250 feet north and west of the shooters' positions and approximately 425 feet east and northeast of the shooters' positions. The trees would significantly inhibit the flight of most lead shot and clay targets; therefore, XRF concentrations up to the tree line and the professional judgment of the FOL will determine how far into the tree line samples will be collected. Proposed sample locations for the Rum Point Skeet Range are presented on Map 17-4.

Based on field observations and conditions, up to 10 discretionary soil and sediment samples will be collected at the Rum Point Skeet Range.

All samples collected, except for the firing line samples, will be analyzed in the field for lead utilizing portable XRF equipment. A minimum of 20 percent of these samples (but no fewer than 20) will then be selected for fixed-base laboratory analysis. Samples selected for submittal to the fixed-base laboratory will be analyzed for specific metals and PAHs as presented in Worksheet #18.4. The two composite firing line samples will be submitted to the fixed-base laboratory for NG and lead analysis. Four of the samples will be selected for pH, TOC, and CEC analysis. Four field duplicate samples are proposed for collection at the Roach Road Rifle Range.

Small Arms (Pistol) Range (UXO 17)

Proposed sample locations for the Small Arms (Pistol) Range are presented on Map 17-5. Three composite samples will be collected, one from each firing line area. The exact number of discrete soil cores required for each composite soil sample will be determined in the field by the FOL or designee. The sampling strategy is described below.

Thirty-four soil samples are proposed for collection in the area of the Small Arms (Pistol) Range. Twenty-one of these samples will be collected within the embankment in front of which the targets were placed. Sloughing of this embankment face has occurred at the site and therefore any possible lead contamination may have been buried deeper than would be expected at other similar ranges. All samples within the sloughed soil will be collected via DPT. Actual depths of samples will be determined in the field based on the amount of sloughing that has occurred. Seven samples will be collected along a transect line on the open hillside face approximately 5 to 8 feet above ground surface. The remaining six samples will be placed directly in front of the sloughed soil to ensure coverage of any lead shot that may have been washed onto this part of the range floor. Proposed sample locations for the Small Arms (Pistol) Range are presented on Map 17-5.

Based on field observations and conditions, up to 10 discretionary soil and sediment samples will be collected at the Rum Point Skeet Range.

All samples collected, except for the firing line samples, will be analyzed in the field for lead utilizing portable XRF equipment. A minimum of 20 percent of these samples (but no fewer than 20) will then be selected for fixed-base laboratory analysis. Samples selected for submittal to the fixed-base laboratory will be analyzed for specific metals as presented in Worksheet #18.5. The three composite firing line samples will be submitted to the fixed-base laboratory for NG and lead analysis. Four of the samples will be selected for pH, TOC, and CEC analysis. Two field duplicate samples are proposed for collection at the Small Arms (Pistol) Range.

SAP Worksheet #18.1 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

MARINE RIFLE RANGE (UXO 14)

Sample Location[1]	Sample ID[2]	Metals		NG and Lead	Miscellaneous
		Field XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050B/6020A	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
Soil					
X14SB001	X14SS0010001	1	TBD[3]	NA[5]	TBD[4]
X14SB002	X14SS0020001	1	TBD[3]	NA[5]	TBD[4]
X14SB003	X14SS0030001	1	TBD[3]	NA[5]	TBD[4]
X14SB004	X14SS0040001	1	TBD[3]	NA[5]	TBD[4]
X14SB005	X14SS0050001	1	TBD[3]	NA[5]	TBD[4]
X14SB006	X14SS0060001	1	TBD[3]	NA[5]	TBD[4]
X14SB007	X14SS0070001	1	TBD[3]	NA[5]	TBD[4]
X14SB008	X14SS0080001	1	TBD[3]	NA[5]	TBD[4]
X14SB009	X14SS0090001	1	TBD[3]	NA[5]	TBD[4]
X14SB010	X14SS0100001	1	TBD[3]	NA[5]	TBD[4]
X14SB011	X14SS0110001	1	TBD[3]	NA[5]	TBD[4]
X14SB012	X14SS0120001	1	TBD[3]	NA[5]	TBD[4]
X14SB013	X14SS0130001	1	TBD[3]	NA[5]	TBD[4]
X14SB014	X14SS0140001	1	TBD[3]	NA[5]	TBD[4]
X14SB015	X14SS0150001	1	TBD[3]	NA[5]	TBD[4]
X14SB016	X14SS0160001	1	TBD[3]	NA[5]	TBD[4]
X14SB017	X14SS0170001	1	TBD[3]	NA[5]	TBD[4]
X14SB018	X14SS0180001	1	TBD[3]	NA[5]	TBD[4]
X14SB019	X14SS0190001	1	TBD[3]	NA[5]	TBD[4]
X14SB020	X14SS0200001	1	TBD[3]	NA[5]	TBD[4]
Field Duplicate		1	TBD[3]	NA[5]	TBD[4]

Sample Location[1]	Sample ID[2]	Metals		NG and Lead	Miscellaneous
		Field XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050B/6020A	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
X14SB021	X14SS0210001	1	TBD[3]	NA[5]	TBD[4]
X14SB022	X14SS0220001	1	TBD[3]	NA[5]	TBD[4]
X14SB023	X14SS0230001	1	TBD[3]	NA[5]	TBD[4]
X14SB024	X14SS0240001	1	TBD[3]	NA[5]	TBD[4]
X14SB025	X14SS0250001	1	TBD[3]	NA[5]	TBD[4]
X14SB026	X14SS0260001	1	TBD[3]	NA[5]	TBD[4]
X14SB027	X14SS0270001	1	TBD[3]	NA[5]	TBD[4]
X14SB028	X14SS0280001	1	TBD[3]	NA[5]	TBD[4]
X14SB029	X14SS0290001	1	TBD[3]	NA[5]	TBD[4]
X14SB030	X14SS0300001	1	TBD[3]	NA[5]	TBD[4]
X14SB031	X14SS0310001	1	TBD[3]	NA[5]	TBD[4]
X14SB032	X14SS0320001	1	TBD[3]	NA[5]	TBD[4]
X14SB033	X14SS0330001	1	TBD[3]	NA[5]	TBD[4]
X14SB034	X14SS0340001	1	TBD[3]	NA[5]	TBD[4]
X14SB035	X14SS0350001	1	TBD[3]	NA[5]	TBD[4]
X14SB036	X14SS0360001	1	TBD[3]	NA[5]	TBD[4]
X14SB037	X14SS0370001	1	TBD[3]	NA[5]	TBD[4]
X14SB038	X14SS0380001	1	TBD[3]	NA[5]	TBD[4]
X14SB039	X14SS0390001	1	TBD[3]	NA[5]	TBD[4]
X14SB040	X14SS0400001	1	TBD[3]	NA[5]	TBD[4]
Field Duplicate		1	TBD[3]	NA[5]	TBD[4]
X14SB041	X14SS0410001	1	TBD[3]	NA[5]	TBD[4]
X14SB042	X14SS0420001	1	TBD[3]	NA[5]	TBD[4]
X14SB043	X14SS0430001	1	TBD[3]	NA[5]	TBD[4]
X14SB044	X14SS0440001	1	TBD[3]	NA[5]	TBD[4]
X14SB045	X14SS0450001	1	TBD[3]	NA[5]	TBD[4]
X14SB046	X14SS0460001	1	TBD[3]	NA[5]	TBD[4]
X14SB047	X14SS0470001	1	TBD[3]	NA[5]	TBD[4]

Sample Location[1]	Sample ID[2]	Metals		NG and Lead	Miscellaneous
		Field XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050B/6020A	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
X14SB048	X14SS0480001	1	TBD[3]	NA[5]	TBD[4]
X14SB049	X14SS0490001	1	TBD[3]	NA[5]	TBD[4]
X14SB050	X14SS0500001	1	TBD[3]	NA[5]	TBD[4]
X14SB051	X14SS0510001	1	TBD[3]	NA[5]	TBD[4]
X14SB052	X14SS0520001	1	TBD[3]	NA[5]	TBD[4]
X14SB053	X14SS0530001	1	TBD[3]	NA[5]	TBD[4]
X14SB054	X14SS0540001	1	TBD[3]	NA[5]	TBD[4]
X14SB055	X14SS0550001	1	TBD[3]	NA[5]	TBD[4]
X14SB056	X14SS0560001	1	TBD[3]	NA[5]	TBD[4]
X14SB057	X14SS0570001	1	TBD[3]	NA[5]	TBD[4]
X14SB058	X14SS0580001	1	TBD[3]	NA[5]	TBD[4]
X14SB059	X14SS0590001	1	TBD[3]	NA[5]	TBD[4]
X14SB060	X14SS0600001	1	TBD[3]	NA[5]	TBD[4]
Field Duplicate		1	TBD[3]	NA[5]	TBD[4]
X14SB061	X14SS0610001	1	TBD[3]	NA[5]	TBD[4]
X14SB062	X14SS0620001	1	TBD[3]	NA[5]	TBD[4]
X14SB063	X14SS0630001	1	TBD[3]	NA[5]	TBD[4]
X14SB064	X14SS0640001	1	TBD[3]	NA[5]	TBD[4]
X14SB065	X14SS0650001	1	TBD[3]	NA[5]	TBD[4]
X14SB066	X14SS0660001	1	TBD[3]	NA[5]	TBD[4]
X14SB067	X14SS0670001	1	TBD[3]	NA[5]	TBD[4]
X14SB068	X14SS0680001	1	TBD[3]	NA[5]	TBD[4]
X14SB069	X14SS0690001	1	TBD[3]	NA[5]	TBD[4]
X14SB070	X14SS0700001	1	TBD[3]	NA[5]	TBD[4]
X14SB071	X14SS0710001	1	TBD[3]	NA[5]	TBD[4]
X14SB072	X14SS0720001	1	TBD[3]	NA[5]	TBD[4]
X14SB073	X14SS0730001	1	TBD[3]	NA[5]	TBD[4]
X14SB074	X14SS0740001	1	TBD[3]	NA[5]	TBD[4]

Sample Location[1]	Sample ID[2]	Metals		NG and Lead	Miscellaneous
		Field XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050B/6020A	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
X14SB075	X14SS0750001	1	TBD[3]	NA[5]	TBD[4]
X14SB076	X14SS0760001	1	TBD[3]	NA[5]	TBD[4]
X14SB077	X14SS0770001	1	TBD[3]	NA[5]	TBD[4]
X14SB078	X14SS0780001	1	TBD[3]	NA[5]	TBD[4]
X14SB079	X14SS0790001	1	TBD[3]	NA[5]	TBD[4]
X14SB080	X14SS0800001	1	TBD[3]	NA[5]	TBD[4]
Field Duplicate		1	TBD[3]	NA[5]	TBD[4]
Total Soil Samples - Marine Rifle Range		**85**	**TBD[3]**	**NA[5]**	**TBD[4]**
Discretionary Samples[6]					
Soil					
X14SB086	X14SS0860001	1	TBD[3]	NA[5]	TBD[4]
X14SB087	X14SS0870001	1	TBD[3]	NA[5]	TBD[4]
X14SB088	X14SS0880001	1	TBD[3]	NA[5]	TBD[4]
X14SB089	X14SS0890001	1	TBD[3]	NA[5]	TBD[4]
X14SB090	X14SS0900001	1	TBD[3]	NA[5]	TBD[4]
X14SB091	X14SS0910001	1	TBD[3]	NA[5]	TBD[4]
X14SB092	X14SS0920001	1	TBD[3]	NA[5]	TBD[4]
X14SB093	X14SS0930001	1	TBD[3]	NA[5]	TBD[4]
X14SB094	X14SS0940001	1	TBD[3]	NA[5]	TBD[4]
X14SB095	X14SS0950001	1	TBD[3]	NA[5]	TBD[4]
Total Discretionary Soil Samples - Marine Rifle Range		**10**	**TBD[3]**	**NA[5]**	**TBD[4]**
Sediment					
X14SW/SD001	X14SD0010006	1	TBD[3]	--	TBD[4]
X14SW/SD002	X14SD0020006	1	TBD[3]	--	TBD[4]
X14SW/SD003	X14SD0030006	1	TBD[3]	--	TBD[4]
X14SW/SD004	X14SD0040006	1	TBD[3]	--	TBD[4]
X14SW/SD005	X14SD0050006	1	TBD[3]	--	TBD[4]

Sample Location[1]	Sample ID[2]	Metals		NG and Lead	Miscellaneous
		Field XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050B/6020A	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
X14SW/SD006	X14SD0060006	1	TBD[3]	--	TBD[4]
X14SW/SD007	X14SD0070006	1	TBD[3]	--	TBD[4]
X14SW/SD008	X14SD0080006	1	TBD[3]	--	TBD[4]
X14SW/SD009	X14SD0090006	1	TBD[3]	--	TBD[4]
X14SW/SD010	X14SD0100006	1	TBD[3]	--	TBD[4]
Total Discretionary Sediment Samples - Marine Rifle Range		**10**	**TBD[3]**	**--**	**TBD[4]**

CEC – Cation exchange capacity.
TBD - To be determined.
TOC - Total organic carbon.

1 X = UXO
2 Sample depth to a maximum of 12 inches bgs for surface soil and 6 inches bgs for sediment. Samples will be collected in accordance with their respective SOP (soil SOP-05 and sediment SOP-08).
3 Dependent on field XRF screenings, up to 30 soil samples and one sediment sample will be selected for specific metals analysis at a fixed-base laboratory.
4 Up to four of the samples (soil and/or sediment) submitted to the fixed-base laboratory will also undergo pH, TOC, and CEC analysis.
5 The composite samples collected at the firing line will undergo laboratory analysis for nitroglycerin and lead.
6 Based on field observations and conditions at the time of sampling, up to 10 discretionary soil and sediment samples may be collected.

SAP Worksheet #18.2 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

OLD SKEET AND TRAP RANGE (UXO 15)

Sample Location[1]	Sample ID[2]	Metals		NG and Lead	SVOC	Miscellaneous
		Field XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050B/6020A	SW-846 8270 (PAH)	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
Soil						
X15SB001	X15SS0010001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB002	X15SS0020001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB003	X15SS0030001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB004	X15SS0040001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB005	X15SS0050001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB006	X15SS0060001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB007	X15SS0070001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB008	X15SS0080001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB009	X15SS0090001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB010	X15SS0100001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB011	X15SS0110001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB012	X15SS0120001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB013	X15SS0130001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB014	X15SS0140001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB015	X15SS0150001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB016	X15SS0160001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB017	X15SS0170001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB018	X15SS0180001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB019	X15SS0190001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB020	X15SS0200001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
Field Duplicate		1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB021	X15SS0210001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB022	X15SS0220001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]

Sample Location[1]	Sample ID[2]	Metals		NG and Lead	SVOC	Miscellaneous
		Field XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050B/6020A	SW-846 8270 (PAH)	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
X15SB023	X15SS0230001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB024	X15SS0240001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB025	X15SS0250001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB026	X15SS0260001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB027	X15SS0270001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB028	X15SS0280001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB029	X15SS0290001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB030	X15SS0300001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB031	X15SS0310001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB032	X15SS0320001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB033	X15SS0330001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB034	X15SS0340001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB035	X15SS0350001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB036	X15SS0360001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB037	X15SS0370001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB038	X15SS0380001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB039	X15SS0390001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB040	X15SS0400001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
Field Duplicate		1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB041	X15SS0410001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB042	X15SS0420001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB043	X15SS0430001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB044	X15SS0440001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB045	X15SS0450001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB046	X15SS0460001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB047	X15SS0470001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB048	X15SS0480001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB049	X15SS0490001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]

Sample Location[1]	Sample ID[2]	Metals		NG and Lead	SVOC	Miscellaneous
		Field XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050B/6020A	SW-846 8270 (PAH)	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
X15SB050	X15SS0500001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB051	X15SS0510001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB052	X15SS0520001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB053	X15SS0530001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB054	X15SS0540001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB055	X15SS0550001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB056	X15SS0560001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
Field Duplicate		1	TBD[3]	NA[6]	TBD[4]	TBD[5]
Total Soil Samples - Old Skeet and Trap Range		**59**	**TBD[3]**	**NA[6]**	**TBD[4]**	**TBD[5]**
Sediment						
X15SW/SD001	X15SD0010006	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SW/SD002	X15SD0020006	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SW/SD003	X15SD0030006	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SW/SD004	X15SD0040006	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SW/SD005	X15SD0050006	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SW/SD006	X15SD0060006	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SW/SD007	X15SD0070006	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SW/SD008	X15SD0080006	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SW/SD009	X15SD0090006	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SW/SD010	X15SD0100006	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SW/SD011	X15SD0110006	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SW/SD012	X15SD0120006	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SW/SD013	X15SD0130006	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SW/SD014	X15SD0140006	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SW/SD015	X15SD0150006	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SW/SD016	X15SD0160006	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SW/SD017	X15SD0170006	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SW/SD018	X15SD0180006	1	TBD[3]	NA[6]	TBD[4]	TBD[5]

Sample Location[1]	Sample ID[2]	Metals		NG and Lead	SVOC	Miscellaneous
		Field XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050B/6020A	SW-846 8270 (PAH)	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
X15SW/SD019	X15SD0190006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD020	X15SD0200006	1	TBD[3]	--	TBD[4]	TBD[5]
Field Duplicate		1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD021	X15SD0210006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD022	X15SD0220006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD023	X15SD0230006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD024	X15SD0240006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD025	X15SD0250006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD026	X15SD0260006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD027	X15SD0270006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD028	X15SD0280006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD029	X15SD0290006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD030	X15SD0300006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD031	X15SD0310006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD032	X15SD0320006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD033	X15SD0330006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD034	X15SD0340006	1	TBD[3]	--	TBD[4]	TBD[5]
Field Duplicate		1	TBD[3]	--	TBD[4]	TBD[5]
Total Sediment Samples - Old Skeet and Trap Range		**36**	**TBD[3]**	**--**	**TBD[4]**	**TBD[5]**
Discretionary Samples[7]						
Soil						
X15SB057	X15SS0570001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB058	X15SS0580001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB059	X15SS0590001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB060	X15SS0600001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB061	X15SS0610001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB062	X15SS0620001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB063	X15SS0630001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]

Sample Location[1]	Sample ID[2]	Metals		NG and Lead	SVOC	Miscellaneous
		Field XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050B/6020A	SW-846 8270 (PAH)	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
X15SB064	X15SS0640001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB065	X15SS0650001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X15SB066	X15SS0660001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
Total Discretionary Soil Samples - Old Skeet and Trap Range		**10**	**TBD[3]**	**NA[6]**	**TBD[4]**	**TBD[5]**
Sediment						
X15SW/SD035	X15SD0350006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD036	X15SD0360006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD037	X15SD0370006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD038	X15SD0380006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD039	X15SD0390006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD040	X15SD0400006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD041	X15SD0420006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD042	X15SD0420006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD043	X15SD0430006	1	TBD[3]	--	TBD[4]	TBD[5]
X15SW/SD044	X15SD0440006	1	TBD[3]	--	TBD[4]	TBD[5]
Total Discretionary Sediment Samples - Old Skeet and Trap		**10**	**TBD[3]**	**--**	**TBD[4]**	**TBD[5]**

CEC = Cation exchange capacity.
TOC = Total organic carbon.
TBD = To be determined.

1 X = UXO.
2 Sample depth to a maximum of 12 inches bgs for surface soil and 6 inches bgs for sediment. Samples will be collected in accordance with their respective SOP (soil SOP-05 and sediment SOP-08).
3 Dependent on field XRF screenings, up to 30 soil samples and up to 20 sediment samples will be selected for specified metals analysis at a fixed-base laboratory.
4 Of the samples selected for fixed-base laboratory analysis, up to 20 will be analyzed for PAHs.
5 Up to four samples (soil and/or sediment) will be selected for pH, TOC, and CEC analysis at a fixed-base laboratory.
6 The composite samples collected from the firing line will undergo laboratory analysis for nitroglycerin and lead.
7 Based on field observations and conditions at the time of sampling, up to 10 discretionary soil and sediment samples may be collected.

SAP Worksheet #18.3 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

ROACH ROAD RIFLE RANGE (UXO 25)

Sample Location[1]	Sample ID[2]	Metals		NG and Lead	Miscellaneous
		Field XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050B/6020A	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
Soil					
X25SB001	X25SS0010001	1	TBD[3]	NA[5]	TBD[4]
X25SB002	X25SS0020001	1	TBD[3]	NA[5]	TBD[4]
X25SB003	X25SS0030001	1	TBD[3]	NA[5]	TBD[4]
X25SB004	X25SS0040001	1	TBD[3]	NA[5]	TBD[4]
X25SB005	X25SS0050001	1	TBD[3]	NA[5]	TBD[4]
X25SB006	X25SS0060001	1	TBD[3]	NA[5]	TBD[4]
X25SB007	X25SS0070001	1	TBD[3]	NA[5]	TBD[4]
X25SB008	X25SS0080001	1	TBD[3]	NA[5]	TBD[4]
X25SB009	X25SS0090001	1	TBD[3]	NA[5]	TBD[4]
X25SB010	X25SS0100001	1	TBD[3]	NA[5]	TBD[4]
X25SB011	X25SS0110001	1	TBD[3]	NA[5]	TBD[4]
X25SB012	X25SS0120001	1	TBD[3]	NA[5]	TBD[4]
X25SB013	X25SS0130001	1	TBD[3]	NA[5]	TBD[4]
X25SB014	X25SS0140001	1	TBD[3]	NA[5]	TBD[4]
X25SB015	X25SS0150001	1	TBD[3]	NA[5]	TBD[4]
X25SB016	X25SS0160001	1	TBD[3]	NA[5]	TBD[4]
X25SB017	X25SS0170001	1	TBD[3]	NA[5]	TBD[4]
X25SB018	X25SS0180001	1	TBD[3]	NA[5]	TBD[4]
X25SB019	X25SS0190001	1	TBD[3]	NA[5]	TBD[4]
X25SB020	X25SS0200001	1	TBD[3]	NA[5]	TBD[4]
Field Duplicate		1	TBD[3]	NA[5]	TBD[4]
X25SB021	X25SS0210001	1	TBD[3]	NA[5]	TBD[4]

Sample Location[1]	Sample ID[2]	Metals		NG and Lead	Miscellaneous
		Field XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050B/6020A	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
X25SB022	X25SS0220001	1	TBD[3]	NA[5]	TBD[4]
X25SB023	X25SS0230001	1	TBD[3]	NA[5]	TBD[4]
X25SB024	X25SS0240001	1	TBD[3]	NA[5]	TBD[4]
X25SB025	X25SS0250001	1	TBD[3]	NA[5]	TBD[4]
X25SB026	X25SS0260001	1	TBD[3]	NA[5]	TBD[4]
X25SB027	X25SS0270001	1	TBD[3]	NA[5]	TBD[4]
X25SB028	X25SS0280001	1	TBD[3]	NA[5]	TBD[4]
X25SB029	X25SS0290001	1	TBD[3]	NA[5]	TBD[4]
X25SB030	X25SS0300001	1	TBD[3]	NA[5]	TBD[4]
X25SB031	X25SS0310001	1	TBD[3]	NA[5]	TBD[4]
X25SB032	X25SS0320001	1	TBD[3]	NA[5]	TBD[4]
Field Duplicate		1	TBD[3]	NA[5]	TBD[4]
Total Soil Samples - Roach Road Rifle Range		27	TBD[3]	NA[5]	TBD[4]
Discretionary Samples[6]					
Soil					
X25SB033	X25SS0330001	1	TBD[3]	NA[5]	TBD[4]
X25SB034	X25SS0340001	1	TBD[3]	NA[5]	TBD[4]
X25SB035	X25SS0350001	1	TBD[3]	NA[5]	TBD[4]
X25SB036	X25SS0360001	1	TBD[3]	NA[5]	TBD[4]
X25SB037	X25SS0370001	1	TBD[3]	NA[5]	TBD[4]
X25SB038	X25SS0380001	1	TBD[3]	NA[5]	TBD[4]
X25SB039	X25SS0390001	1	TBD[3]	NA[5]	TBD[4]
X25SB040	X25SS0400001	1	TBD[3]	NA[5]	TBD[4]
X25SB041	X25SS0410001	1	TBD[3]	NA[5]	TBD[4]
X25SB042	X25SS0420001	1	TBD[3]	NA[5]	TBD[4]
Total Discretionary Soil Samples - Roach Road Rifle Range		**10**	**TBD[3]**	**NA[5]**	**TBD[4]**

Sample Location[1]	Sample ID[2]	Metals		NG and Lead	Miscellaneous
		Field XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050B/6020A	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
Sediment					
X25SW/SD001	X25SD0010006	1	TBD[3]	NA[5]	TBD[4]
X25SW/SD002	X25SD0020006	1	TBD[3]	NA[5]	TBD[4]
X25SW/SD003	X25SD0030006	1	TBD[3]	NA[5]	TBD[4]
X25SW/SD004	X25SD0040006	1	TBD[3]	NA[5]	TBD[4]
X25SW/SD005	X25SD0050006	1	TBD[3]	NA[5]	TBD[4]
X25SW/SD006	X25SD0060006	1	TBD[3]	NA[5]	TBD[4]
X25SW/SD007	X25SD0070006	1	TBD[3]	NA[5]	TBD[4]
X25SW/SD008	X25SD0080006	1	TBD[3]	NA[5]	TBD[4]
X25SW/SD009	X25SD0090006	1	TBD[3]	NA[5]	TBD[4]
X25SW/SD010	X25SD0100006	1	TBD[3]	NA[5]	TBD[4]
Total Discretionary Sediment Samples - Roach Road Rifle Range		**10**	**TBD[3]**	**NA[5]**	**TBD[4]**

CEC = Cation exchange capacity.
TOC = Total organic carbon.
TBD = To be determined.

1 X = UXO
2 Sample depth to a maximum of 12 inches bgs for surface soil and 6 inches bgs for sediment. Samples will be collected in accordance with their respective SOP (soil SOP-05 and sediment SOP-08).
3 Dependent on field XRF screenings, up to 20 soil samples and one sediment sample will be selected for specified metals analysis at a fixed-base laboratory.
4 Up to four samples (soil and/or sediment) will be selected for pH, TOC, and CEC analysis at a fixed-base laboratory.
5 The composite samples collected from the firing line will undergo laboratory analysis for nitroglycerin and lead.
6 Based on field observations and conditions at the time of sampling, up to 10 discretionary soil and sediment samples may be collected.

SAP Worksheet #18.4 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

RUM POINT SKEET RANGE (UXO 16)

Sample Location[1]	Sample ID[2]	METALS		NG and Lead	SVOC	Miscellaneous
		FIELD XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050B/6020A	SW-846 8270 (PAH)	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
Soil						
X16SB001	X16SS0010001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB002	X16SS0020001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB003	X16SS0030001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB004	X16SS0040001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB005	X16SS0050001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB006	X16SS0060001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB007	X16SS0070001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB008	X16SS0080001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB009	X16SS0090001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB010	X16SS0100001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB011	X16SS0110001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB012	X16SS0120001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB013	X16SS0130001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB014	X16SS0140001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB015	X16SS0150001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB016	X16SS0160001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB017	X16SS0170001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB018	X16SS0180001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB019	X16SS0190001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB020	X16SS0200001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
Field Duplicate		1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB021	X16SS0210001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB022	X16SS0220001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB023	X16SS0230001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]

Sample Location[1]	Sample ID[2]	METALS		NG and Lead	SVOC	Miscellaneous
		FIELD XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050B/6020A	SW-846 8270 (PAH)	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
X16SB024	X16SS0240001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB025	X16SS0250001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB026	X16SS0260001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB027	X16SS0270001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB028	X16SS0280001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB029	X16SS0290001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB030	X16SS0300001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB031	X16SS0310001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB032	X16SS0320001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB033	X16SS0330001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB034	X16SS0340001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB035	X16SS0350001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB036	X16SS0360001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB037	X16SS0370001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB038	X16SS0380001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB039	X16SS0390001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB040	X16SS0400001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
Field Duplicate		1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB041	X16SS0410001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB042	X16SS0420001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB043	X16SS0430001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB044	X16SS0440001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB045	X16SS0450001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB046	X16SS0460001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB047	X16SS0470001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB048	X16SS0480001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB049	X16SS0490001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB050	X16SS0500001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB051	X16SS0510001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB052	X16SS0520001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB053	X16SS0530001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]

Sample Location[1]	Sample ID[2]	METALS		NG and Lead	SVOC	Miscellaneous
		FIELD XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050B/6020A	SW-846 8270 (PAH)	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
X16SB054	X16SS0540001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB055	X16SS0550001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB056	X16SS0560001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB057	X16SS0570001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB058	X16SS0580001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB059	X16SS0590001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB060	X16SS0600001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
Field Duplicate		1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB061	X16SS0610001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB062	X16SS0620001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB063	X16SS0630001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB064	X16SS0640001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB065	X16SS0650001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB066	X16SS0660001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB067	X16SS0670001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB068	X16SS0680001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB069	X16SS0690001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB070	X16SS0700001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB071	X16SS0710001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB072	X16SS0720001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB073	X16SS0730001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB074	X16SS0740001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB075	X16SS0750001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB076	X16SS0760001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB077	X16SS0770001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB078	X16SS0780001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB079	X16SS0790001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB080	X16SS0800001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]

Sample Location[1]	Sample ID[2]	METALS		NG and Lead	SVOC	Miscellaneous
		FIELD XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050B/6020A	SW-846 8270 (PAH)	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
X16SB081	X16SS0810001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB082	X16SS0820001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
Total Soil Samples - Rum Pont Skeet Range		**86**	**TBD[3]**	**NA[6]**	**TBD[4]**	**TBD[5]**
Discretionary Samples[7]						
Soil						
X16SB086	X16SS0860001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB087	X16SS0870001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB088	X16SS0880001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB089	X16SS0890001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB090	X16SS0900001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB091	X16SS0910001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB092	X16SS0920001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB093	X16SS0930001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB094	X16SS0940001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
X16SB095	X16SS0950001	1	TBD[3]	NA[6]	TBD[4]	TBD[5]
Total Discretionary Soil Samples - Rum Point Skeet Range		**10**	**TBD[3]**	**NA[6]**	**TBD[4]**	**TBD[5]**
Sediment						
X16SW/SD001	X16SD0010006	1	TBD[3]	--	TBD[4]	TBD[5]
X16SW/SD002	X16SD0020006	1	TBD[3]	--	TBD[4]	TBD[5]
X16SW/SD003	X16SD0030006	1	TBD[3]	--	TBD[4]	TBD[5]
X16SW/SD004	X16SD0040006	1	TBD[3]	--	TBD[4]	TBD[5]
X16SW/SD005	X16SD0050006	1	TBD[3]	--	TBD[4]	TBD[5]
X16SW/SD006	X16SD0060006	1	TBD[3]	--	TBD[4]	TBD[5]
X16SW/SD007	X16SD0070006	1	TBD[3]	--	TBD[4]	TBD[5]
X16SW/SD008	X16SD0080006	1	TBD[3]	--	TBD[4]	TBD[5]
X16SW/SD009	X16SD0090006	1	TBD[3]	--	TBD[4]	TBD[5]
X16SW/SD010	X16SD0100006	1	TBD[3]	--	TBD[4]	TBD[5]
Total Discretionary Sediment Samples - Rum Point Skeet Range		**10**	**TBD[3]**	--	**TBD[4]**	**TBD[5]**

CEC = Cation exchange capacity.
TAL = Target Analyte List.
TOC = Total organic carbon.
TBD = To be determined.

1 X = UXO
2 Sample depth to a maximum of 6 inches bgs for surface soil and 6 inches bgs for sediment. Samples will be collected in accordance with their respective SOP (soil SOP-05 and sediment SOP-08).
3 Dependent on field XRF screenings, up to 25 soil samples and one sediment sample will be selected for TAL metals analysis at a fixed-base laboratory.
4 Of the samples selected for fixed-base laboratory analysis, up to 20 will be analyzed for PAHs.
5 Up to four samples (soil and/or sediment) will be selected for pH, TOC, and CEC analysis at a fixed-base laboratory.
6 The composite samples collected from the firing line will undergo laboratory analysis for nitroglycerin and lead.
7 Based on field observations and conditions at the time of sampling, up to 10 discretionary soil and sediment samples may be collected.

SAP Worksheet #18.5 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

SMALL ARMS (PISTOL) RANGE (UXO 17)

Sample Location[1]	Sample ID[2]	Metals		NG and Lead	Miscellaneous
		Field XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050/6020A	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
Soil					
X17SB001	X17SS0010001	1	TBD[3]	NA[5]	TBD[4]
	X17SB001XXXX	1	TBD[3]	NA[5]	TBD[4]
	X17SB001XXXX	1	TBD[3]	NA[5]	TBD[4]
X17SB002	X17SS0020001	1	TBD[3]	NA[5]	TBD[4]
	X17SB002XXXX	1	TBD[3]	NA[5]	TBD[4]
	X17SB002XXXX	1	TBD[3]	NA[5]	TBD[4]
X17SB003	X17SS0030001	1	TBD[3]	NA[5]	TBD[4]
	X17SB003XXXX	1	TBD[3]	NA[5]	TBD[4]
	X17SB003XXXX	1	TBD[3]	NA[5]	TBD[4]
X17SB004	X17SS0040001	1	TBD[3]	NA[5]	TBD[4]
	X17SB004XXXX	1	TBD[3]	NA[5]	TBD[4]
	X17SB004XXXX	1	TBD[3]	NA[5]	TBD[4]
X17SB005	X17SS0050001	1	TBD[3]	NA[5]	TBD[4]
	X17SB005XXXX	1	TBD[3]	NA[5]	TBD[4]
	X17SB005XXXX	1	TBD[3]	NA[5]	TBD[4]
X17SB006	X17SS0060001	1	TBD[3]	NA[5]	TBD[4]
	X17SS006XXXX	1	TBD[3]	NA[5]	TBD[4]
	X17SS006XXXX	1	TBD[3]	NA[5]	TBD[4]
X17SB007	X17SS0070001	1	TBD[3]	NA[5]	TBD[4]
	X17SB007XXXX	1	TBD[3]	NA[5]	TBD[4]
	X17SB007XXXX	1	TBD[3]	NA[5]	TBD[4]
Field Duplicate		1	TBD[3]	NA[5]	TBD[4]

Sample Location[1]	Sample ID[2]	Metals		NG and Lead	Miscellaneous
		Field XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050/6020A	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
X17SB008	X17SS0080001	1	TBD[3]	NA[5]	TBD[4]
X17SB009	X17SS0090001	1	TBD[3]	NA[5]	TBD[4]
X17SB010	X17SS0100001	1	TBD[3]	NA[5]	TBD[4]
X17SB011	X17SS0110001	1	TBD[3]	NA[5]	TBD[4]
X17SB012	X17SS0120001	1	TBD[3]	NA[5]	TBD[4]
X17SB013	X17SS0130001	1	TBD[3]	NA[5]	TBD[4]
X17SB014	X17SS0140001	1	TBD[3]	NA[5]	TBD[4]
X17SB015	X17SS0150001	1	TBD[3]	NA[5]	TBD[4]
X17SB016	X17SS0160001	1	TBD[3]	NA[5]	TBD[4]
X17SB017	X17SS0170001	1	TBD[3]	NA[5]	TBD[4]
X17SB018	X17SS0180001	1	TBD[3]	NA[5]	TBD[4]
X17SB019	X17SS0190001	1	TBD[3]	NA[5]	TBD[4]
X17SB020	X17SS0200001	1	TBD[3]	NA[5]	TBD[4]
Field Duplicate		1	TBD[3]	NA[5]	TBD[4]
Total Soil Samples - Small Arms (Pistol) Range		**36**	**TBD[3]**	**NA[5]**	**TBD[4]**
Discretionary Samples[6]					
Soil					
X17SB021	X17SS0310001	1	TBD[3]	NA[5]	TBD[4]
X17SB022	X17SS0320001	1	TBD[3]	NA[5]	TBD[4]
X17SB023	X17SS0330001	1	TBD[3]	NA[5]	TBD[4]
X17SB024	X17SS0340001	1	TBD[3]	NA[5]	TBD[4]
X17SB025	X17SS0350001	1	TBD[3]	NA[5]	TBD[4]
X17SB026	X17SS0360001	1	TBD[3]	NA[5]	TBD[4]
X17SB027	X17SS0370001	1	TBD[3]	NA[5]	TBD[4]
X17SB028	X17SS0380001	1	TBD[3]	NA[5]	TBD[4]
X17SB029	X17SS0390001	1	TBD[3]	NA[5]	TBD[4]
X17SB030	X17SS0400001	1	TBD[3]	NA[5]	TBD[4]
Total Discretionary Soil Samples - Small Arms (Pistol) Range		**10**	**TBD[3]**	**NA[5]**	**TBD[4]**

Sample Location[1]	Sample ID[2]	Metals		NG and Lead	Miscellaneous
		Field XRF (Lead Only)	SW-846 3050B/6020A (Sb, As, Cu, Pb, Sn, Zn)	SW-846 8330B SW-846 3050/6020A	SW-846 9045D (pH), SW-846 9081 (CEC), Lloyd Kahn Method (TOC)
Sediment					
X17SW/SD001	X17SD0010006	1	TBD[3]	NA[5]	TBD[4]
X17SW/SD002	X17SD0020006	1	TBD[3]	NA[5]	TBD[4]
X17SW/SD003	X17SD0030006	1	TBD[3]	NA[5]	TBD[4]
X17SW/SD004	X17SD0040006	1	TBD[3]	NA[5]	TBD[4]
X17SW/SD005	X17SD0050006	1	TBD[3]	NA[5]	TBD[4]
X17SW/SD006	X17SD0060006	1	TBD[3]	NA[5]	TBD[4]
X17SW/SD007	X17SD0070006	1	TBD[3]	NA[5]	TBD[4]
X17SW/SD008	X17SD0080006	1	TBD[3]	NA[5]	TBD[4]
X17SW/SD009	X17SD0090006	1	TBD[3]	NA[5]	TBD[4]
X17SW/SD010	X17SD0100006	1	TBD[3]	NA[5]	TBD[4]
Total Discretionary Sediment Samples - Small Arms (Pistol) Range		**10**	**TBD[3]**	**NA[5]**	**TBD[4]**

CEC = Cation exchange capacity.
TAL = Target Analyte List.
TBD = To be determined.
TOC = Total organic carbon.

1 X = UXO
2 Sample depth to a maximum of 6 inches bgs for surface soil and 6 inches bgs for sediment. Samples will be collected in accordance with their respective SOP (soil SOP-05 and -06, and sediment SOP-08). The XXXX in the nomenclature indicates depth of subsurface soil samples, which will be determined in the field.
3 Dependent on field XRF screenings, up to 20 soil samples and one sediment sample will be selected for specific metals analysis at a fixed-base laboratory.
4 Up to four samples (soil and/or sediment) will be selected for pH, TOC, and CEC analysis at a fixed-base laboratory.
5 The composite samples collected from the firing line will undergo laboratory analysis for nitroglycerin and lead.
6 Based on field observations and conditions at the time of sampling, up to 10 discretionary soil and sediment samples may be collected.

SAP Worksheet #19 -- Analytical SOP Requirements Table

Fixed-Base Laboratory Analyses

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference	Container (number, size, and type)	Sample Volume (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time(1) (preparation / analysis)
Aqueous	Select Metals (Sb, As, Cu, Pb, Sn, Zn)	SW846 6020, 3010/ KAS SOP CA-627, CA-604	Two 100-mL HDPE container	100-mL HDPE container	Nitric acid to pH < 2; Cool to 4° C	6 months
	TCL PAH	SW846 8270 SIM, 3510/KAS SOP CA213, CA502	Two 1-liter glass amber bottles	One 1-liter glass amber bottle	Cool to 4° C	7 days until extraction/40 days to analysis
	Nitroglycerin	SW-846 8330B/09-8330W, 1B-8330	(2) 1000 mL amber glass	1000 mL	Cool 4°C	14 days to extraction/40 days to analysis
Solid	Select Metals (Sb, As, Cu, Pb, Sn, Zn)	SW846 6020, 3050/ KAS SOP CA-627, CA-605	One 4 oz glass soil jar	Same	Cool to 4° C	6 months
	TCL PAH	SW846 8270 SIM, 3550/KAS SOP CA-213, CA512	One 4 oz glass soil jar	Same	Cool to 4° C	14 days until extraction/40 days to analysis
	Nitroglycerin	SW-846 8330B/09-8330B Soil, 1B-8330B	8 ounce glass	30 g	Cool 4°C	14 days to extraction/40 days to analysis
Solid	Total Organic Carbon	Lloyd Kahn/KAS SOP CA-741	One 4 oz glass soil jar	Same	Cool to 4° C	14 Days
Solid	Cation Exchange Capacity	SW846 9081/ KAS SOP CA-737	One 4 oz glass soil jar	Same	Cool to 4° C	14 days until extraction/7 days to analysis
Solid	pH	SW846 9045/ KAS SOP CA-709	One 4 oz glass soil jar	Same	Cool to 4° C	24 Hours

Field Portable X-Ray Fluorescence Analyses

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference	Containers (number, size, and type)	Sample Volume (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[1] (preparation / analysis)
Solid	Lead	Tetra Tech XRF SOP (SOP-13) and Manufacturer SOP	One 1 gallon zip closure bag	Same	Cool to 4° C	6 months

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

HDPE - High-density polyethylene.

SOP - Standard Operating Procedure.

TBD - To be determined.

TCL - Target Compound List polynuclear aromatic hydrocarbons.

XRF - X-ray fluorescence.

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

Fixed-Base Laboratory Analyses

Matrix	Analytical Group	Concentration Level	No. of Sampling Locations [1]	No. of Field Duplicates	No. of Equip. Blanks	Total No. of Samples to Lab
Solid (Soil)	Select Metals	Low to High	125	7	5	144
Solid (Soil)	PAH	Low to Moderate	35	2	2	41
Solid (Soil)	Nitroglycerin	Low	18	1	1	20
Solid (Sediment)	Select Metals	Low to High	24	2	2	30
Solid (Sediment)	PAH	Low to Moderate	10	1	1	12
Solid (Soil)	pH, CEC, TOC	Low to Moderate	18	NA	NA	18
Solid (Sediment)	pH, CEC, TOC	Low to Moderate	2	NA	NA	2

1 If samples are collected at different depths at the same location, count each discrete sampling depth as a separate sampling location or station.

Equip - Equipment
MS/MSD - Matrix Spike/Matrix Spike Duplicate
No. - Number
PAH - Polycyclic Aromatic Hydrocarbons
PT - Proficiency Testing
TBD - To Be Determined

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
SOP-01	Sample Labeling	Tetra Tech	NA	N	Contained in Appendix A
SOP-02	Sample Identification Nomenclature	Tetra Tech	NA	N	Contained in Appendix A
SOP-03	Sample Custody and Documentation of Field Activities	Tetra Tech	Fieldlog book, sample log sheets, boring logs	N	Contained in Appendix A
SOP-04	Decontamination of Field Sampling Equipment	Tetra Tech	Decontamination equipment, scrub brushes, 5-gallon buckets, spray bottles, phosphate-free detergent, deionized (DI) water	N	Contained in Appendix A
SOP-05	Soil Coring and Sampling Using Hand Auger Techniques	Tetra Tech	Stainless steel auger bucket, extension rods, and T-handle	N	Contained in Appendix A
SOP-06	Borehole Advancement and Soil Coring and Sampling Using Direct-Push Technology	Tetra Tech	Drilling rig and accessories	N	Contained in Appendix A
SOP-07	Surface Water Sampling	Tetra Tech	Multi-parameter water quality meter, turbidity meter, peristaltic pump, silicone tubing, 0.45 micron filter	N	Contained in Appendix A
SOP-08	Sediment Sampling	Tetra Tech	Stainless steel or disposable trowels	N	Contained in Appendix A
SOP-09	Management of Investigation-Derived Waste	Tetra Tech	NA	N	Contained in Appendix A
SOP-10	Borehole and Soil Sample Logging	Tetra Tech	NA	N	Contained in Appendix A

Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
SOP-11	Sample Preservation, Packaging, and Shipping	Tetra Tech	NA	N	Contained in Appendix A
SOP-12	Calibration and Care of Water Quality Meters	Tetra Tech	Multi-parameter water quality meter	N	Contained in Appendix A
SOP-13	Use of Field-Portable X-Ray Fluorescence Analysis of Soil and Sediment	Tetra Tech	Portable X-ray fluorescence analyzer and accessories	N	Contained in Appendix A
SOP-14	Large Water Body Sediment Sampling	Tetra Tech	Stainless steel trowel, large stainless steel bowl, clamshell dredge with an 18-foot rope, wide-bodied, flat-bottomed boat powered by an outboard motor	N	Contained in Appendix A
SOP-15	Composite Sampling for Soil and Sediment	Tetra Tech	Stainless steel trowels and mixing bowls	N	Contained 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	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference	Comments
DPT Rig	Testing	Prior to daily use	Pass all operational and safety checks	Correct deficiencies before operating	DPT subcontractor/ FOL	SOP-06	
Multi-Parameter Water Quality Meter	Calibrated in accordance with manufacturer specifications	Prior to daily use	Prepared standards	Charge battery. Clean probes. Replace membrane. Return to vendor for replacement.	FOL	SOP-12	
Turbidity Meter	Calibrated in accordance with manufacturer specifications	Prior to daily use	Prepared standards	Clean vials. Clean unit. Return to vendor for replacement.	FOL	SOP-12	
XRF	Verification/ standardization check in accordance with manufacturer instructions.	Prior to daily use and after every 20 samples throughout the day	Lead recovery to be between 60 and 135 percent of standard.	Restandardize. If still unacceptable, contact manufacturer for possible replacement	FOL	SOP-13	

SAP Worksheet #23 -- Analytical SOP References Table

Fixed-Base Laboratory Analyses

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? (Y/N)
CA-213	PAHs 8270 (SIM)-A-1/14/06Analysis of Semivolatile Organic Compounds By: SW 846 Method 8270 – Modified For Selected Ion Monitoring (SIM), Revision 4, 06/07	Definitive	Solid – PAHs Aqueous – PAHs	GC/MS (SIM)	Katahdin Analytical Services	N
CA-502	Preparation Of Aqueous Samples For Extractable Semivolatile Analysis, Revision 4, 09/07	Definitive	Aqueous – PAHs	Separatory Funnel	Katahdin Analytical Services	N
CA-512	Preparation Of Sediment/Soil Samples By Sonication Using Method 3550 For Subsequent Extractable Semi-Volatiles Analysis, Revision 5, 09/07	Definitive	Solid - PAHs	Sonicator	Katahdin Analytical Services	N

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
CA-604	Acid Digestion of Aqueous Samples By EPA Method 3010 for ICP Analysis of Total or Dissolved Metals, Revision 3, 04/06	Definitive	Aqueous – Sb, As, Cu, Pb, Sn, Zn	Acid Digestion	Katahdin Analytical Services	N
CA-605	Acid Digestion Of Solid Samples By USEPA Method 3050 For Metals Analysis By ICP-AES And GFAA, Revision 2, 03/06	Definitive	Solid – Sb, As, Cu, Pb, Sn, Zn	Acid Digestion	Katahdin Analytical Services	N
CA-627	Trace Metals Analysis By ICP-MS Using USEPA Method 6020Metals Analysis EPA 6010B-I-12/14/06	Definitive	Solid – Sb, As, Cu, Pb, Sn, Zn Aqueous -– Sb, As, Cu, Pb, Sn, Zn	GC/MSICP-MS	TBDKatahdin Analytical Services	N
CA-709	pH Concentration Measurements In Soil Matrices - SW 846 Method 9045, Revision 6, 02/07	Definitive	Solid - pH	pH Meter	Katahdin Analytical Services	N
CA-737	Cation Exchange Capacity, Revision 0, 01/08	Definitive	Solid - CEC	ICP	Katahdin Analytical Services	N

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
CA-741	Determination of Total Organic Carbon in Solids Using the EPA Region II Lloyd Kahn Method, Revision 1, 01/07	Definitive	Solid - TOC	Lloyd Kahn	Katahdin Analytical Services	N
09-8330W	Solid Phase Extraction of Water for the Analysis of Explosives by EPA Method 8330A (HPLC) Revision 3	Definitive	Aqueous Explosives	NA	Analytical Laboratory Services, Inc.	N
09-8330B Soil	Ultrasonication of Solids for the Analysis of Explosives by EPA Method 8330B (HPLC) Revision 0	Definitive	Solid Explosives	NA	Analytical Laboratory Services, Inc.	N
1B-8330	Nitroaromatics and Nitroamines by HPLC with Ultraviolet Detection Revision 7	Definitive	Aqueous and Solid Explosives	HPLC (various)	Analytical Laboratory Services, Inc.	N
19-COC	Standard Operating Procedure for Chain of Custody Entry Revision 4	Definitive	All	NA	Analytical Laboratory Services, Inc.	N

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
19-Rec/Han	Standard Operating Procedure for Sample Receipt/Sample Handling revision 12	Definitive	All	NA	Analytical Laboratory Services, Inc.	N
19-Waste Disposal	Waste Disposal revision 1	Definitive	All	NA	Analytical Laboratory Services, Inc.	N

Field Portable X-Ray Fluorescence Analyses

Tetra Tech NUS, Inc. 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)
SOP-13	SOP for FPXRF	Definitive	Solid – Pb	FPXRF	Tetra Tech	N

FPXRF - Field portable X-ray fluorescence.
GC/MS - Gas chromatography/mass spectrometry.
GC/MS (SIM) - Gas chromatography/mass spectrometry (selective ion monitoring).
ICP - Inductively coupled plasma optical emission sepctroscopy
PAH - Polynuclear aromatic hydrocarbons.
SOP - Standard Operating Procedure.
TBD - To be determined.

SAP Worksheet #24 -- Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
ICP-MS	Tune	Daily prior to calibration	Mass calibration within 0.1 amu of true value, Resolution < 0.9 amu at 10% peak height	Perform necessary equipment maintenance	Analyst, Supervisor	CA-627
	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	
ICP-AES	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	
GCMS-SVOCs - 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	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
	DFTPP Tune	Every 12 hours	Criteria listed in section 7.4 current rev. of SOP CA-204	Retune and/or clean source	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	
pH Meter	Initial Calibration	Once per day	± 0.05 pH units for every buffer	If calibration is not achieved, check meter, buffer solutions, and probe; replace if necessary; repeat calibration	Analyst, Supervisor	CA-709
FPXRF (Field Analysis)	IC - Per manufacturer's recommendations	Per manufacturer's recommendations	Per manufacturer's recommendations	Repeat calibration and/or contact instrument manufacturer	Analyst/supervisor	Instrument Manufacturer Operator's Manual TBD
FPXRF (Field Analysis)	CCV - Per manufacturer's recommendations	One set per 20 samples analyzed after the instrument and method blanks	70%R to 130%R	Per manufacturer's recommendations	Analyst/supervisor	Instrument Manufacturer Operator's Manual TBD
HPLC - Explosives	Minimum of 5 levels of calibration standards; 6 levels are usually run.	As needed, when CCV criteria cannot be met, of following major instrument maintenance.	Average response factor <= 20 %; if a linear fit is used coefficient of determination (r^2) >=0.990.	Determine and correct reason for failure. Repeat calibration.	HPLC Analyst	1B-8330

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
HPLC - Explosives	ICV	Following initial calibration prior to the analysis of samples.	±20% Difference.	Re-analyze once. If still unacceptable repeat calibration.	HPLC Analyst	1B-8330
HPLC – Explosives	CCV	Prior to the analysis of samples and every 12 hours or every 10 field samples, which ever is more frequent.	±20% Difference.	Re-analyze once. If still unacceptable repeat calibration.	HPLC Analyst	1B-8330

CCV - Continuing Calibration Verification.
ICV - Initial Calibration Verification
FPXRF - Field portable X-ray fluorescence.
IC - Initial Calibration.
ICP-AES - Inductively Coupled-Atomic Emission Spectrometry.
ICP-MS - Inductively Coupled-Mass Spectrometry.
HPLC - High Performance Liquid Chromatography

%D - Percent Difference.
%R - Percent Recovery.
%RSD - Percent Relative Standard Deviation.
TBD - To be determined.

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

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP
Lead Analysis (Field) – FPXRF	Energy calibration checks	Lead	Visual/ Analytical	Per manufacturer's recommendations	Per manufacturers recommendations	Per manufacturer's recommendations	Analyst/ supervisor	Tetra Tech SOP-13
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	Corrective Action	Responsible Person	SOP
GCMS- SVOC	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-213
HPLC	Check pressure.	HPLC	Initial Calibration; Initial and Continuing Calibration Verification.	Initial Calibration – Instrument receipt, after major maintenance, when instrument quality control criteria are not met. Initial Calibration Verification - Following initial calibration prior to the analysis of samples. Continuing Calibration Verification – Prior to the analysis of samples and every 12 hours or every 10 field samples, whichever is more frequent.	Refer to Worksheet #24 Instrument Calibration Table HPLC acceptance criteria.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Re-analyze affected analytes. Record activities in maintenance logbook.	HPLC Analyst	1B-8330

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP
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
pH meter	Clean probe	QC standards	probe	As necessary	Refer to SOP	Refer to SOP	Analyst, Department Manager	

FPXRF - Field portable X-ray fluorescence.
IS - Internal Standard.
PAHs - Polynuclear aromatic hydrocarbons.
psi - Pounds per square inch.
SOP - Standard Operating Procedure.
TBD - To be determined.

SAP Worksheet #26 -- Sample Handling System

(UFP-QAPP Manual Appendix A)

SAMPLE HANDLING SYSTEM

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Field Operations Leader/Tetra Tech
Sample Packaging (Personnel/Organization): Field Operations Leader/Tetra Tech
Coordination of Shipment (Personnel/Organization): Field Operations Leader/Tetra Tech
Type of Shipment/Carrier: Overnight via Federal Express
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Receipt Personnel/Katahdin Analytical Services, Inc./Analytical Laboratory Services, Inc.
Sample Custody and Storage (Personnel/Organization): Sample Receipt Personnel/Katahdin Analytical Services, Inc./Analytical Laboratory Services, Inc.
Sample Preparation (Personnel/Organization): Extractions Personnel/Katahdin Analytical Services, Inc./Analytical Laboratory Services, Inc.
Sample Determinative Analysis (Personnel/Organization): Analytical Personnel/Katahdin Analytical Services, Inc./Analytical Laboratory Services, Inc.
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): Katahdin Analytical Services, Inc. at least 60 days/Analytical Laboratory Services, Inc., 24 to 48 hours
Sample Extract/Digestate Storage (No. of days from extraction/digestion): Katahdin Analytical Services, Inc. 1 year/Extracts may be disposed of 90 days after extraction Analytical Laboratory Services, Inc.
Biological Sample Storage (No. of days from sample collection): N/A

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

SAP Worksheet #27 – Sample Custody Requirements Table

(UFP-QAPP Manual Section 3.3.3)

SAMPLE CUSTODY REQUIREMENTS

Field Sample Custody Procedures (sample collections, packaging, ship, and delivery to laboratory): Following sample collections in the appropriate bottle ware, all samples will be immediately placed on ice in a cooler. The glass sample containers will be enclosed in bubble wrap in order to protect the bottle ware during shipment and to prevent cross contamination should a bottle break in transit. The cooler will be secured using duct tape or clear packaging tape along with two signed custody seals. Sample coolers will be delivered to a local courier location for priority overnight delivery to the selected laboratory for analysis.

Laboratory Sample Custody Procedures: KAS SOP SD-902, KAS SOP SD-903, and 19-COC

Sample Identification Procedures: Tetra Tech SOP-02

Chain-of-Custody Procedures: Tetra Tech SOP-03

SAMPLE NOMENCLATURE

Sample Identification

Refer to Worksheet # 18 for how the samples will be labeled.

Also, refer to Worksheet # 20 for how the field QA/QC samples will be labeled.

SAMPLE COLLECTION DOCUMENTATION, HANDLING, TRACKING, AND CUSTODY PROCEDURES

Sample Collection Documentation

Documentation of field observations will be recorded in a field logbook and/or on field log sheets including sample collection logs, boring logs, and monitoring well construction logs. Bound, water-resistant field logbooks will be utilized for this project. All pages of each logbook will be numbered sequentially, and observations will be recorded with indelible ink.

Field sample log sheets will be used to document sample collection details, and other observations and activities will be recorded in the field logbook. Daily instrument calibration will be recorded on instrument calibration logs. Example field forms are included in Appendix C.

For sampling and field activities, the following types of information will be recorded in the field logbook as appropriate:

- Site name and location
- Date and time of logbook entries
- Personnel and their affiliations
- Weather conditions
- Activities associated with sampling
- Subcontractor activity summary
- Site observations including site entry and exit times
- Site sketches made on site
- Visitor names, affiliations, and arrival and departure times
- Health and safety issues including PPE

Sample Handling and Tracking System

Procedures that will be used by field and laboratory personnel to document project activities and sample collection procedures during these SIs are outlined in the following subsections. All forms will be filled in as completely as possible.

Sample Handling

Sample handling is described in SAP Worksheet #26.

Sample Delivery

Samples will be delivered to the laboratory via a public courier (i.e., Federal Express). Samples will be sent to the laboratory within 48 hours of being collected. Under no circumstances should sample holding times be exceeded.

Sample Custody

To ensure the integrity of a sample from collection through analysis, it is necessary to have an accurate, written record that traces the possession and handling of the sample. This documentation is referred to as the COC form. Chain of custody begins at the time of sample collection.

A sample is under custody if:

- It is in your actual possession, or
- It is in your view, after being in your physical possession, or
- It was in your possession and then you locked or sealed it up to prevent tampering, or
- It is in a secure area.

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

Preservation of the integrity of the samples collected during the site investigation will be the responsibility of identified persons from the time the samples are collected until the samples, or their derived data, are incorporated into the final report. Sample custody is described in Worksheet #27.

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

Laboratory Custody Procedures

The following SOPs are saved on CD and describe, in detail, the procedures referenced below:

KAS SOP SD-902 – Sample Receipt and Internal Control
KAS SOP SD-903 – Sample Disposal
19-Rec/Han - Standard Operating Procedure for Sample Receipt/Sample Handling
19-Waste Disposal - Waste Disposal
19-COC - Standard Operating Procedure for Chain of Custody Entry

Typically, samples are received by the laboratories during normal business hours (8:00 am to 6:00 pm), Monday through Friday and, when known in advance, from 8:00 am to noon on Saturday.

Upon sample receipt, the coolers are inspected for the general condition of the Custody Seal, if present. The coolers are then opened and each sample is inspected for damage. The sample containers are removed from the packing material and identities are verified against the Chain-of-Custody. All information regarding sample condition upon receipt is documented on the Sample Receipt Condition Report (SRCR). The report documents:

- Name of person if hand delivered;
- Presence/Absence of COC forms and Custody Seals;
- Condition of the custody seals if present;
- Discrepancies noted;
- Holding times;
- Proper preservation (i.e. pH of all samples, except volatiles samples, is verified). The pH of volatile samples is checked at analysis and recorded in the analytical run log.
- Proper sample containers, properly labeled according to the COC and unbroken;
- Appropriate sample volume; and
- Cooler temperature

The Sample Receipt Condition Report is completed by signing and recording the date and time of sample receipt. If there are any discrepancies or problems with the samples or accompanying documentation, the Sample Custodian immediately notifies the client or the appropriate Katahdin Analytical Services/Analytical Laboratory Services, Inc. project manager for resolution.

After completion of sample analysis and submission of the analytical report, unused portions of samples are retained by the laboratory for a minimum of thirty days. Unless otherwise specified by the client or analytical program, after submission of the data report, samples will be moved from the refrigerators for subsequent disposal according to the nature of the samples. The Katahdin/Analytical Laboratory Services Environmental Health and Safety Officer (EHS) generates a KIMS sample disposal summary and uses that information to select the appropriate waste stream for the samples. Samples determined to be hazardous waste are handled by state and federally licensed hazardous waste disposal firms.

Upon disposal of samples, a record is generated by the Katahdin/Analytical Laboratory Services EHS Officer listing the sample number, inherent waste stream and date disposed. This record is maintained by the Katahdin/Analytical Laboratory Services EHS Officer. Please refer to the current revision of SOP SD-903, Sample Disposal, and 19-Waste Disposal for further information.

SAP Worksheet #28 -- Laboratory QC Samples Table

Matrix	Aqueous
Analytical Group	PAH's (SIM)
Analytical Method/ SOP Reference	SW846 8270 SIM/ CA-213

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch of twenty or fewer samples of similar matrix	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.	Analyst, Supervisor, QA Manager	Bias Contamination	No analytes detected > PQL.
Reagent Blank	1 per Lot	No analyte detected > QL	(1) Investigate source of contamination (2) If required replace Lot	Analyst, Supervisor, QA Manager	Contamination	No analyte detected > QL
Matrix Spike	One per prep batch of twenty or fewer samples of similar matrix	Statistically derived acceptance 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 the samples and QC.	Analyst, Supervisor, QA Manager	Accuracy Bias	Statistically derived acceptance limits.
Matrix Spike Duplicate	One per prep batch of twenty or fewer samples of similar matrix	≤ 50% RPD.	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 the samples and QC.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	≤ 50% RPD.
LCS	One per prep batch of twenty or fewer samples of similar matrix.	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.	Analyst, Supervisor, QA Manager	Accuracy Bias	Statistically derived acceptance limits.
Internal Standards (IS)	Every sample, control, standard, and method blank	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. If reanalysis confirms matrix interference, report sample and narrate.	Analyst, Supervisor, QA Manager	Accuracy Bias	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS

Matrix	Aqueous
Analytical Group	PAH's (SIM)
Analytical Method/ SOP Reference	SW846 8270 SIM/ CA-213

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
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 Surr. Recovered high but sample results < PQL, narrate (4) If still out reextract and analyze sample (5) If reextract 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 #28 -- Laboratory QC Samples Table

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

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch of twenty or fewer samples of similar matrix	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.	Analyst, Supervisor, QA Manager	Bias Contamination	
Reagent Blank	1 per Lot	No analyte detected > QL	(1) Investigate source of contamination (2) If required replace Lot	Analyst, Supervisor, QA Manager	Bias Contamination	
Matrix Spike	One per prep batch of twenty or fewer samples of similar matrix	Statistically derived acceptance 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 the samples and QC.	Analyst, Supervisor, QA Manager	Accuracy Bias	
Matrix Spike Duplicate	One per prep batch of twenty or fewer samples of similar matrix	≤ 50% RPD.	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 the samples and QC.	Analyst, Supervisor, QA Manager	Accuracy Bias Precision	
LCS	One per prep batch of twenty or fewer samples of similar matrix.	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.	Analyst, Supervisor, QA Manager	Accuracy Bias	
Internal Standards (IS)	Every sample, control, standard, and method blank	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. If reanalysis confirms matrix interference, report sample and narrate.	Analyst, Supervisor, QA Manager	Accuracy Bias	

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

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
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 Surr. Recovered high but sample results < PQL, narrate (4) If still out reextract and analyze sample (5) If reextract is out, flag data	Analyst, Supervisor, QA Manager	Accuracy Bias	
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 #28 -- Laboratory QC Samples Table

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

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Independent Calibration Verification (ICV)	Immediately after calibration	± 10 %	Correct problem, recalibrate and reanalyze ICV	Analyst, Supervisor, QA Manager	Accuracy/bias	± 10 %
Initial Calibration Blank (ICB)	Immediately after the ICV	≤ PQL	Correct problem, recalibrate and reanalyze ICV and ICB	Analyst, Supervisor, QA Manager	Accuracy/bias, Contamination	≤ PQL
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	80-120 % 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. 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-	Absolute value < PQL.
Interference Check Solutions (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	
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 ± 10% of the original result.
Laboratory Control Sample (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.

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

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike 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, Precision	Recovery ± 25 % of true value if sample < 4x spike value
Matrix Spike Duplicate	One per prep batch of twenty or fewer samples of similar matrix	Recovery ± 25 % of true value if sample < 4x spike value RPD $\leq$ 20 %	Flag results for affected analytes for all associated samples with "N".	Analyst, Supervisor, QA Manager	Accuracy/Bias, Precision	Recovery ± 25 % of true value if sample < 4x spike value RPD $\leq$ 20 %
Post-Digestion Spike	One per prep batch of 20 or fewer samples	Recovery within 75-125 % expected	Narrate	Analyst, Supervisor, QA Manager	Accuracy/Bias, Precision	Recovery within 75-125 % expected
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 #28 -- Laboratory QC Samples Table

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	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Independent Calibration Verification (ICV)	Immediately after calibration	± 10 %	Correct problem, recalibrate and reanalyze ICV	Analyst, Supervisor, QA Manager	Accuracy/bias	± 10 %
Initial Calibration Blank (ICB)	Immediately after the ICV	≤ PQL	Correct problem, recalibrate and reanalyze ICV and ICB	Analyst, Supervisor, QA Manager	Accuracy/bias, Contamination	≤ PQL
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	80-120 % 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. 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-	Absolute value < PQL.
Interference Check Solutions (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, Precision	
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.
Laboratory Control Sample (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.	Analyst, Supervisor, QA Manager	Accuracy/Bias	80-120 % recovery.
Matrix Spike 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, Precision	Recovery ± 25 % of true value if sample < 4x spike value

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	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicate	One per prep batch of twenty or fewer samples of similar matrix	Recovery ± 25 % of true value if sample < 4x spike value RPD ≤ 20 %	Flag results for affected analytes for all associated samples with "N".	Analyst, Supervisor, QA Manager	Accuracy/Bias, Precision	Recovery ± 25 % of true value if sample < 4x spike value RPD ≤ 20 %
Post-Digestion Spike	One per prep batch of twenty or fewer samples of similar matrix	Recovery within 75-125 % expected	Narrate.	Analyst, Supervisor, QA Manager	Accuracy/Bias, Precision	Recovery within 75-125 % expected
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 #28 -- Laboratory QC Samples Table

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

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Independent Calibration Verification (ICV)	Immediately after calibration	± 10 %	Correct problem, recalibrate and reanalyze ICV	Analyst, Supervisor, QA Manager	Accuracy/bias	± 10 %
Initial Calibration Blank (ICB)	Immediately after the ICV	≤ PQL	Correct problem, recalibrate and reanalyze ICV and ICB	Analyst, Supervisor, QA Manager	Accuracy/bias, Contamination	≤ PQL
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 samoples analyzed since last good PQL Std.	Analyst, Supervisor, QA Manager	Sensitivity	80-120 % recovery .
Preparation Blank (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. (2) 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-	Absolute value < PQL.
Interference Check Solutions (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, Precision	
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 ± 10% of the original result.
Laboratory Control Sample (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.
Matrix Spike 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, Precision	Recovery ± 25 % of true value if sample < 4x spike value

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

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicate	One per prep batch of twenty or fewer samples of similar matrix	Recovery ± 25 % of true value if sample < 4x spike value RPD ≤ 20 %	Flag results for affected analytes for all associated samples with "N".	Analyst, Supervisor, QA Manager	Accuracy/Bias, Precision	Recovery ± 25 % of true value if sample < 4x spike value RPD ≤ 20 %
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.	Narrate	Analyst, Supervisor, QA Manager	Accuracy/Bias, Precision	Recovery ± 25% of true value, if sample < 4x spike added.
Internal Standards	Internal Standard (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 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.
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 #28 -- Laboratory QC Samples Table

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	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Independent Calibration Verification (ICV)	Immediately after calibration	± 10 %	Correct problem, recalibrate and reanalyze ICV	Analyst, Supervisor, QA Manager	Accuracy/bias	± 10 %
Initial Calibration Blank (ICB)	Immediately after the ICV	≤ PQL	Correct problem, recalibrate and reanalyze ICV and ICB	Analyst, Supervisor, QA Manager	Accuracy/bias, Contamination	≤ PQL
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 samoples analyzed since last good PQL Std.	Analyst, Supervisor, QA Manager	Sensitivity	80-120 % 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. 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-	Absolute value < PQL.
Interference Check Solutions (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, Precision	
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.
Laboratory Control Sample (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.	Analyst, Supervisor, QA Manager	Accuracy/Bias	80-120 % recovery.
Matrix Spike 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, Precision	Recovery ± 25 % of true value if sample < 4x spike value

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	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicate	One per prep batch of twenty or fewer samples of similar matrix	Recovery ± 25 % of true value if sample < 4x spike value RPD ≤ 20 %	Flag results for affected analytes for all associated samples with "N".	Analyst, Supervisor, QA Manager	Accuracy/Bias, Precision	Recovery ± 25 % of true value if sample < 4x spike value RPD ≤ 20 %
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.	Narrate	Analyst, Supervisor, QA Manager	Accuracy/Bias, Precision	Recovery ± 25% of true value, if sample < 4x spike added.
Internal Standards	Internal Standard (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 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.
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 #28 -- Laboratory QC Samples Table

Matrix	Soil/Sediment
Analytical Group	Total Organic Carbon
Analytical Method/ SOP Reference	Lloyd Kahn/CA-763

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch of 20 or fewer samples	No TOC detected ≥ QL	Investigate source of contamination Re-analyze all affected samples.	Analyst, Supervisor, QA Manager	Accuracy/bias-Contamination	No TOC > QL
Laboratory Duplicate	One per prep batch of 10 or fewer samples	≤ 20% RPD	Narrate any results that are outside control limits	Analyst, Supervisor, QA Manager	Precision	Precision: ≤ 20% RPD
Matrix Spike	One per prep batch of 10 or fewer samples	75-125 %	No CA will be taken for Samples where recoveries are outside limits and LCS criteria are met. Narrate outages	Analyst, Supervisor, QA Manager	Accuracy/bias	75-125 %
LCS	One per prep batch of 20 or fewer samples	80-120%	Re-analyze associated samples If sample is within holding time, re-analyze affected sample batch. 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

Matrix	Aqueous
Analytical Group	Explosives
Analytical Method / SOP Reference	SW-846 09-8330W; 1B-8330

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per batch of 20 or fewer samples.	Less than ½ QL.	Re-analyze the blank. If still unacceptable investigate the source of contamination. Samples in the batch must be < the QL or ≥ 10x the method blank. If not, samples must be re-analyzed. If re-analysis is not possible, report with a qualifying comment	Analyst.	Bias/Contamination	Less than the QL.

Matrix	Aqueous					
Laboratory Control Sample (LCS)	One per batch of 20 or fewer samples.	Control Limits listed in DoD QSM Table D-12.	Re-analyze once. If still unacceptable re-analyze all associated samples. If re-analysis is not possible, report with a qualifying comment. If the LCS recoveries are biased high and sample results are <QL data is not affected and results are acceptable.	Analyst.	Accuracy/Bias	Control Limits listed in DoD QSM Table D-12.
Matrix Spike (MS)	One per batch of 20 or fewer samples.	Control Limits listed in DoD QSM Table D-12.	If batch Laboratory Control Sample is acceptable flag result. If batch Laboratory Control Sample is not acceptable re-analyze sample and QC.	Analyst	Accuracy/Bias	Control Limits listed in DoD QSM Table D-12.
Matrix Spike Duplicate (MSD) or Sample Duplicate	One per batch of 20 or fewer samples.	Control Limits listed in DoD QSM Table D-12.	Flag results.	Analyst	Precision	Control Limits listed in DoD QSM Table D-12.
Surrogates	One per Sample	± 50% of true value.	If a matrix interference can be identified report with a qualifying comment. If not, re-analyze to confirm matrix interference. If confirmed report with a qualifying comment.	Analyst	Accuracy/Bias	± 50% of true value.

Matrix	Solid
Analytical Group	Explosives
Analytical Method / SOP Reference	09-8330B Soil; 1B-8330

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per batch of 20 or fewer samples.	Less than ½ QL.	Re-analyze the blank. If still unacceptable investigate the source of contamination. Samples in the batch must be < the QL or ≥ 10x the method blank. If not, samples must be re-analyzed. If re-analysis is not possible, report with a qualifying comment	Analyst	Bias/Contamination	Less than the QL.

Matrix	Solid					
Laboratory Control Sample (LCS)	One per batch of 20 or fewer samples.	Control Limits listed in DoD QSM Table D-13.	Re-analyze once. If still unacceptable re-analyze all associated samples. If re-analysis is not possible, report with a qualifying comment. If the LCS recoveries are biased high and sample results are <QL data is not affected and results are acceptable.	Analyst.	Accuracy/Bias	Control Limits listed in DoD QSM Table D-13.
Matrix Spike (MS)	One per batch of 20 or fewer samples.	Control Limits listed in DoD QSM Table D-13.	If batch Laboratory Control Sample is acceptable flag result. If batch Laboratory Control Sample is not acceptable re-analyze sample and QC.	Analyst	Accuracy/Bias	Control Limits listed in DoD QSM Table D-13.
Matrix Spike Duplicate (MSD) or Sample Duplicate	One per batch of 20 or fewer samples.	Control Limits listed in DoD QSM Table D-13.	Flag results.	Analyst	Precision	Control Limits listed in DoD QSM Table D-13.

Matrix	Solid					
Surrogates	One per Sample.	± 50% of true value.	If a matrix interference can be identified report with a qualifying comment. If not, re-analyze to confirm matrix interference. If confirmed report with a qualifying comment.	Analyst	Accuracy/Bias	± 50% of true value.

CCV - Continuing Calibration Verification.
FOL - Field Operation Leader.
IS - Internal Standard.
LCS/LCSD - Laboratory control sample/laboratory control sample duplicate.
MS/MSD - Matrix spike/,atrix spike duplicate.
PAH - Polynuclear Aromatic Hydrocarbons.
PM - Project Manager.
QC - Quality control.
RPD - Relative Percent Difference.
RT - Retention Time.
RTW - Retention Time Window.
SIM - Selective Ion Monitoring.
SOP - Standard Operating Procedure.
TBD - To be determined.
Tetra Tech - Tetra Tech NUS, Inc.

SAP Worksheet #29 -- Project Documents and Records Table

(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
Sample Collection Documents and Records Field logbook (and sampling notes) Field sample forms (e.g. boring logs, sample log sheets, drilling logs, etc.) Chain-of-custody records Sample shipment airbills Equipment calibration logs Photographs Field Task Modification Forms Sampling and Analysis Plan Field Sampling SOPs	Tetra Tech project file, results will be discussed in subject document.
Laboratory Documents and Records Sample receipt/log-in form Sample storage records Sample preparation logs Standard traceability logs Equipment Calibration logs Sample analysis run logs Equipment maintenance, testing, and inspection logs Field Task Modification Reports Reported field sample results Reported results for standards, quality control checks, and quality control samples Data completeness checklists Sample storage and disposal records Telephone logs Extraction/clean-up records Raw data	Tetra Tech Project File, Long-term data package storage at third-party professional document storage firm (BRM), results will be discussed in subject document.
Data Assessment Documents and Records Field Sampling Audit Checklist (if an audit is conducted) Analytical Audit Checklist (if an audit is conducted) Data Validation Memoranda	Tetra Tech project file, results will be discussed in subject document.

SAP Worksheet #30 -- Analytical Services Table

Matrix	Analytical Group	Sample Locations/ID Numbers	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
Aqueous	Select Metals – Sb, As, Cu, Pb, Sn, Zn	See Worksheet #s 18.1 through 18.5	SW-846 6010B	21 calendar days	Katahdin Analytical Services, Inc 600 Technology Way Scarborough, ME 04074 Andrea Colby 207.874.2400	NA
	PAHs	See Worksheet #s 18.1 through 18.5	SW-846 8270C SIM	21 calendar days	Katahdin Analytical Services, Inc 600 Technology Way Scarborough, ME 04074 Andrea Colby 207.874.2400	NA
	Nitroglycerin	See Worksheet #s 18.1 through 18.5	SW-846 8330B	21 calendar days	Scott Brunk Analytical Laboratory Services, Inc. 34 Dogwood Lane Middletown, PA 17057 Phone: 800-794-7709	NA
Solid	Select Metals – Sb, As, Cu, Pb, Sn, Zn	See Worksheet #s 18.1 through 18.5	SW-846 6010B	21 calendar days	Katahdin Analytical Services, Inc 600 Technology Way Scarborough, ME 04074 Andrea Colby 207.874.2400	NA
	PAHs	See Worksheet #s 18.1 through 18.5	SW-846 8270C SIM	21 calendar days	Katahdin Analytical Services, Inc 600 Technology Way Scarborough, ME 04074 Andrea Colby 207.874.2400	NA

Matrix	Analytical Group	Sample Locations/ID Numbers	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
	Nitroglycerin	See Worksheet #s 18.1 through 18.5	SW-846 8330B	21 calendar days	Scott Brunk Analytical Laboratory Services, Inc. 34 Dogwood Lane Middletown, PA 17057 Phone: 800-794-7709	

NA - Not applicable.
PAHs - Polynuclear aromatic hydrocarbons.
SIM - Selective Ion Monitoring.
TBD - To be determined.

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 Corrective Actions (CA) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Laboratory Systems Audit	Every 18 months	External	NFESC	TBD	Laboratory QAM	Laboratory QAM Katahdin Analytical Services, Inc.	(Project Manager) Katahdin Analytical Services, Inc.
Laboratory Systems Audit	Every 18 months	External	NFESC	TBD	Laboratory QAM	Laboratory QAM Analytical Laboratory Services, Inc.	(Project Manager) Analytical Laboratory Services, Inc.
Field Sampling Systems Audit	One per contract year	Internal	Tetra Tech	TBD	PM	Auditor and QAM Tetra Tech	QAM Tetra Tech

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

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Time Frame of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Time Frame for Response
Field Sampling Systems Audit	Audit checklist (as per IRCDQM) and written audit report	PM – Tetra Tech FOL – Tetra Tech Program Management – Tetra Tech	Dependent on findings; if major, a stop work maybe issued immediately; however, if minor, within 1 week of audit	Written memorandum	QAM - Tetra Tech Auditor - Tetra Tech Program Manager – Tetra Tech	Within 4 weeks of notification
Laboratory Systems Audit	Written audit report	Laboratory QAM	Not specified by NFESC	Letter	NFESC	Specified by NFESC

FOL - Field Operations Leader.
IRCDQM - Navy Installation Restoration Chemical Data Quality Manual.
NFESC - Naval Facilities Engineering Service Center.
PM - Project Manager.
QAM - Quality Assurance Manager.

SAP Worksheet #33 -- QA Management Reports Table

(UFP QAPP Manual Section 4.2)

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Data validation report	Per SDG	DVM or designee	DVM or designee	PM (Tetra Tech), project file
Major analysis problem identification (internal memo)	When persistent analysis problems are detected	Immediately	QAM (Tetra Tech)	PM (Tetra Tech), QAM (Tetra Tech), Program Manager, project file
Project monthly progress report	Monthly for duration of project	Monthly	PM (Tetra Tech)	Navy, project file
Field progress reports	Daily, oral, during the course of sampling	Every day that field sampling is occurring	FOL (Tetra Tech)	PM (Tetra Tech)
Laboratory QA report	When significant plan deviations result from unanticipated circumstances	Immediately	PM (Katahdin Analytical Services, Inc. and Analytical Laboratory Services, Inc.)	Tetra Tech, 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)
Sample Tables	Proposed samples verified to have been collected	Internal	FOL or designee (Tetra Tech)
Chain-of-custody forms	Chain-of-custody records will be reviewed internally by the PM or designee and compared to sample tables listing the proposed samples to verify that all planned samples have been collected.	Internal	PM or designee (Tetra Tech)
Sample Coordinates	Sample locations have been verified to be correct and in accordance with the QAPP (overlay maps of proposed locations with maps of actual locations)	Internal	FOL, PM, or designee (Tetra Tech)
Data Package	Verify that the data package contains all the elements required by the functional guidelines and scope of work, this occurs as part of the data validation process.	Internal	Data Validator (Tetra Tech)
Sample Log Sheets	Log sheets completed as samples are collected in the field are verified for completeness and are maintained at the project office.	Internal	PM or designee (Tetra Tech)

FOL – Field Operations Leader
PM – Project Manager

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

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

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Field SOPs/Field Logs/Sample Collection	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and MPCs have been achieved. Particular attention should be given to verify that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken trail of documented chain of custody from sample collection to report generation. Verify that the correct sampling and analytical methods/SOPs were applied. Verify that the sampling plan was implemented and carried out as written and that any deviations are documented.	PM, FOL, or designee (Tetra Tech)
IIa	Analytical SOPs	Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied.	Laboratory QAM (Katahdin Analytical Services, Inc. and Analytical Laboratory Services, Inc.)
IIa	Documentation of Method QC Results	Establish that all method QC samples were analyzed and in control as listed in the analytical SOPs. If method QA is not in control, the laboratory will contact Tetra Tech for guidance prior to report preparation.	PM or designee (Tetra Tech)
IIa, IIb	SAP QC Sample Documentation	Verify that all QC samples specified in the SAP were collected and analyzed and that the associated results were within prescribed SAP acceptance limits. Verify that QC samples and standards prescribed in analytical SOPs were analyzed and within the prescribed control limits. If any significant QC deviations occur, the laboratory shall have contacted the Tetra Tech PM	PM or designee (Tetra Tech)
IIa, IIb	Documentation of Analytical Reports for Completeness	Ensure that the chain-of-custody form generated in the field to delivery of analytical data that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. Validator will verify that elements of the data package required for validation are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet #36. Verify all data have been transferred correctly and completely to the final Structured Query Language (SQL) database.	Data Validator (Tetra Tech)

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIb	Project Quantitation Limits	Verify that detection and quantitation limits prescribed in SAP Worksheet #15 were achieved or that deviations are documented and justified.	Data Validator (Tetra Tech)
IIa/IIb	Project Action Limits	Review and add project action limits to the laboratory electronic data deliverable. Flag samples and notify PM of samples that exceed project action limits as listed oi Worksheet #15.	PM or designee (Tetra Tech)

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

(UFP-QAPP Manual Section 5.2.2.1) (Figure 37, page 110 UFP-QAPP Manual)

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Soil, Sediment, and Surface water	PAHs	Criteria for SW-846 8270C/8270C SIM listed in Worksheets #12, #15 and #28, DOD QSM (January 2006). If not included in the aforementioned the logic outlined in USEPA Region 3 Modification to National Functional Guidelines for Organic Data Review (September 1994) should be used to apply qualifiers to data.	Data Validation Specialist (Tetra Tech)
IIa and IIb	Soil, Sediment, and Surface water	Metals	Criteria for SW-846 6020 listed in Worksheets #12, #15 and #28, DOD QSM (January 2006). If not included in the aforementioned the logic outlined in USEPA Region 3 Modifications to the Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses (April 1993) should be used to apply qualifiers to data.	Data Validation Specialist (Tetra Tech)

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Aqueous and Solid	Explosives	Criteria for SW-846 8330B listed in Worksheets #12, #15, #24, #25, and #28, DOD QSM (January 2006). If not included in the aforementioned the logic outlined in U.S. EPA Region 3 Modifications to the Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses (September 2004) should be used to apply qualifiers to data.	Data Validation Specialist (Tetra Tech)
IIa and IIb	Solid	TOC, CEC, and pH	Method-specific criteria listed in Worksheets #12, #15, #24, #25, and #28.	Data Validation Specialist (Tetra Tech)

SAP Worksheet #37 -- Usability Assessment

(UFP-QAPP Manual Section 5.2.3)

Data Usability Assessment

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

Completeness

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

Precision

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

Accuracy

-The Project Chemist acting on behalf of the project team will determine whether the accuracy/bias goals were met for project data. This will be accomplished by comparing percent recoveries of LCS, LCSD, MS, MSD, and surrogate compounds to accuracy goals identified in Worksheet 28. This assessment will include an evaluation of field and laboratory contamination; instrument calibration variability; and analyte recoveries for surrogates, matrix spike, and

laboratory control samples. If the goals are not met, limitations on the use of the data will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report.

Representativeness

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

Comparability

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

Field XRF/Laboratory Lead Data Correlation

- The project statistician will evaluate the correlation of field XRF data to laboratory data. Factors considered in this evaluation will include the magnitude of the slope and intercept of the correlation equation, the distribution of data points across the plotted concentration range, and the value of the correlation coefficient. If the correlation coefficient is less than 0.65 or the plotted data do not appear to be well correlated according to standard statistical principles, limitations on the use of the data will be described in the project report.

Sensitivity

- The Project Chemist acting on behalf of the project team will determine whether project sensitivity goals listed in Worksheet #15 are achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not

achieved, the limitations on the data will be described. The Project Chemist will enlist the help of the project risk assessor to evaluate deviations from planned sensitivity goals.

Project Assumptions and Data Outliers

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

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

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

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

Identify the personnel responsible for performing the usability assessment:

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

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:

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

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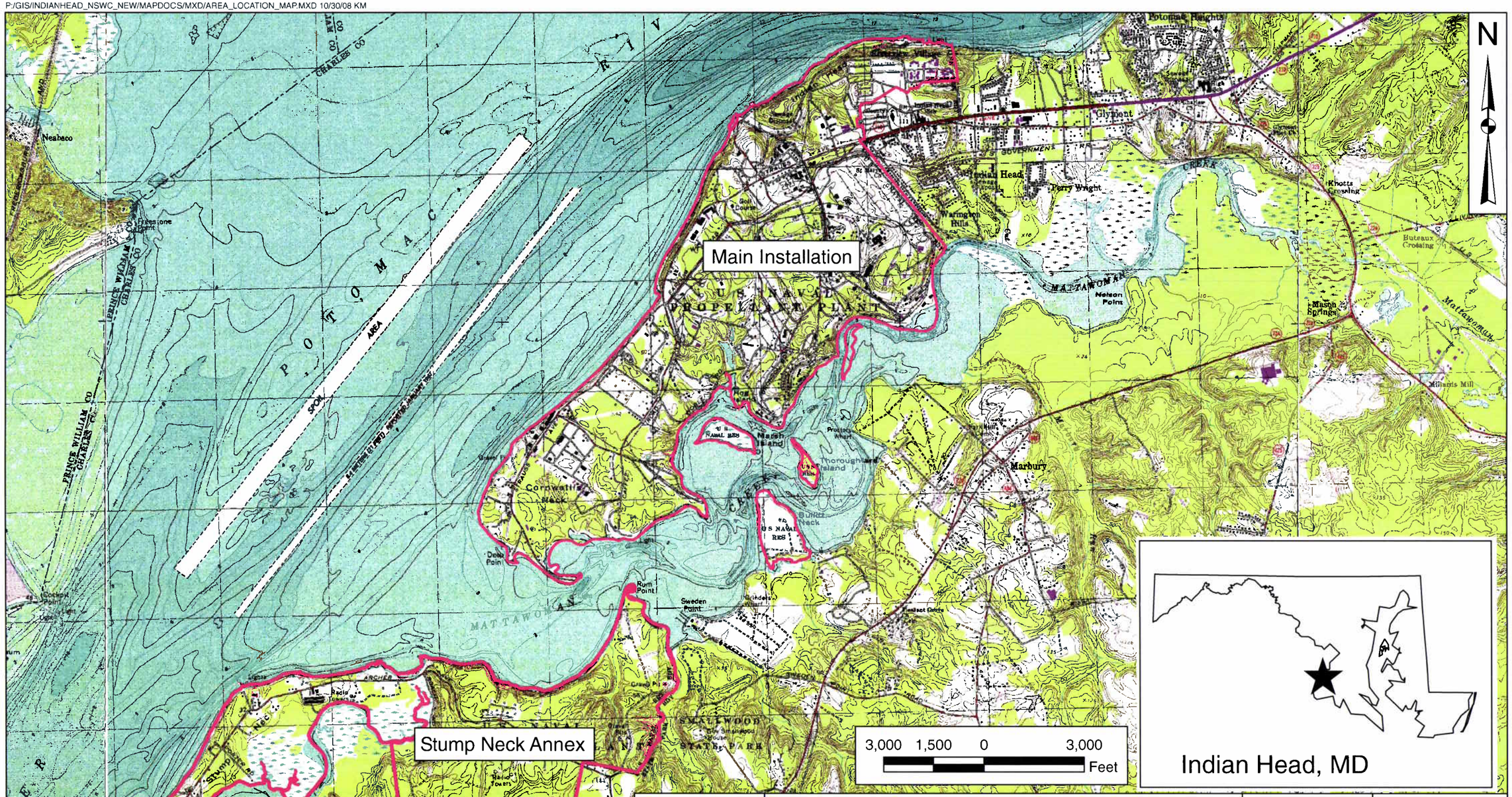

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COST/SCHEDULE-AREA		NAVAL SUPPORT FACILITY INDIAN HEAD	APPROVED BY	DATE
		INDIAN HEAD, MARYLAND	—	—
SCALE			FIGURE NO.	REV
AS NOTED			MAP 1-1	0

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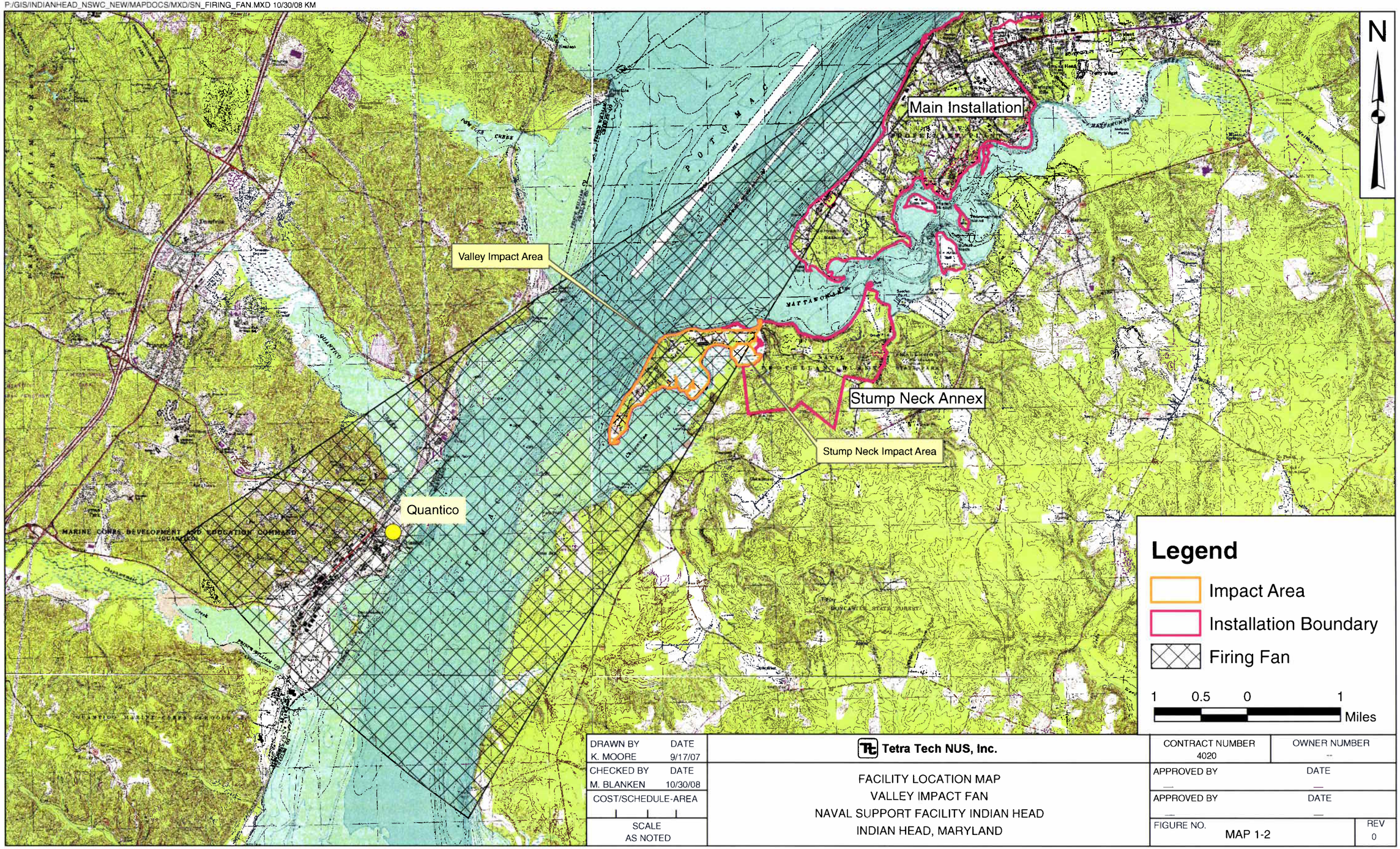

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COST/SCHEDULE-AREA			APPROVED BY —	DATE —
SCALE AS NOTED			FIGURE NO. MAP 1-2	REV 0

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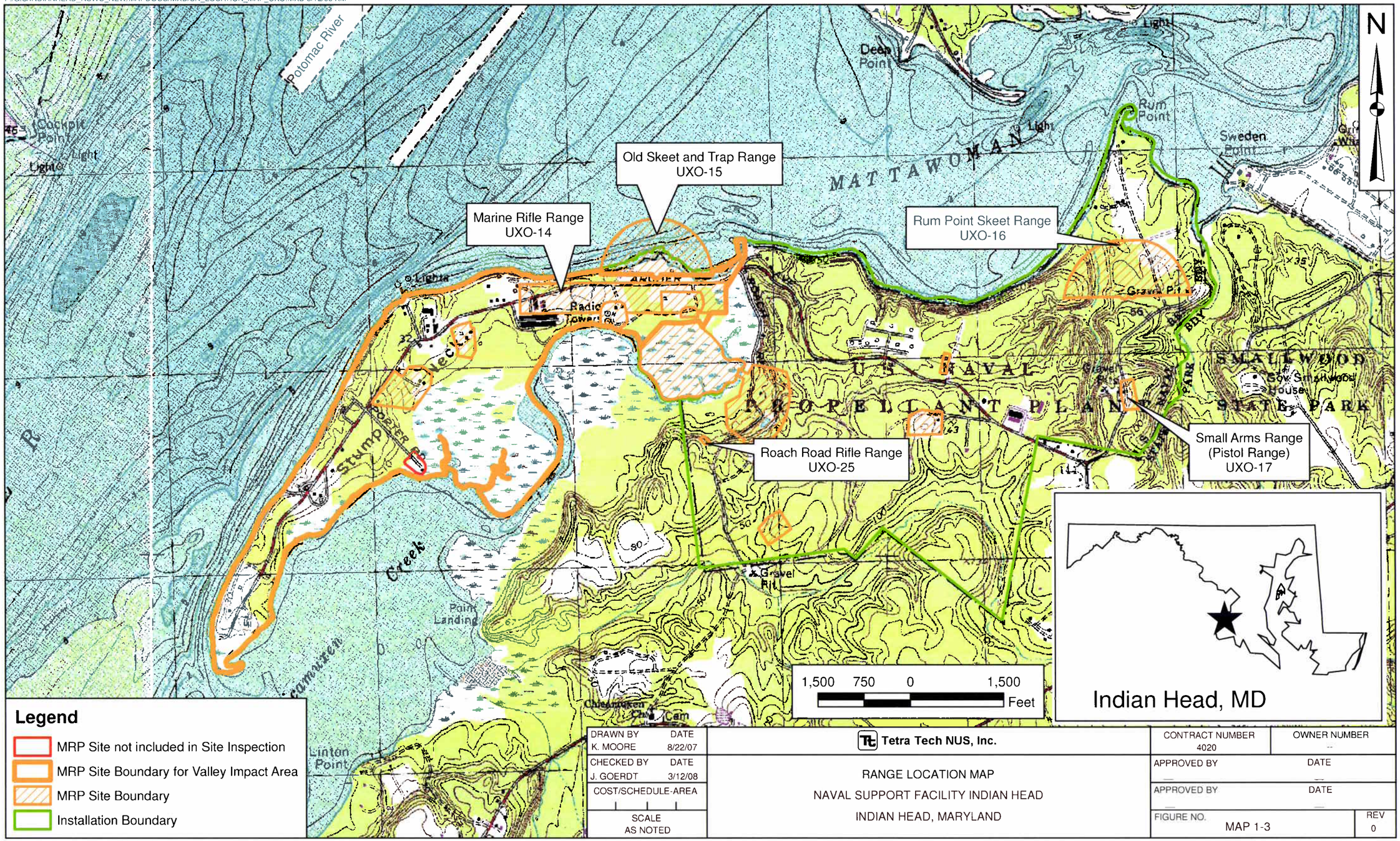

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J. GOERDT	3/12/08	NAVAL SUPPORT FACILITY INDIAN HEAD	—	—
COST/SCHEDULE-AREA		INDIAN HEAD, MARYLAND	APPROVED BY	DATE
			—	—
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AS NOTED			MAP 1-3	0

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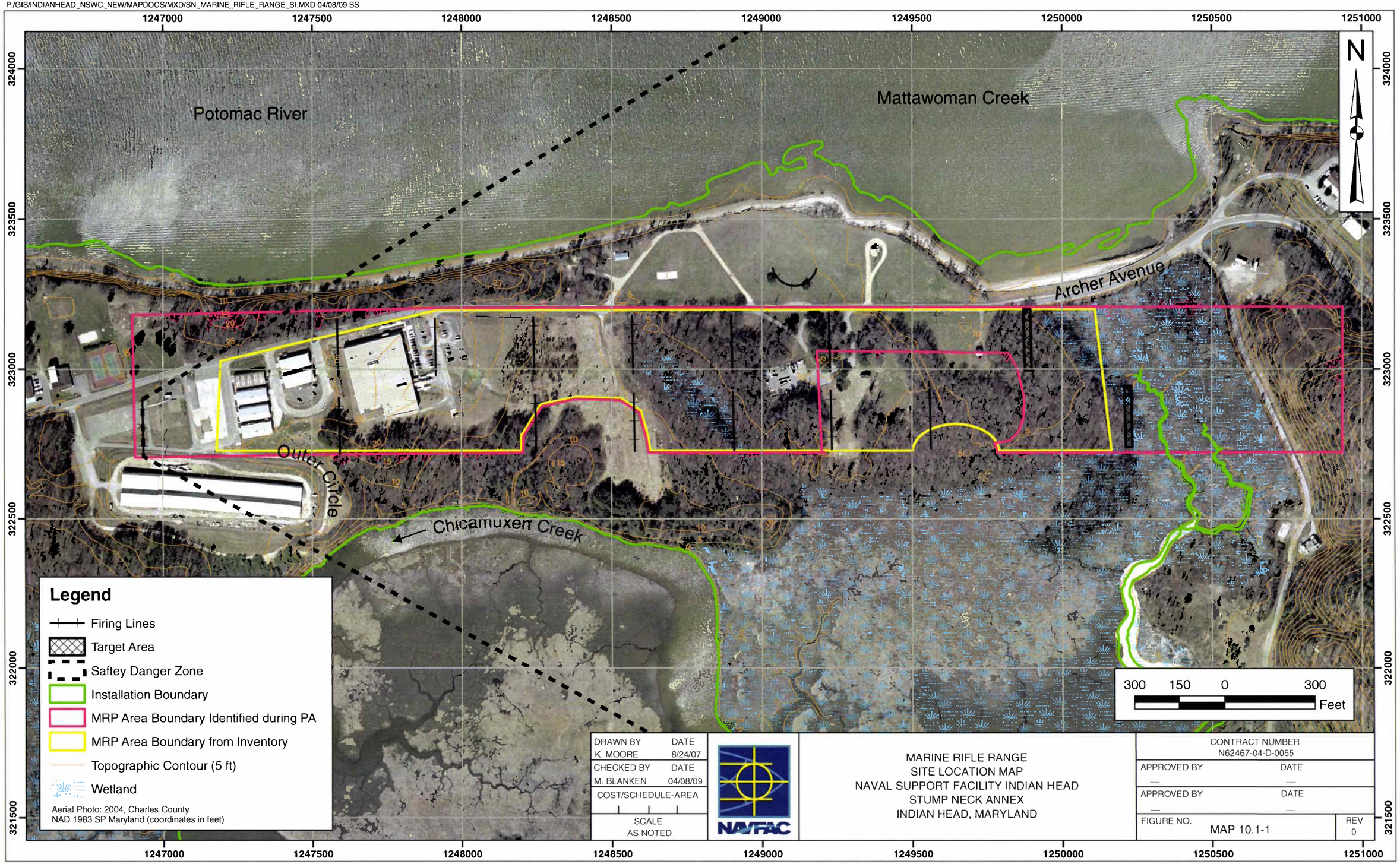

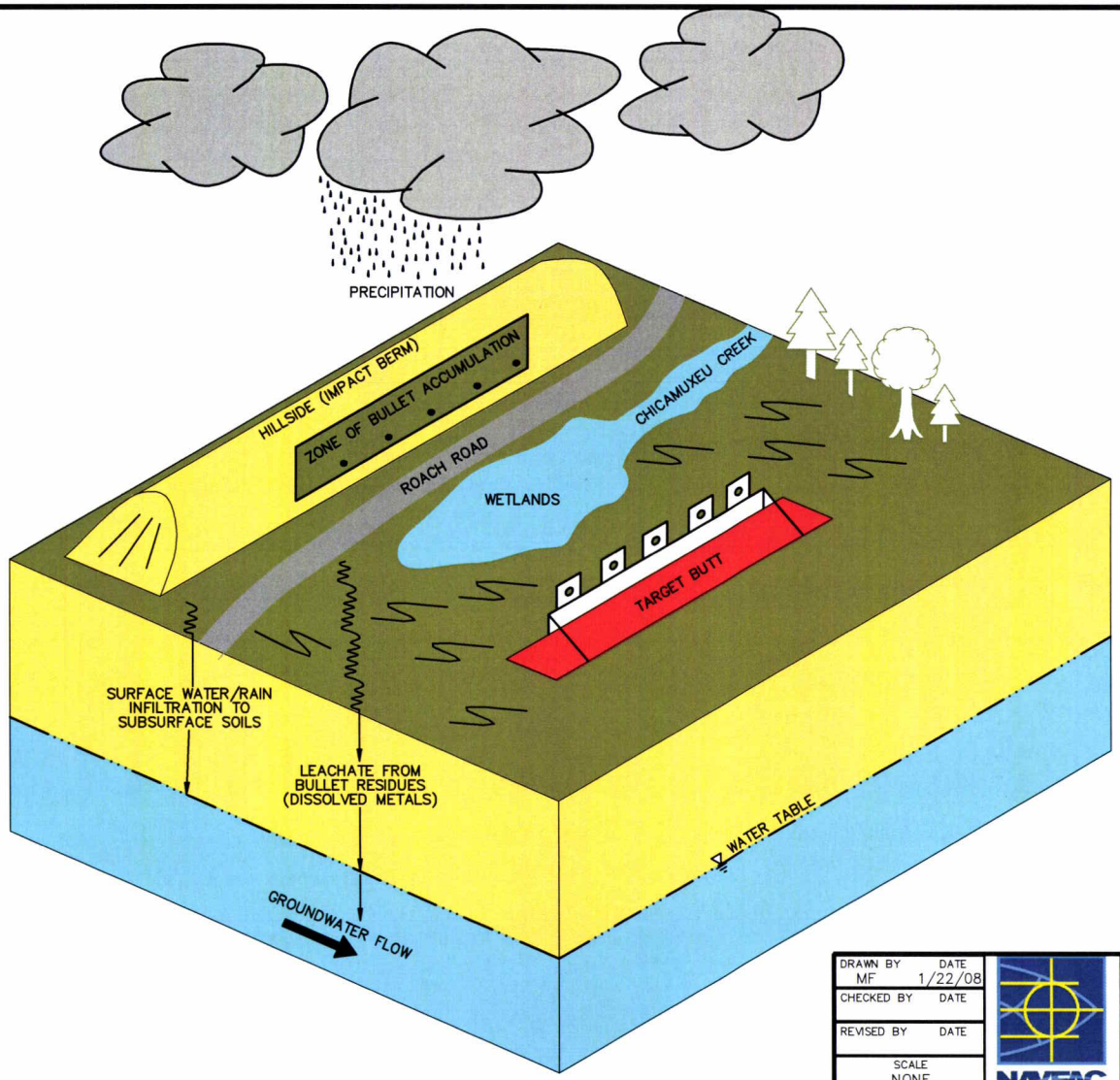

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REVISED BY	DATE		APPROVED BY / DATE
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NAVFAC

ACAD: 0810GD26.dwg 03/18/08 MF PIT

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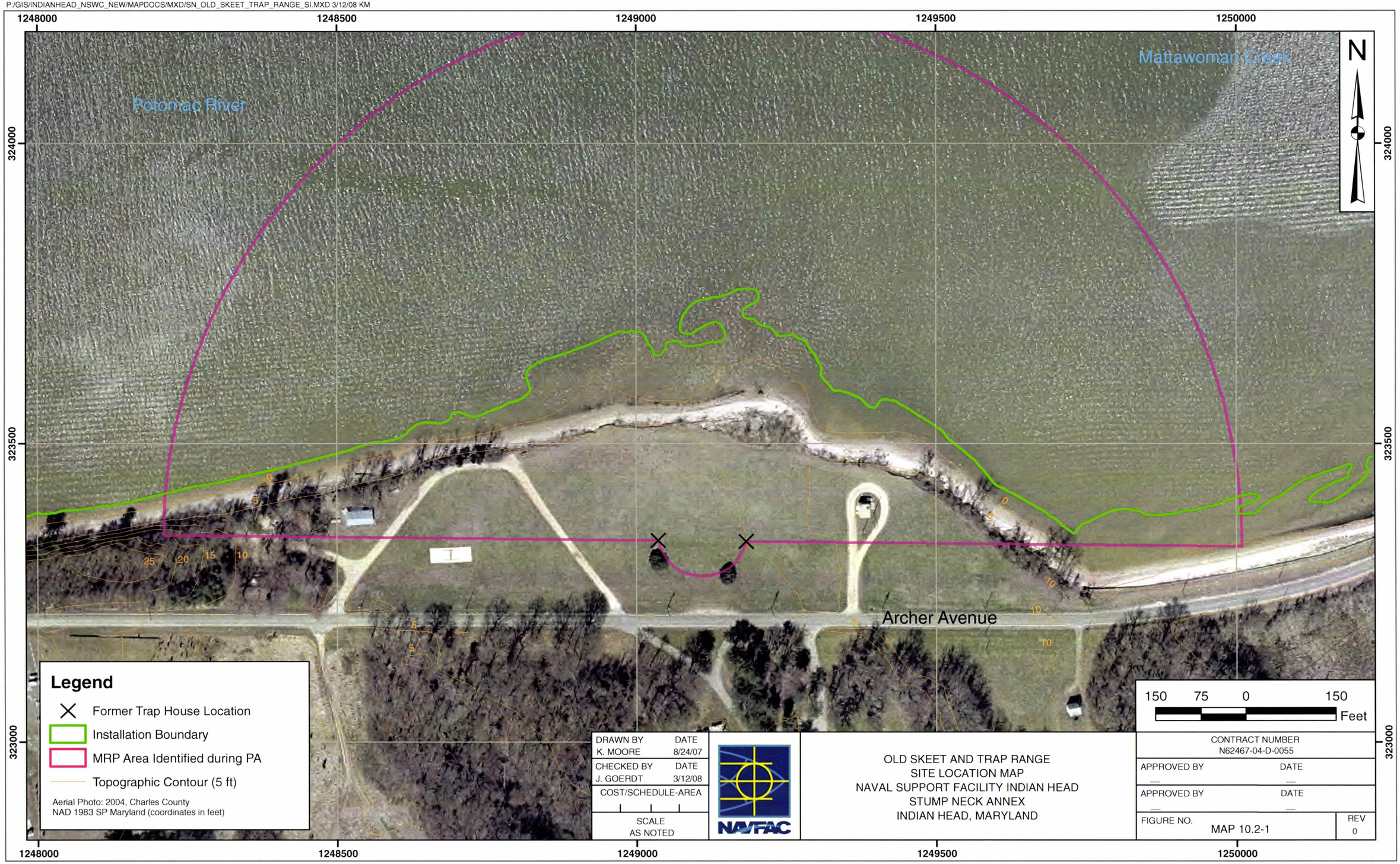

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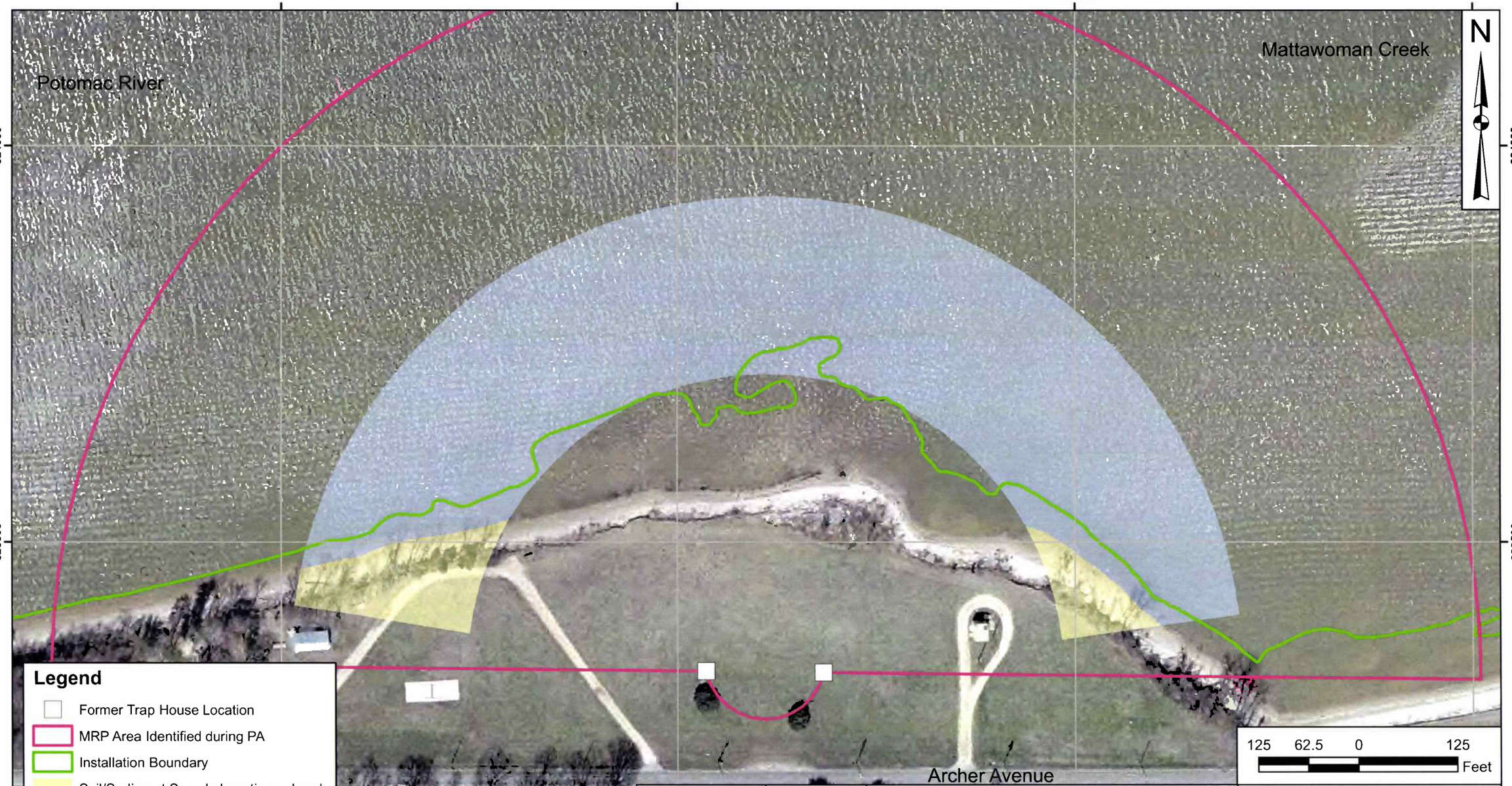

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CHECKED BY	DATE
M. BLANKEN	4/07/09
COST/SCHEDULE-AREA	
SCALE AS NOTED	

NAVFAC

OLD SKEET AND TRAP RANGE
MAXIMUM SHOT FALL
NAVAL SUPPORT FACILITY INDIAN HEAD
STUMP NECK ANNEX
INDIAN HEAD, MARYLAND

CONTRACT NUMBER N62467-04-D-0055	
APPROVED BY —	DATE —
APPROVED BY —	DATE —
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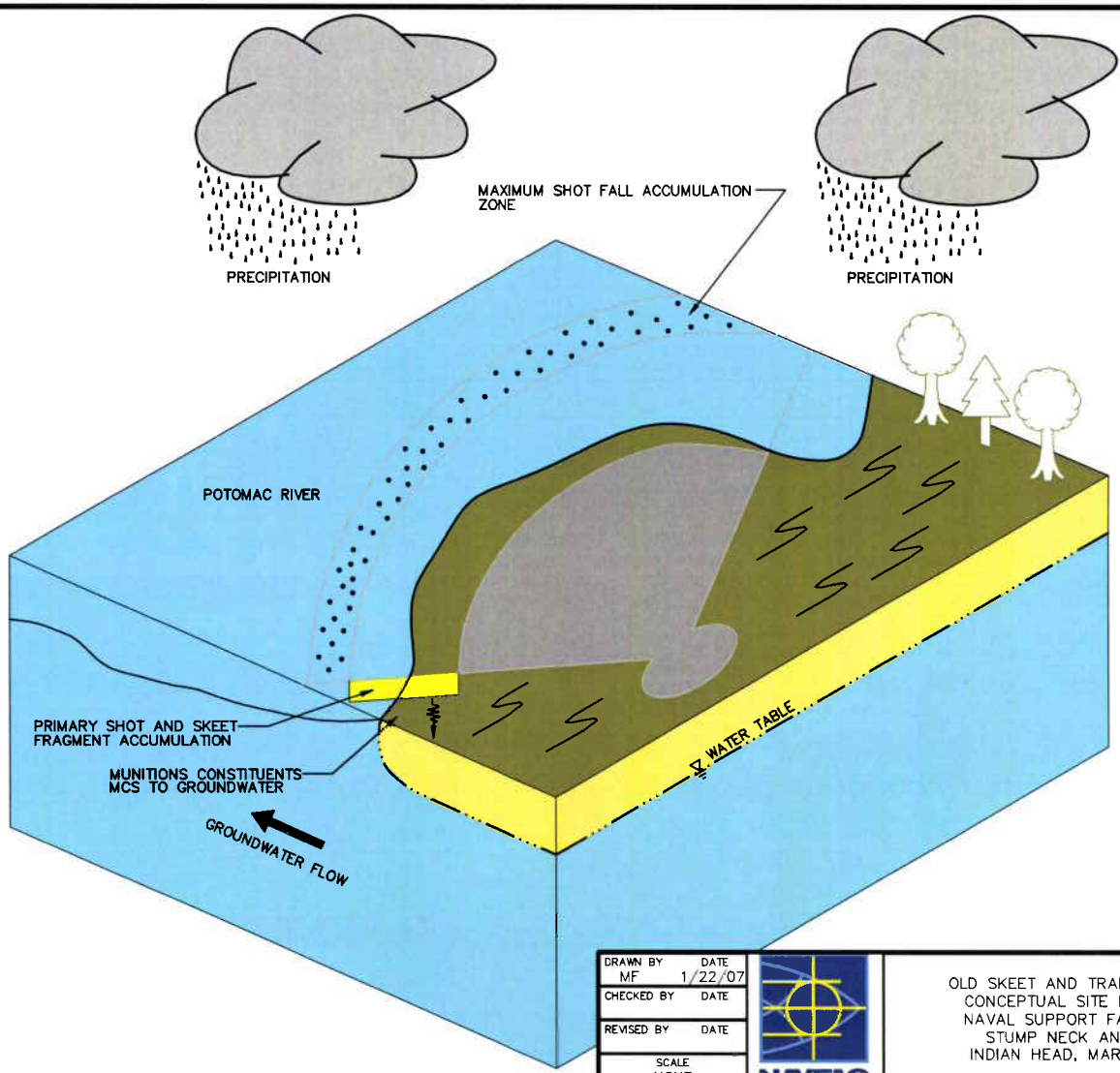

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REVISED BY	DATE		APPROVED BY / DATE
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NAVFAC

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FORM CADD NO SDIV-BHLDWG – REV 1 – 9/10/98

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K. MOORE	8/24/07
CHECKED BY	DATE
R. BARRINGER	10/26/07
COST/SCHEDULE-AREA	
SCALE AS NOTED	

NAVFAC

ROACH ROAD RIFLE RANGE
MRP AREA/SITE DETAILS MAP
NAVAL SUPPORT FACILITY INDIAN HEAD
STUMP NECK ANNEX
INDIAN HEAD, MARYLAND

CONTRACT NUMBER N62467-04-D-0055	
APPROVED BY —	DATE —
APPROVED BY —	DATE —
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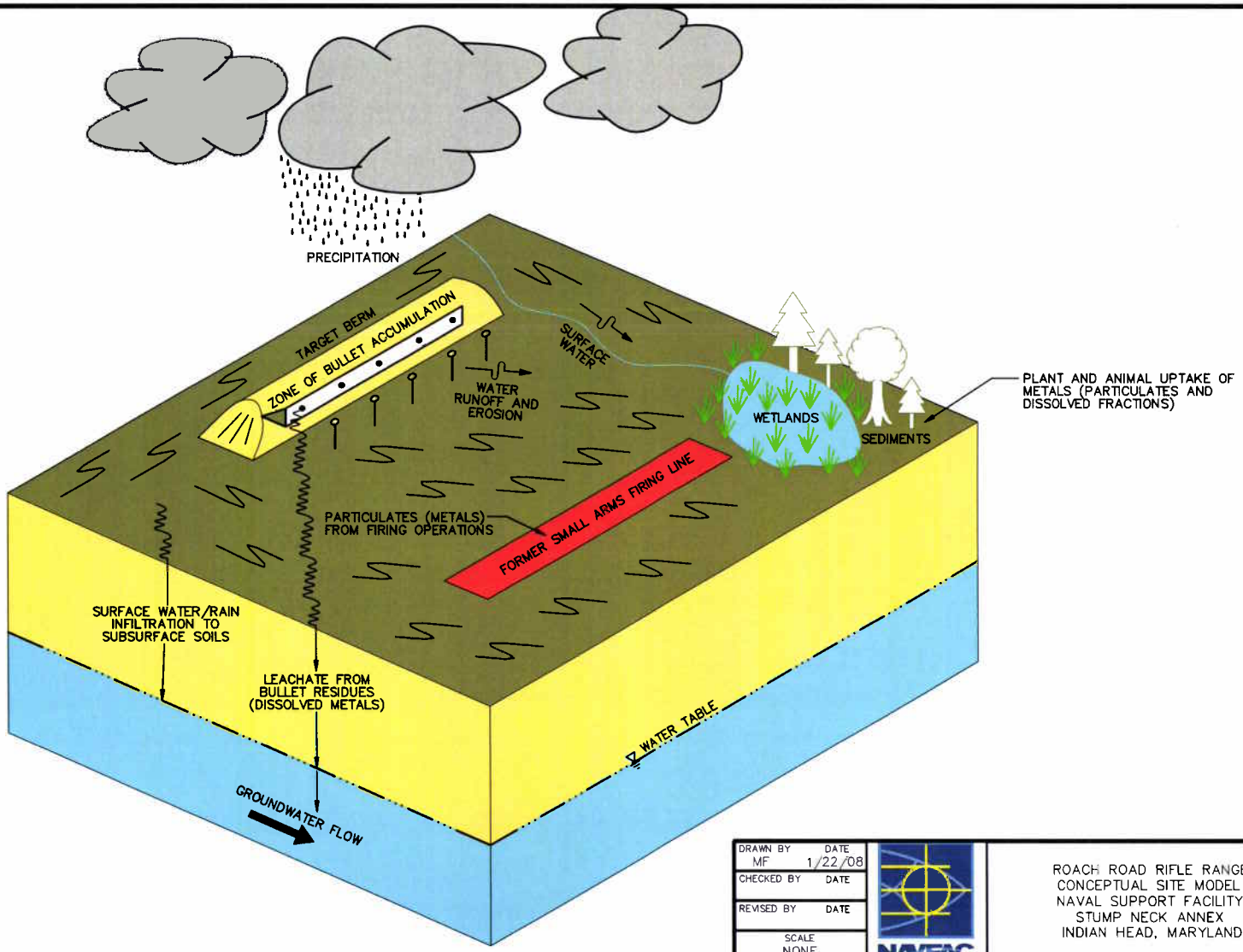

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REVISED BY	DATE		APPROVED BY / DATE
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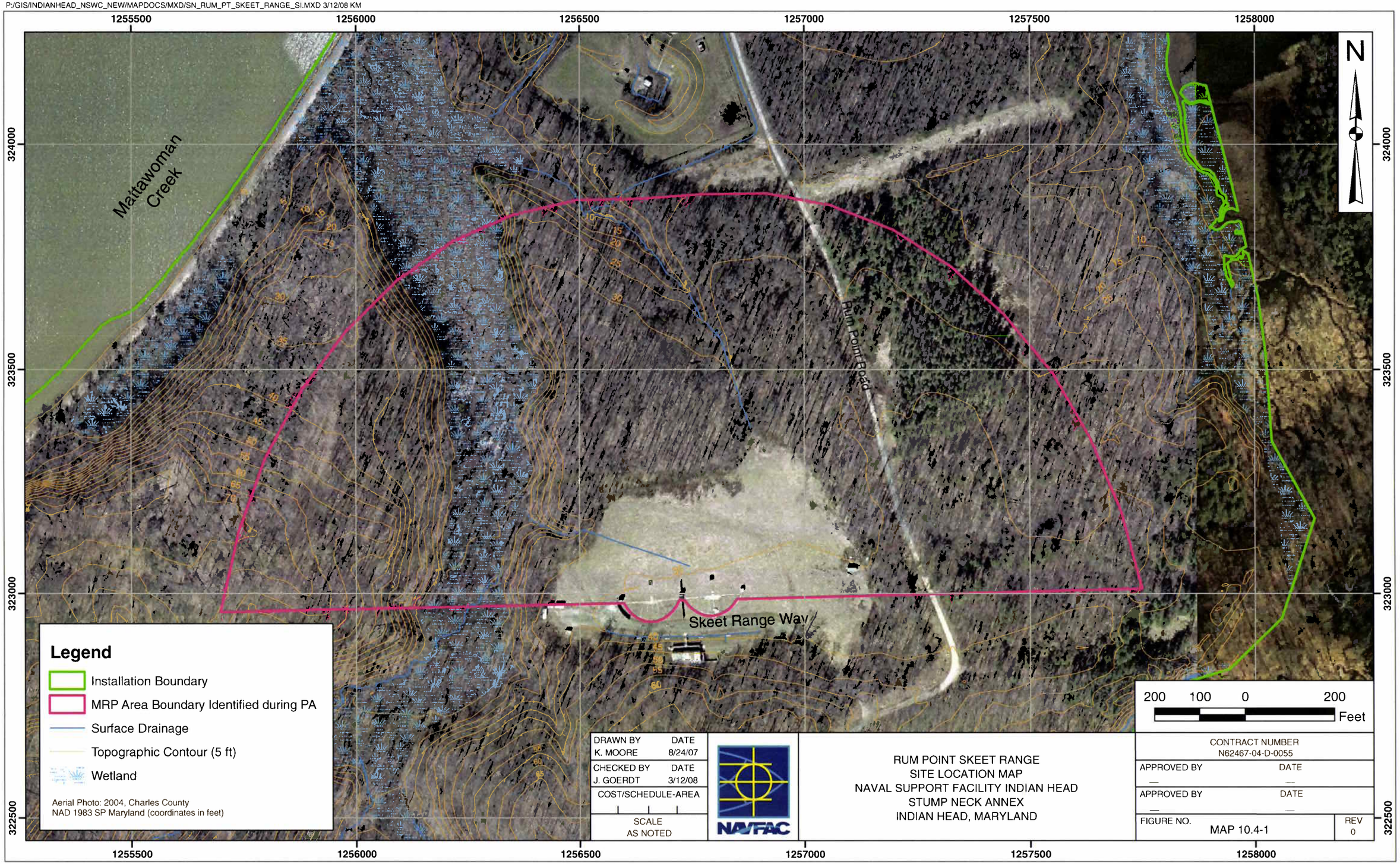

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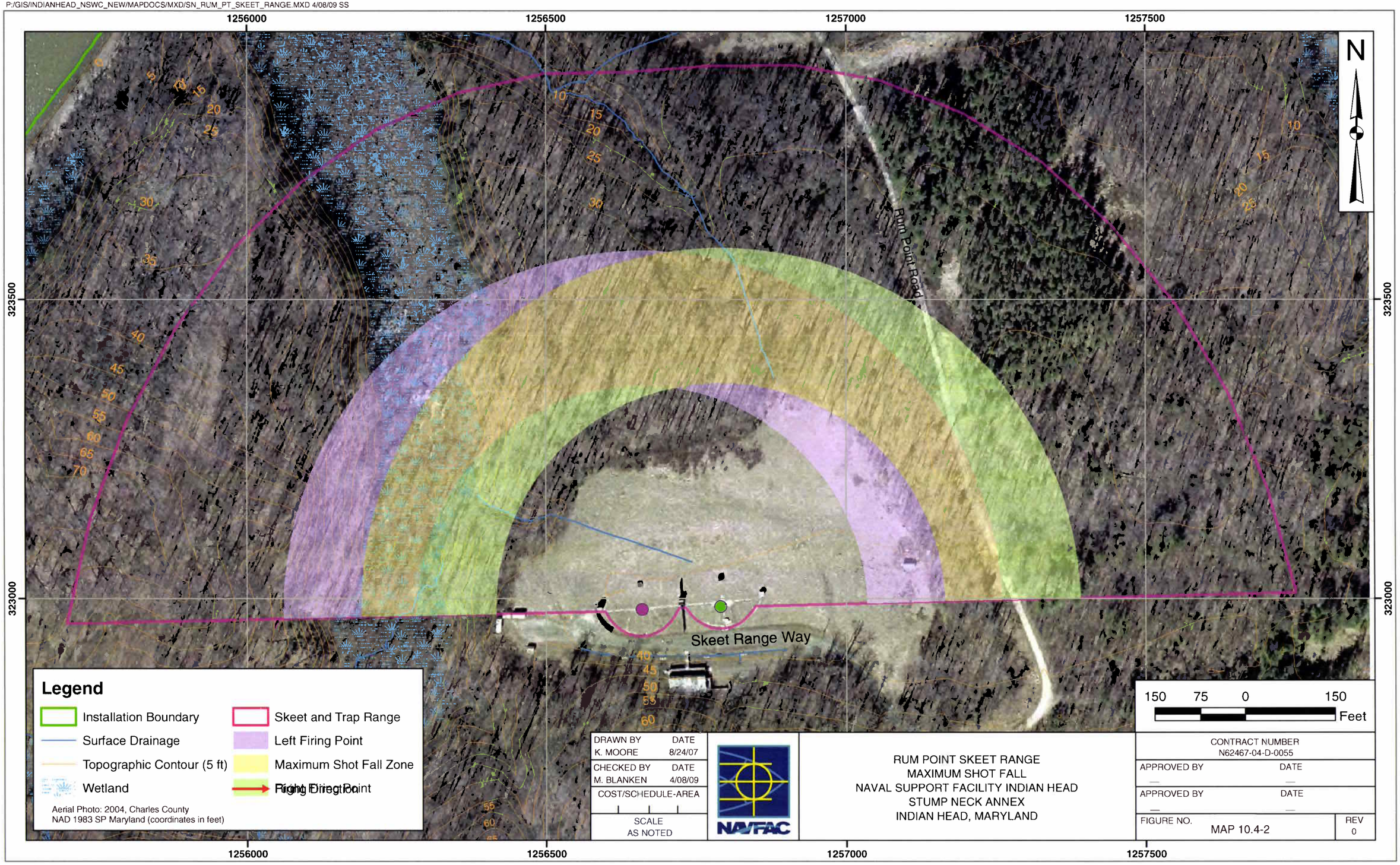

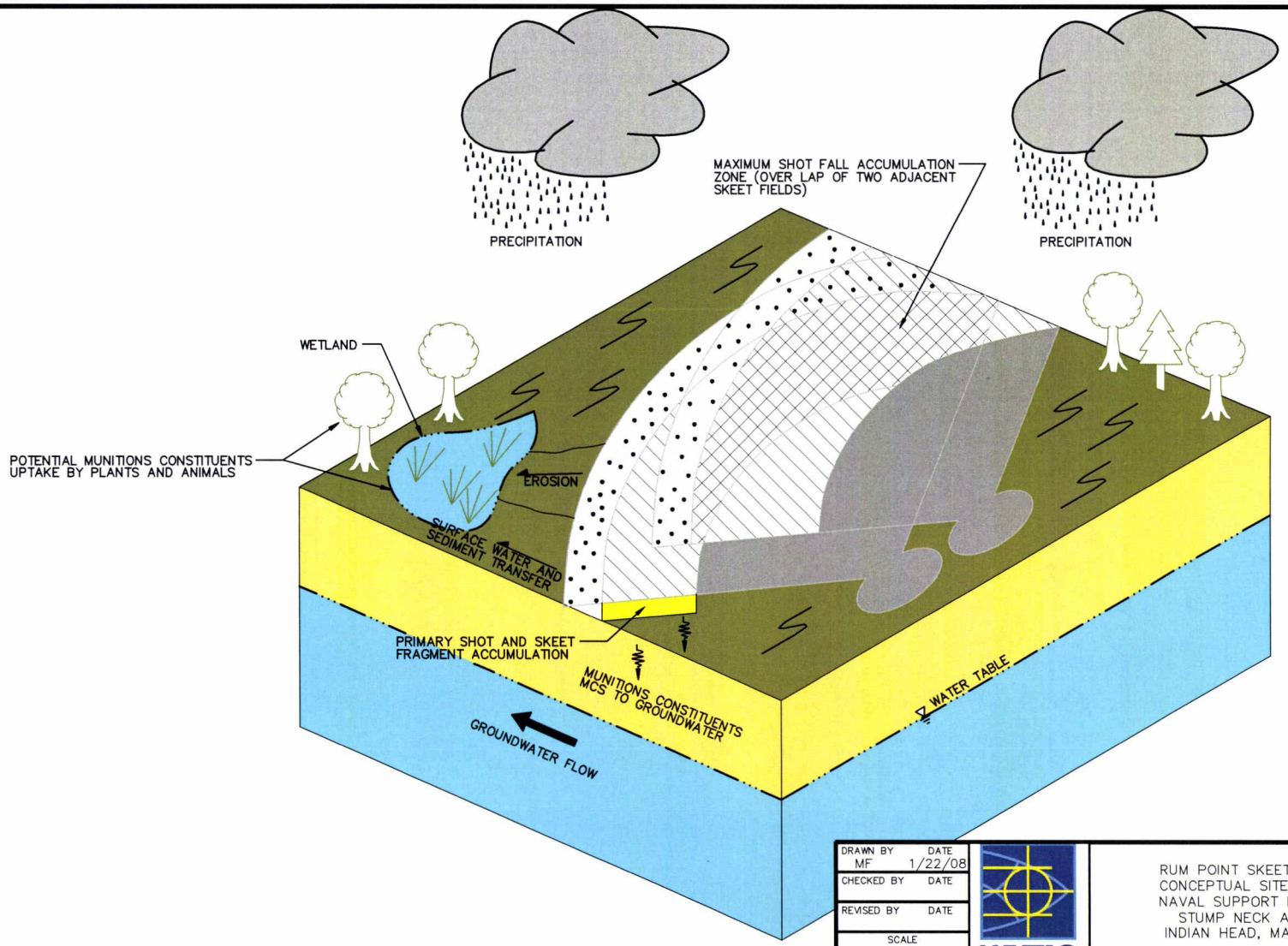

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K. MOORE	8/24/07
CHECKED BY	DATE
J. GOERDT	3/12/08
COST/SCHEDULE-AREA	
SCALE AS NOTED	

NAVFAC

SMALL ARMS (PISTOL) RANGE
SITE LOCATION MAP
NAVAL SUPPORT FACILITY INDIAN HEAD
STUMP NECK ANNEX
INDIAN HEAD, MARYLAND

CONTRACT NUMBER N62467-04-D-0055	
APPROVED BY —	DATE —
APPROVED BY —	DATE —
FIGURE NO. MAP 10.5-1	REV 0

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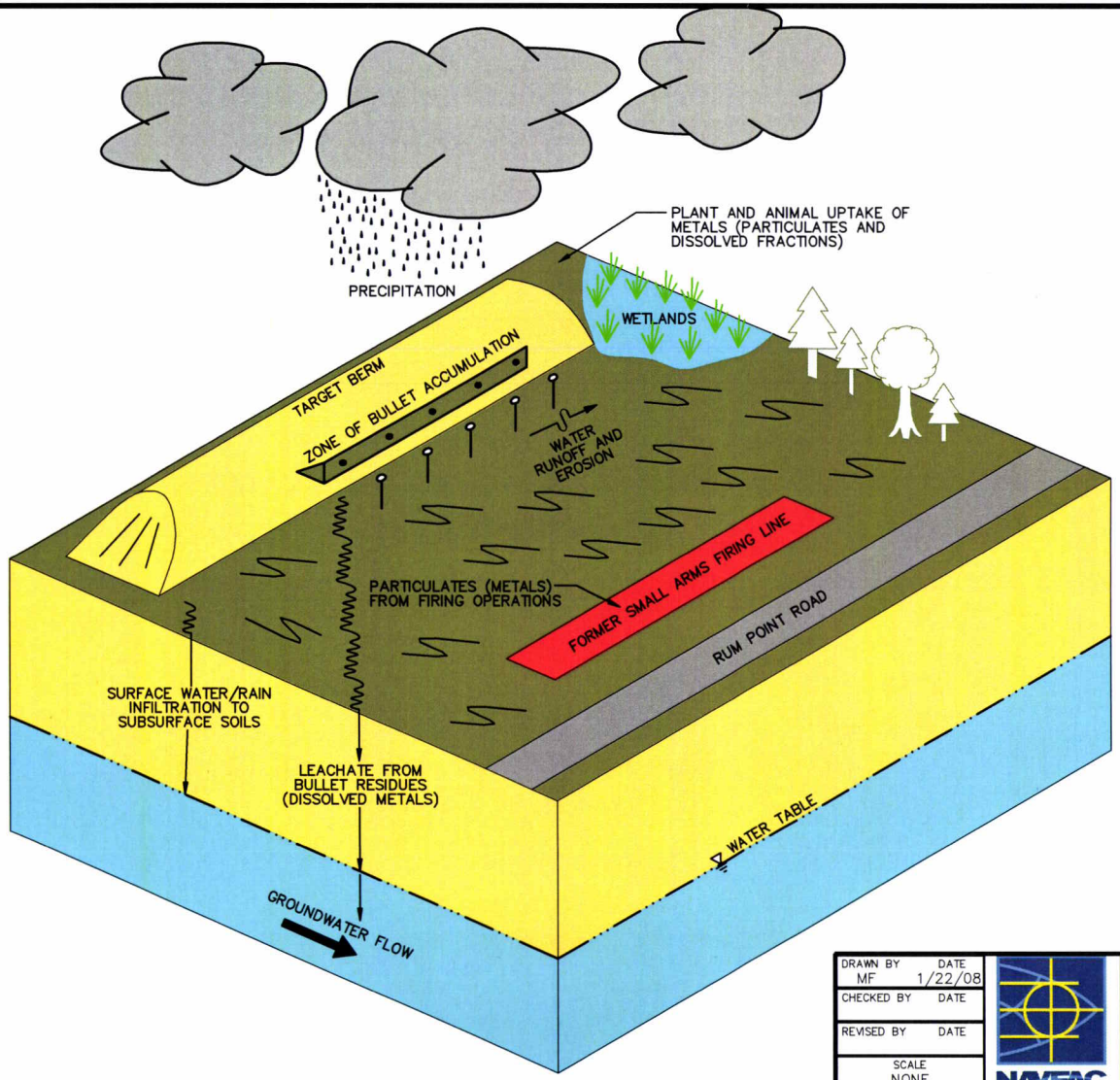

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REVISED BY	DATE		APPROVED BY / DATE
SCALE NONE			DRAWING NO. MAP 10.5-2 / REV. 0

NAVFAC

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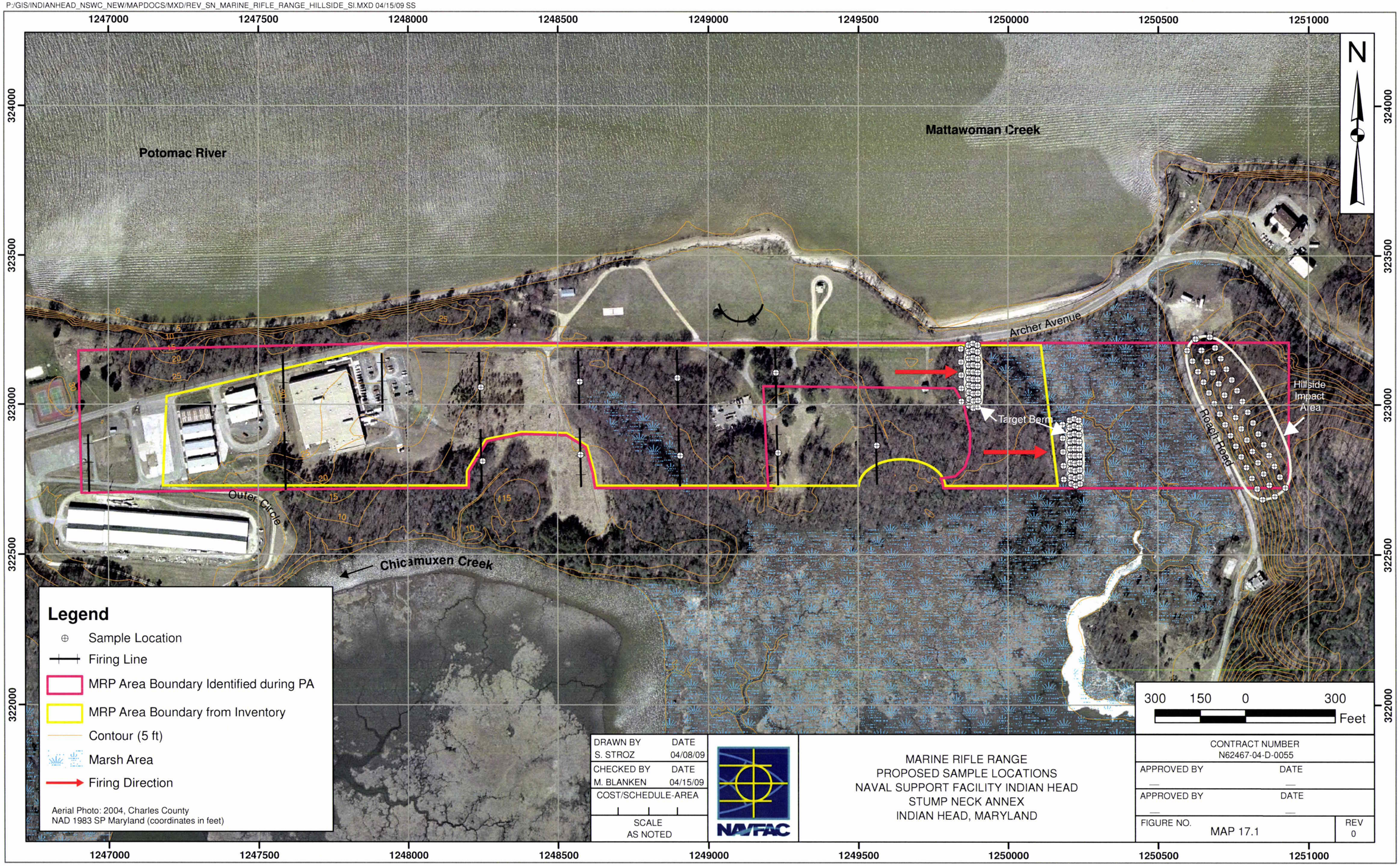

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S. STROZ	04/08/09
CHECKED BY	DATE
M. BLANKEN	04/15/09
COST/SCHEDULE-AREA	
SCALE AS NOTED	

NAVFAC

MARINE RIFLE RANGE
PROPOSED SAMPLE LOCATIONS
NAVAL SUPPORT FACILITY INDIAN HEAD
STUMP NECK ANNEX
INDIAN HEAD, MARYLAND

CONTRACT NUMBER N62467-04-D-0055	
APPROVED BY —	DATE —
APPROVED BY —	DATE —
FIGURE NO. MAP 17.1	REV 0

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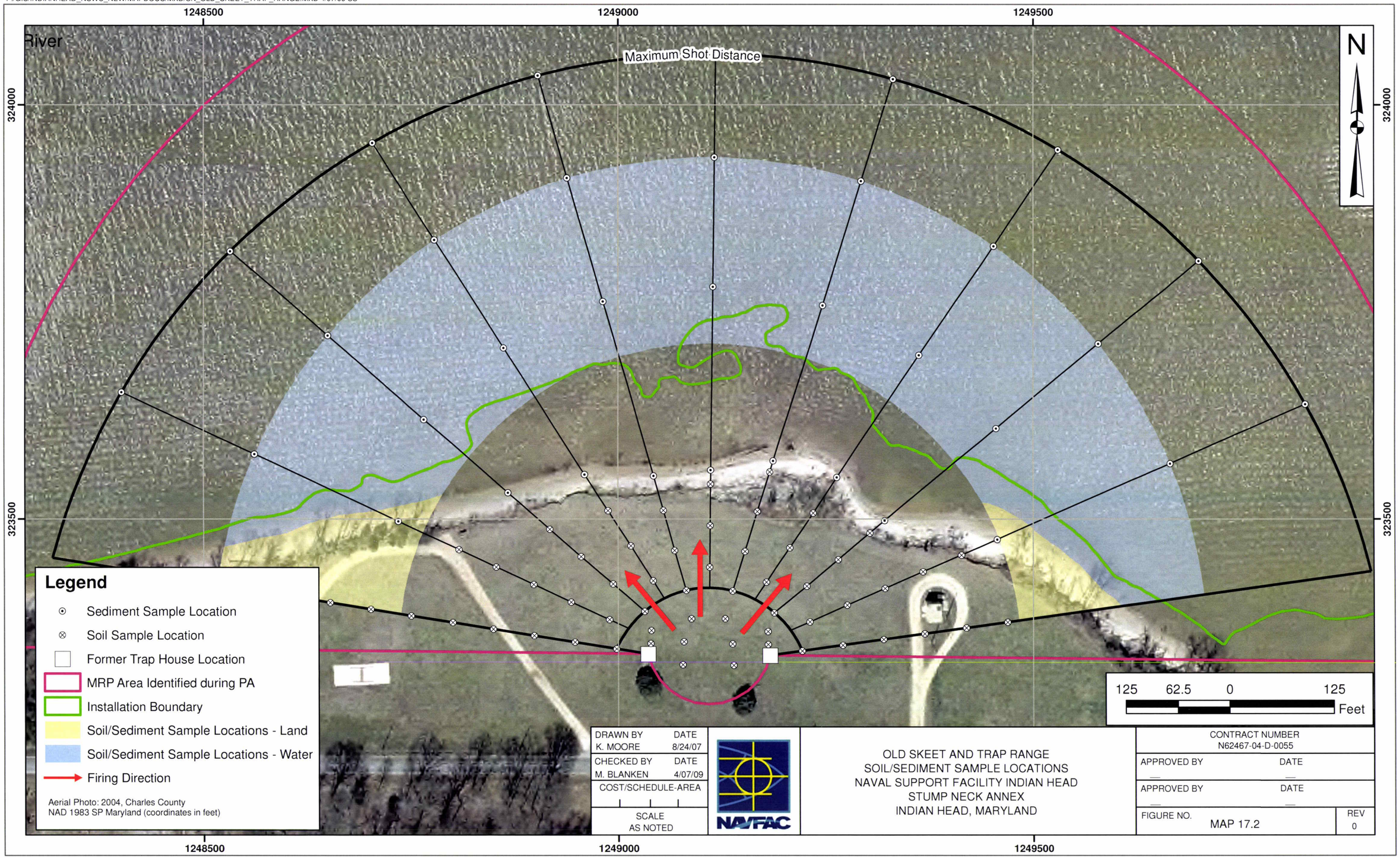

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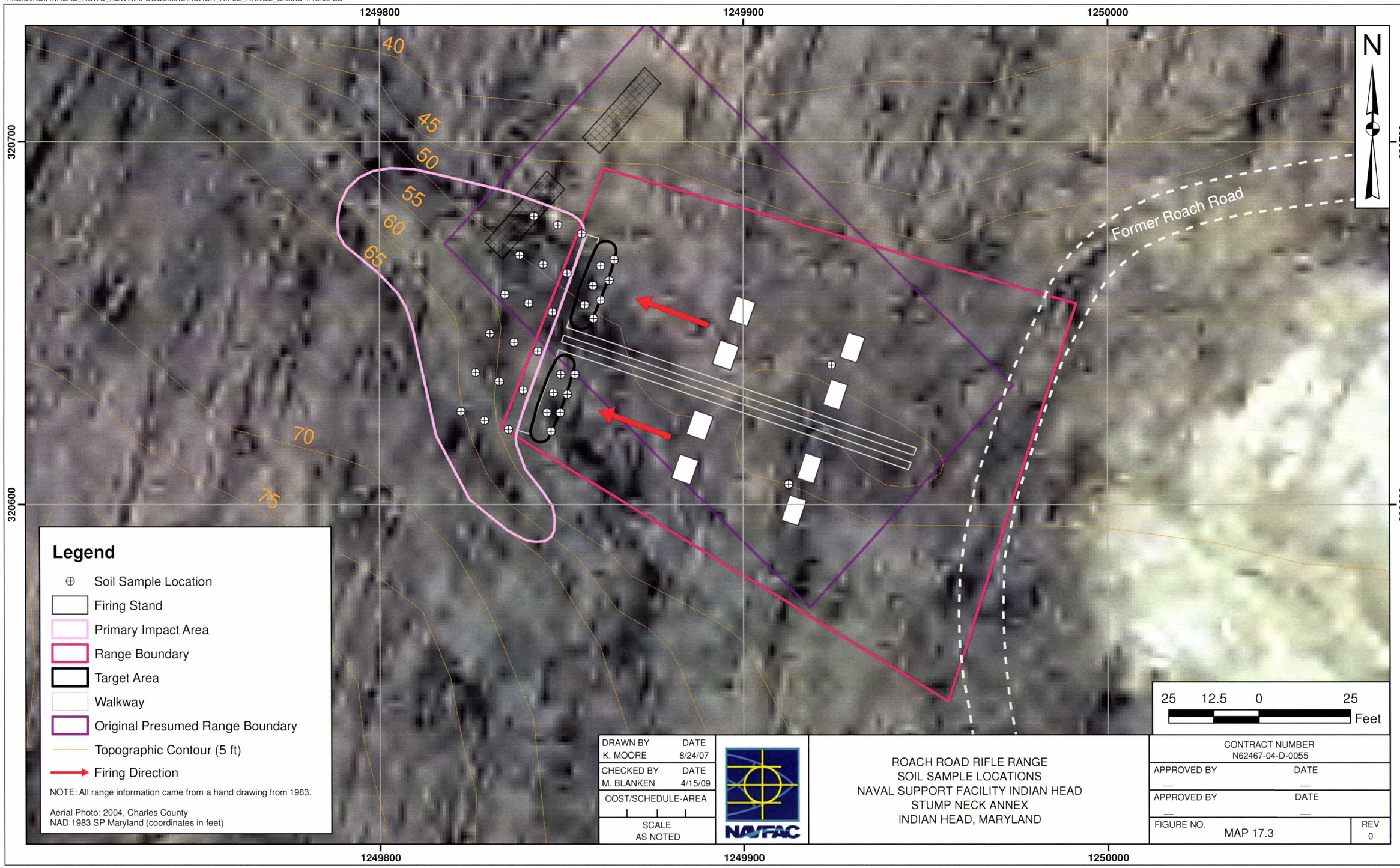

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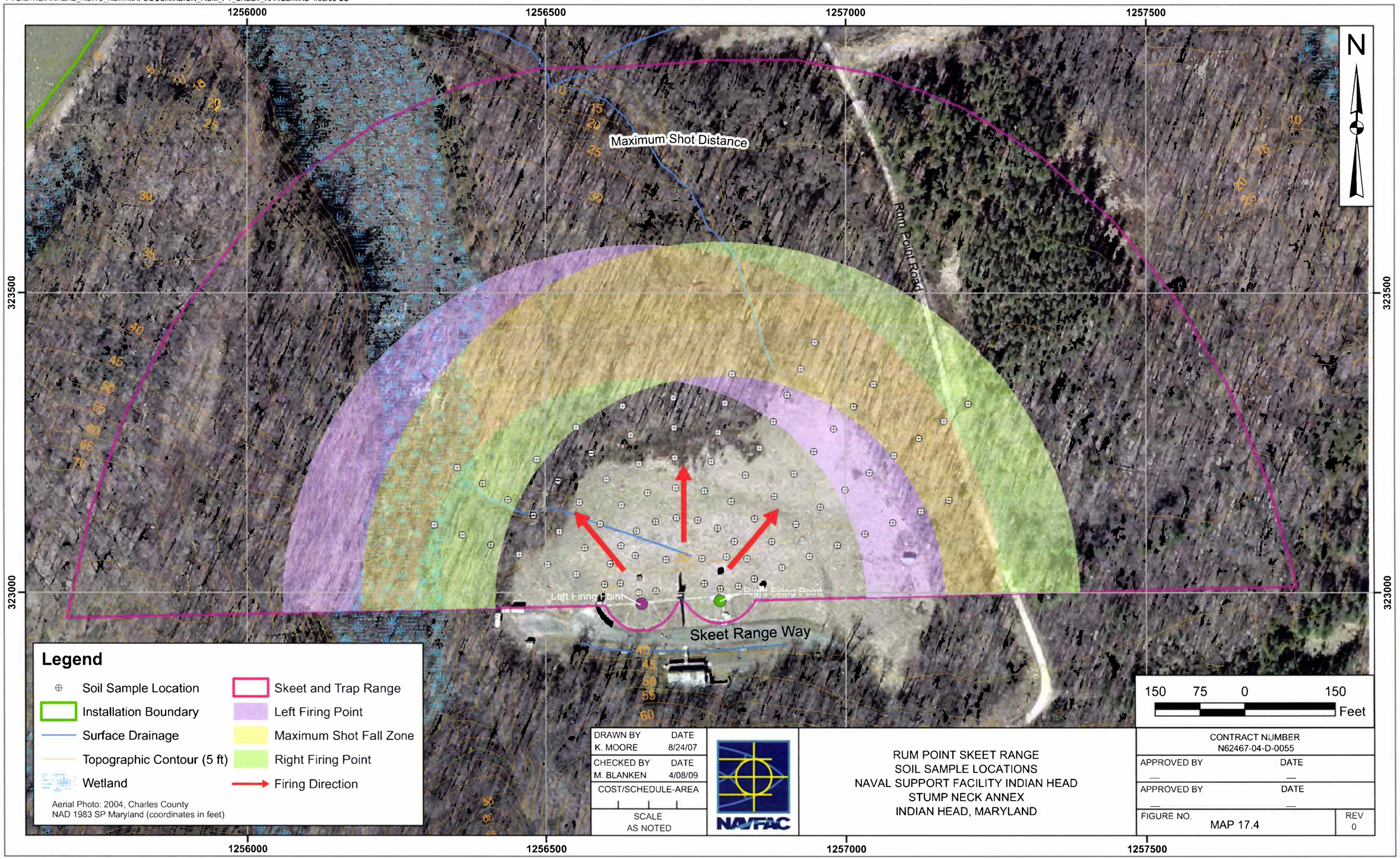

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APPENDIX A

FIELD STANDARD OPERATING PROCEDURES

APPENDIX A

SOP TABLE OF CONTENTS

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SOP-02 Sample Identification Nomenclature
SOP-03 Sample Custody and Documentation of Field Activities
SOP-04 Decontamination of Field Sampling Equipment
SOP-05 Soil Coring and Sampling Using Hand Auger Techniques
SOP-06 Borehole Advancement and Soil Coring and Sampling Using Direct-Push Technology
SOP-07 Surface Water Sampling
SOP-08 Sediment Sampling
SOP-09 Management of Investigation-Derived Waste
SOP-10 Borehole and Soil Sample Logging
SOP-11 Sample Preservation, Packaging, and Shipping
SOP-12 Calibration and Care of Water Quality Meter
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STANDARD OPERATING PROCEDURE
SOP-01

SAMPLE LABELING

1.0 PURPOSE

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

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Writing utensil (preferably black pen with indelible ink)
Disposable medical-grade gloves (e.g. latex, nitrile)
Sample log sheets
Required sample containers: All sample containers for analysis by fix-based laboratories will be supplied and deemed certified clean by the laboratory.
Sample labels
Chain-of-custody records
Sealable polyethylene bags
Heavy-duty cooler
Ice

3.0 PROCEDURES

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

- Project number (CTO 0423)
- Sample location ID
- Contract Task Order number
- Sample ID

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

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

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

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

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

4.0 ATTACHMENTS

1. Sample Label

ATTACHMENT 1

SAMPLE LABEL

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

STANDARD OPERATING PROCEDURE
SOP-02

SAMPLE IDENTIFICATION NOMENCLATURE

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to establish a consistent sample nomenclature system that will facilitate subsequent data management at the Naval Support Facility Indian Head Stump Neck Annex. The sample nomenclature system has been devised such that the following objectives can be attained.

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

The Maryland Department of Environmental Protection must approve any deviations from this procedure.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Writing utensil (preferably black pen with indelible ink)
Sample container labels

3.0 SAMPLE IDENTIFICATION NOMENCLATURE

3.1 Samples

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

3.1.1 Sample Numbering Scheme

The sample tracking number will consist of a four- or five-segment alpha-numeric code that identifies the sample's associated Unexploded Ordnance (UXO) site, sample type, location, and for aqueous samples, where applicable, whether a sample is filtered, and/or the sample round number. For soil samples, the final four tracking numbers will identify the depth in units of feet below ground surface (bgs) at which the sample was collected. For sediment samples, the final four tracking numbers will identify the depth in units of inches bgs at which the sample was collected.

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

ANN	**AA**	**NNN**	**NNNN (Soils and Sediment only)**
UXO Number	Matrix	Sample Location Number	Sequential depth interval from freshly exposed surface

Character Type:

A = Alpha

N = Numeric

UXO Number (ANN):

X01 = UXO 01

X14 = UXO 14

Matrix Code (AA):

SS = Surface Soil Sample

SB = Subsurface Soil Sample

SD = Sediment Sample

SW = Surface Water Sample

Location Number (NNN):

Sequential number beginning with "001" for each matrix.

Depth Interval:

This code section will be used for soil and sediment samples only.

The depth code is used to note the depth 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 (soil) or inches (sediment) bgs of the sample. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet, boring log, logbook, etc.

3.1.2 Examples of Confirmation Sample Nomenclature

A surface soil sample collected from UXO 1, sampling location 003, to a depth of 1 foot bgs would be labeled as “X01SS0030001”. A sediment sample collected from UXO 14, sampling location 010, to a depth of 6 inches bgs would be labeled as “X14SD0100006”.

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

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

3.2.1 QC Sample Numbering

The QC code will consist of a three- to four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

AA	NNNNNN	NN
QC Type	Date	Sequence Number (per day)

Character Type:

A = Alpha

N = Numeric

QC Types:

FD = Field Duplicate

RB = Rinsate Blank

SB = Source Blank

TB = Trip Blank

The sampling time recorded on the Chain-of-Custody Form, labels, and tags for field duplicate samples will be 0000 so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory).

3.2.2 Examples of Field QA/QC Sample Nomenclature

The first duplicate of the day at UXO 1 for a surface soil sample collected on March 24, 2008 would be designated as FD03240801.

The third duplicate of the day taken at UXO 14 of a surface soil sample collected on April 12, 2008 would be designated as FD04120803.

The first trip blank associated with samples collected on March 18, 2008 would be designated as TB03180801.

STANDARD OPERATING PROCEDURE
SOP-03

SAMPLE CUSTODY AND DOCUMENTATION OF FIELD ACTIVITIES

1.0 PURPOSE

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

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

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

Writing utensil (preferably black pen with indelible ink)
Site logbook
Field logbook
Sample label
Chain-of-Custody Form
Custody seals
Equipment calibration log
Soil and Sediment Sample Log Sheet
Surface Water Sample Log Sheet

3.0 PROCEDURES

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

3.1 Site Logbook

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

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

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

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

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

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

3.2 Field Logbooks

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

3.3 Sample Labels

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

3.4 Chain-of-Custody Form

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

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

3.5 Custody Seal

The custody seal is an adhesive-backed label, and it is part of the chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transit to the laboratory. The custody seals are signed and dated by the samplers and affixed across the opening edges of each cooler (two seals per cooler) containing environmental samples. The laboratory sample custodian will examine the custody seal for evidence of tampering and will notify the Tetra Tech TOM if evidence of tampering is observed.

3.6 Equipment Calibration Log

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

3.7 Sample Log Sheets

The Soil and Sediment Sample Log Sheets are used to document the sampling of soils and sediments (see SOPs-05, 06, and -08). The surface water sample log sheets are used to document the sampling of surface waters (see SOP-07).

4.0 ATTACHMENTS

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

ATTACHMENT 1
CHAIN-OF-CUSTODY RECORD

CHAIN OF CUSTODY | NUMBER | PAGE ____ OF _____

PROJECT NO:	SITE NAME:	PROJECT MANAGER AND PHONE NUMBER	LABORATORY NAME AND CONTACT:
SAMPLERS (SIGNATURE)		FIELD OPERATIONS LEADER AND 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	MATRIX	GRAB (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)

3/99
FORM NO. TtNUS-001

ATTACHMENT 2
EQUIPMENT CALIBRATION LOG

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : _____

SITE NAME: _____

PROJECT No.: _____

INSTRUMENT NAME/MODEL: _____

MANUFACTURER: _____

SERIAL NUMBER: _____

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

ATTACHMENT 3

SOIL AND SEDIMENT SAMPLE LOG SHEET

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:

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

COMPOSITE SAMPLE DATA:

	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Date:				
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 4

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	ORP
Date:	Visual	Standard	mS/cm	Degrees C	NTU	mg/l	%	mV
Time:								
Depth:								
Method:								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

STANDARD OPERATING PROCEDURE
SOP-04

DECONTAMINATION OF FIELD SAMPLING EQUIPMENT

1.0 PURPOSE

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

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Writing utensil (preferably black pen with indelible ink)
Non-latex rubber or plastic gloves
Cotton gloves
Field logbook
Potable water
Deionized water
Isoproponal (optional)
LiquiNox detergent
Brushes, spray bottles, paper towels, etc.
Container to collect and transport decontamination fluids

3.0 DECONTAMINATION PROCEDURES

3.1 Don non-latex and/or cotton gloves and decontaminate sampling equipment (in accordance with the following steps) prior to field sampling and between samples.

3.2 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.

3.3 Wash the equipment with a solution of LiquiNox detergent. Prepare the LiquiNox wash solution in accordance with the instructions on the LiquiNox container. Collect the LiquiNox wash solution into a container. Use brushes or sprays as appropriate for the equipment. If oily residue has

accumulated on the sampling equipment, remove the residue with an isopropanol wash and repeat the Liquinox wash.

3.4 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.

3.5 Rinse the equipment with deionized water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the deionized water rinsate into a container.

3.6 Remove excess water by air drying, shaking, or by wiping with paper towels as necessary.

3.7 Document decontamination by recording it in the field logbook.

3.8 Containerized decontamination solutions will be managed in accordance with the procedures described in SOP-09 and this UFP SAP.

STANDARD OPERATING PROCEDURE

SOP-05

SOIL CORING AND SAMPLING USING HAND AUGER TECHNIQUES

1.0 PURPOSE

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

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Disposable medical-grade gloves (e.g., latex, nitrile)
Writing utensil (preferably black pen with indelible ink)
Indelible marker
Stainless Steel Auger Buckets
Stainless Steel Extension Rods
Cross Handle
Required decontamination materials
Bentonite pellets
Sealable polyethylene bags
Sample labels
Shipping containers (containing ice)
Disposable plastic trowels or stainless steel trowels
Stainless steel mixing bowls
Sample containers: Sample containers are certified clean by the laboratory supplying the containers.
Soil Sample Log Forms
Daily Activity Logs
Chain-of-Custody Form
Soil Boring Log

3.0 BOREHOLE ADVANCEMENT AND SOIL SAMPLING USING A HAND AUGER

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

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

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

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

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

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

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

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

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

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

3.8 Decontaminate all soil sampling equipment in accordance with SOP-04 before collecting the next sample.

3.9 Soil samples will be transported to the field office where a portion of the sample will undergo XRF analysis for lead (See SOP-14).

3.10 For soil samples selected for fixed-base laboratory analysis, a portion of the sample will be used to fill the required sample containers as supplied by the laboratory. The sample labels will be completed and affixed to the sample container. The samples will then be packaged and shipped to the fixed-base laboratory in accordance with SOP-11.

4.0 ATTACHMENTS

1. Soil and Sediment Sample Log Sheet
2. Soil Boring Log Sheet

ATTACHMENT 1

SOIL AND SEDIMENT SAMPLE LOG SHEET

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:

	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Date:			
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):

ATTACHMENT 2

BORING LOG

Tetra Tech NUS, Inc.

BORING LOG

Page ___ of ___

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: 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 reponse read.

Drilling Area Background (ppm): _____

Remarks: _____

Converted to Well: Yes _____ No _____ Well I.D. #: _____

STANDARD OPERATING PROCEDURE
SOP-06

BOREHOLE ADVANCEMENT AND SOIL CORING FOR SOIL SAMPLING USING DIRECT-PUSH TECHNOLOGY

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures for collecting surface and subsurface soil cores from unconsolidated overburden materials using direct-push technology (DPT). For these investigations, a Geoprobe® rig with a Macrocore Sampler will be the type of DPT used.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Cut-resistant non-latex impermeable gloves
Cotton gloves
Disposable medical-grade gloves (e.g., latex, nitrile)
Writing utensil (preferably black pen with indelible ink)
Indelible marker
Boring log sheets: A copy of this form is included in SOP-10
Geoprobe® or equivalent DPT equipment
Geoprobe® Macrocore Sampler or equivalent
Geoprobe® Sampling Kit or equivalent
Clear acetate liners: one new liner for each soil core
Required decontamination materials (see SOP-04)
Bentonite pellets

3.0 BOREHOLE ADVANCEMENT AND SOIL SAMPLING USING A GEOPROBE®

Direct-push technology (DPT) will be employed to collect soil cores. DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional rotary drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools. Geoprobe® is a manufacturer of a hydraulically powered, percussion/probing machine utilizing DPT to

collect subsurface environmental samples. This type of rig with a Macrocore Sampler will be used at the Naval Support Indian Head Stump Neck Annex to collect soil cores.

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

3.2 Place a new clear acetate liner in the detachable Macrocore core barrel and attach coring device to the Geoprobe® rig.

3.3 Drive macrocore sampler (lined with acetate) into the ground to a specified depth using hydraulic pressure. The 0- to 1-foot depth soil interval is considered to be the surface soil. Subsurface soil samples will be collected at depths greater than 1 foot below ground surface.

3.4 Retract the sampler from the borehole and remove the acetate liner and the soil core from the Macrocore barrel.

3.5 Attach the metal trough from the Geoprobe® Sampling Kit firmly to a suitable surface.

3.6 Place the acetate liner containing the soil core in the trough.

3.7 While wearing cut-resistant gloves, 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. **CAUTION**: Do not attempt to cut the acetate liner while holding it in your hand.

3.8 Log the soil core on the Boring Log Sheet (see SOP-10).

3.9 Place the desired interval of the soil core in a sealable polyethylene bag, thoroughly homogenize the sample material and write sample ID, date, and time on the bag with an indelible marker.

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

3.11 Repeat steps 3.2 through 3.11 for the next depth intervals, if required.

3.12 The depth water table if encountered should be recorded on the Boring Log, and the estimated moisture content of the soil and the presence or absence of water in the boring should be noted.

3.13 If readings from the PID are all at background levels below field screening criteria, then excess soil core materials will be returned to the hole and tamped. If insufficient soil is available to fill the hole to the ground surface, then bentonite pellets mixed with the soil will be used to backfill the hole, and hydrated as per manufacture recommendation.

3.14 If screening instruments indicate that contaminants may be present in the soil materials, then all excess soil core materials will be placed in a plastic bag (or drum if larger quantities). The bag will be tagged identifying the location and depths from where the soils came and the date. The bag will then be placed in a 55-gallon drum and stored on-site until laboratory analyses of the soil are completed and classification of the soil waste materials can be determined (see SOP-09).

3.15 If soil materials from the boring are suspected of being contaminated (see 3.15 above), the soil boring will be backfilled with bentonite pellets up to the ground surface.

3.16 Decontaminate all soil sampling equipment in accordance with SOP-04 prior to collecting the next sample.

3.17 Soil samples will be transported to the field office where a portion of the sample will undergo XRF analysis for lead (See SOP-14).

3.18 For soil samples selected for fixed-base laboratory analysis, a portion of the sample will be used to fill the required sample containers as supplied by the laboratory. The sample labels will be completed and affixed to the sample container. The samples will then be packaged and shipped to the fixed-base laboratory in accordance with SOP-11.

STANDARD OPERATING PROCEDURE
SOP-07

SURFACE WATER SAMPLING/ SMALL STREAM FLOW ESTIMATION

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for collecting surface water samples and provides technical guidance on the measurement of stream channel cross-section and flow in a small stream. Stream flow measurements will be obtained to evaluate the migration potential of contaminants in a stream channel. This method is applicable for free flowing streams with uniform flow and widths greater than 3 feet and depths greater than about 6 inches. Stream flow rates will be measured at the same time when water level measurements are collected.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT (SURFACE WATER SAMPLING)

Surface Water Sample Log Sheet: A copy of this form is attached at the end of this SOP.

Field logbook

Writing utensil (preferably black pen with indelible ink)

Indelible marker

Multi-parameter water-quality meter: The water-quality meter is used for the measurement of dissolved oxygen, pH, specific conductance, temperature, oxidation-reduction potential, and turbidity (see SOP CTO331-14).

LaMotte Turbidity Meter: Used to measure turbidity in the field.

Disposable sample containers: Disposable sample containers are used to fill sample containers and transport sample(s) to a pump for filtering.

Sample containers: Certified-clean sample containers will be provided by the laboratory that performs the analyses.

0.45-micron filter assembly: These are single-use filter cartridges used to filter samples scheduled for dissolved metals or dissolved thorium isotope analyses. The filters become investigation-derived waste (IDW) after one use.

Peristaltic pump

Silicon tubing

Sealable polyethylene bags

Shipping containers (coolers)

2.1 REQUIRED FIELD FORMS AND EQUIPMENT (SMALL STREAM FLOW ESTIMATION)

Measuring rod or yardstick
Measuring tape
Waders or rubber boots
Colored flagging tape
2x2 inch wooden stakes
Bound Field Log Book
Waterproof pen
Orange or apple
Surface Water Sample Log Sheet
Indelible marker
Stopwatch

3.0 SURFACE WATER SAMPLING PROCEDURES

3.1 Sampling will start at the downstream end of a stream and proceed to the farthest upstream location.

3.2 While standing downstream or from the bank, gently remove any floating leaves or twigs that may be present in a sample pool area in a manner that will not disturb the bottom sediment.

3.3 While standing downstream or from the bank, place the sample container in the water at the sampling location at a 45-degree angle and lower it to approximately half the sample pool depth. With the mouth of the container facing upstream, fill the container with water, being careful not to disturb the sediment.

3.4 All samples will be collected into certified-clean, pre-preserved bottles (if preservation is required for the analysis to be performed) supplied by the laboratory performing the analyses. Sample containers should <u>not</u> be filled completely; a small amount of air should be left at the top.

3.5 Record the date and time that the sample containers are filled on the Surface Water Sample Log Sheet, the sample labels, and the Chain-of-Custody Form.

3.6 After the sample label is completed and checked, place the sample container into a sealable polyethylene bag and place the plastic storage bag holding the sample container into a cooler containing ice.

3.7 Repeat steps 3.3 through 3.6 until all the sample bottles containing unfiltered samples have been filled.

3.8 Fill two 1-liter unpreserved polyethylene bottles. Use these bottles to transfer the sample for field filtering. Set up a peristaltic pump for filtering of the dissolved metals samples. Using new, clean, disposable silicone tubing and a 0.45-micron filter, place the intake tubing from the pump into the transfer bottle with the filter attached to the discharge end and start the pump. Pre-rinse the filter with approximately 50-mL of sample water prior to filling the sample containers.

3.9 Using the discharge from the filter cartridge, fill one 1-liter polyethylene sample bottle for dissolved metals. Repeat steps 3.8 and 3.9 for these sample containers.

3.10 Obtain measurements of dissolved oxygen, pH, specific conductance, temperature, turbidity, and oxidation-reduction potential using the multi-parameter water-quality meter and LaMotte Turbidity Meter (see SOP-12). Record the readings in the appropriate fields on the Surface Water Sample Log Sheet.

3.11 Decontaminate all equipment and load the equipment and the sample cooler in the sample vehicle for transport.

4.0 SMALL STREAM FLOW ESTIMATION PROCEDURES

4.1 Establish a transect across the stream, along a straight reach where the stream is relatively narrow and velocity across the stream is relatively uniform. Orient the transect perpendicular to the flow.

4.2 Mark both ends of the transect with colored plastic flagging tape. Tie the flagging to a tree branch or other elevated (about eye level) object. Drive wooden stakes into the ground at both ends of transect and tack a short piece of brightly colored flagging to the stakes. Use a black

marker to identify the transect number, which will be the same as the surface water sample location.

4.3 Measure the total distance across the stream with a measuring tape and record the stream width (in feet) in the field logbook.

4.4 Measure the depth of water in the stream at three or four points across the stream. Record these measurements and calculate an average depth. Record the average depth (in feet) in the field logbook.

4.5 Measure a distance of 20 feet upstream of the transect. Tie a piece of flagging on a tree branch or other visible location above the stream.

4.6 Go another 20 feet upstream and place the orange or apple in the center of the stream or where the water current is fastest.

4.7 Using a watch with a timer or a stopwatch, measure the length of time it takes the orange (or apple) to travel from the first marker flag to the transect location (distance of 20 feet). Record the time.

4.8 Repeat step 3.7 two more times. Record the readings and calculate an average time.

4.9 Calculate approximate flow rate in the stream channel by multiplying the stream width (from step 3.3) times the average depth (from step 3.4) times the average velocity (from step 3.8). Record the estimated flow rate (in ft^3/s) in the field logbook and on the Surface Water Sample Collection Log Sheet.

5.0 ATTACHMENTS

1. Surface Water Sample Log Sheet

ATTACHMENT 1

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	ORP
Date:	Visual	Standard	mS/cm	Degrees C	NTU	mg/l	%	mV
Time:								
Depth:								
Method:								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

STANDARD OPERATING PROCEDURE
SOP-08

SEDIMENT SAMPLING

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for sediment sampling in streams and other waterways.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following field forms and equipment are required for sediment sampling.

Sediment Sample Log Forms: A copy of this form is attached at the end of this SOP.
Writing utensil (preferably black pen with indelible ink)
Indelible marker
Bound field logbook
Disposable plastic trowels
Survey stakes and flagging: Used to mark sampling locations after completion of sampling.
Labeled sample containers: See SOP-01 for sample identification procedures. Sample containers are certified clean by the laboratory supplying the containers.
Sealable polyethylene bags
Shipping containers (containing ice)
Disposable medical-grade gloves (e.g., latex, nitrile)
Chain-of-Custody Form

3.0 SEDIMENT SAMPLE LOCATION SELECTION

In general, sediments composed of fine-grained materials with greater surface area available for adsorption are more desirable for sample selection. The fined-grained materials may act as a sink or reservoir for adsorbing heavy metals and organic contaminants even if surface runoff concentrations are below detection limits. Therefore, it is important to locate the specific sampling points where the sediment has the greatest percentage of fine particles. The sampling personnel will determine specific sampling locations with these goals in mind.

4.0 SEDIMENT SAMPLING PROCEDURES

4.1 The sampler will wear clean, disposable medical-grade gloves. Clear vegetative matter or debris, if present, from the sample location using a disposable sampling trowel or spoon. Use the trowel to dig up and homogenize the sediment in an 18-inch-diameter circular area that is 6 inches deep. Stir the sediment within the circular area; do not move the sediment outside the circle. Also, do not dig or stir sediment that is deeper than 6 inches below the ground surface, until the next depth interval is sampled.

- Use the same trowel to scoop the homogenized sediment into the requisite labeled sample container(s).

4.2 Record the sample time (using military time) on the Sediment Sample Log Form and sample container labels. Record all other information required on the labels as specified by SOP-01.

4.3 Place the labeled sample container into a sealable polyethylene bag and then place the bag holding the sample container into a cooler containing ice.

4.4 Record date, sampling site, site conditions, location map, and other information (e.g., presence and flow rate of water in channel) on the Sediment Collection Log Sheet. Enter the sample information onto the Chain-of-Custody Form in accordance with SOP-03.

4.5 Using an indelible marker, write the sample identification on a survey stake, and drive the stake into the ground at the sample location. Tack a piece of brightly colored flagging to the stake. In addition, tie a piece of flagging to an overhead tree branch or other eye-level object to improve the ability to relocate the sampling site in the future.

5.0 ATTACHMENTS

1. Soil and Sediment Sample Log Sheet

ATTACHMENT 1

SOIL AND 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:

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

COMPOSITE SAMPLE DATA:

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

STANDARD OPERATING PROCEDURE
SOP-09

MANAGEMENT OF INVESTIGATION-DERIVED WASTE

1.0 PURPOSE

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

- Decontamination solutions
- Personal protective equipment and clothing (PPE)
- Miscellaneous trash and incidental items

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Health and safety equipment (with PPE)
Decontamination equipment
Field logbook
Writing utensil (preferably black pen with indelible ink)
Plastic sheeting and/or tarps
55-gallon drums with sealable lids
IDW labels for drums
Plastic garbage bags

3.0 PROCEDURES

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

3.1 Liquid Wastes

Liquid wastes that will be generated during the site activities include decontamination solutions from sampling equipment. These wastes will be collected and containerized in a central location at Indian Head Stump Neck Facility for proper disposal.

3.2 Solid Wastes

Solid wastes that may be generated during the site activities include collection of bullets and lead shot from surface soil samples. This waste will be containerized and handed over to the base point of contact at the conclusion of field activities.

3.3 PPE and Incidental Trash

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

STANDARD OPERATING PROCEDURE
SOP-10

BOREHOLE AND SOIL SAMPLE LOGGING

1.0 PURPOSE

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

2.0 FIELD FORMS AND EQUIPMENT

Knife
Ruler (marked in tenths and hundredths of feet)
Boring Log: An example of this form is attached.
Photoionization detector (PID) (see SOP-13)
Writing utensil (preferably black pen with indelible ink)

3.0 RESPONSIBILITIES

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

4.0 PROCEDURES FOR BOREHOLE AND SAMPLE LOGGING

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

4.1 USCS Classification

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

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

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

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

4.2 Color

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

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

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

4.3 Relative Density and Consistency

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

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

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

CONSISTENCY FOR COHESIVE SOILS

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

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

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

4.4 Weight Percentages

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

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

Examples:

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

4.5 Moisture

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

4.6 Classification of Soil Grain Size for Chemical Analysis

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

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

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

4.7 Summary of Soil Classification

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

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

4.0 ATTACHMENTS

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

ATTACHMENT 1

FIGURE 1 - UNIFIED SOIL CLASSIFICATION SYSTEM

Unified Soil Classification System

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

Grain Size Chart

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

Relative Density (SPT)

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

Consistency (SPT)

SILTS AND CLAYS	BLOWS/FOOT
VERY SOFT	0 – 2
SOFT	2 – 4
MEDIUM STIFF	4 – 8
STIFF	8 – 16
VERY STIFF	16 – 22
HARD	OVER 32

ATTACHMENT 2

BORING LOG

Tetra Tech NUS, Inc.

BORING LOG

Page ___ of ___

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: 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 reponse read.

Drilling Area Background (ppm): _____

Remarks: _____

Converted to Well: Yes _____ No _____ Well I.D. #: _____

STANDARD OPERATING PROCEDURE
SOP-11

SAMPLE PRESERVATION, PACKAGING, AND SHIPPING

1.0 PURPOSE

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

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Shipping labels
Custody seals
Chain-of-custody (COC) form(s)
Sample containers with preservatives: All sample containers for analysis by fixed-base laboratories will be supplied, with preservatives added (if required) and deemed certified clean by the laboratory.
Sample shipping containers (coolers): All sample shipping containers are supplied by the laboratory.
Packaging material: Bubble wrap, sealable polyethylene bags, strapping tape, etc.

3.0 PROCEDURES FOR SAMPLE PRESERVATION, PACKAGING, AND SHIPPING

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

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

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

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

3.5 Inspect the insulated shipping cooler. Check for any cracks, holes, broken handles, etc. If the cooler has a drain plug, make certain it is sealed shut, both inside and outside of the cooler. If the cooler is questionable for shipping, the cooler must be discarded.

3.6 Line the cooler with large plastic bag, and line the bottom of the cooler with a layer of bubble wrap. Place the sample containers into the shipping cooler in an upright position (containers will be upright, with the exception of any 40-ml vials). Continue filling the cooler with ice until the cooler is nearly full and the movement of the sample containers is limited.

3.7 Wrap the large plastic bag closed and secure with tape.

3.8 Place the original (top) signed copy of the COC form inside a sealable polyethylene bag. Tape the bag to the inside of the lid of the shipping cooler.

3.9 Close the cooler and seal the cooler with approximately four wraps of strapping tape at each end of the cooler. Prior to wrapping the last wrap of strapping tape, apply a signed and dated custody seal to each side of the cooler (one per side). Cover the custody seal with the last wrap of tape. This will provide a tamper evident custody seal system for the sample shipment.

3.10 Affix shipping labels to each of the coolers, ensuring all of the shipping information is filled in properly. Overnight (e.g., FedEx Priority Overnight) courier services will be used for all sample shipments.

3.11 All samples will be shipped to the laboratory no more than 72 hours after collection. Under no circumstances should sample hold times be exceeded.

STANDARD OPERATING PROCEDURE
SOP-12

CALIBRATION AND CARE OF WATER QUALITY METER

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures for the calibration and maintenance of field instruments used to measure water quality and for the proper documentation of calibration and maintenance. The multi-parameter water quality meter will be used to measure pH, temperature, oxidation-reduction potential (ORP), specific conductance (SC), and dissolved oxygen (DO). A LaMotte Model 2020 turbidity meter will be used to measure turbidity in water. The water meter will have a multiprobe sensor that can be used in conjunction with a flow-through cell attached to a pump discharge tube to measure water-quality parameters in a surface water body such as a stream, pond, or drainage ditch.

2.0 FIELD FORMS AND EQUIPMENT LIST

The following logbooks, forms, equipment, and supplies are required:

Site logbook
Writing utensil (preferably black pen with indelible ink)
Equipment calibration log sheet
Multi-parameter Water Quality Meter
Equipment manual
Calibration kit
Deionized water, paper towels, spray bottle, etc.
Disposable medical-grade gloves (e.g., latex, nitrile)

3.0 PROCEDURES

This section describes the general calibration procedure for the multi-parameter water quality meter. The meter will be supplied with an instruction manual. The manual will be on site and will be used as the calibration guidance document for the meter's calibration. This procedure will list requirements for frequency of calibration and checks to be performed on the meter.

The multi-parameter water-quality meter may be used to measure open water bodies (streams, ponds, springs, etc.) with the probe guard installed. The parameters to be measured by the meter for this field effort are as follows:

- DO
- SC
- Temperature
- pH
- ORP

3.1 Documentation

The Equipment Calibration Log is used to document calibration of measuring equipment used in the field. The Equipment Calibration Log documents that the manufacturer's instructions were followed for calibration of the equipment, including the frequency of calibration, type of standards used, and checks performed on calibration during the course of using the equipment. An Equipment Calibration Log must be maintained for each measuring device that requires calibration. Entries must be made for each day the equipment is used. A blank Equipment Calibration Log form is attached at the end of this SOP.

3.2 Calibration

All the parameters listed in Section 3.0 must be calibrated prior to the start of each field effort. After this initial calibration, the meter will be checked each day that it is used. If the check shows any out-of-specification readings, the specific probe will be recalibrated. Meter specifications can be found in the equipment manual. Calibration and calibration checks will be documented in the field logbook and on the Equipment Calibration Log. The name, lot number, and expiration date for all calibration buffers and standards used will be recorded on the Equipment Calibration Log. The meter's model, serial number, and name of rental company will also be recorded on the equipment calibration form.

3.3 Tips for Good Calibration

- The DO calibration is a water-saturated air calibration. Make certain to loosen the calibration cup seal to allow pressure to equilibrate before calibrating.

- Make certain that sensors are completely submersed in solution and readings are stable when calibration values are entered.

- Use a small amount of calibration solution (previously used solution may be used, then discarded for this purpose) to pre-rinse the sonde.

- Fill a bucket with ambient temperature water to rinse the sonde between calibration solutions.

- Make sure to rinse and dry the probe between calibration solutions. This will reduce carry-over contamination and increase the accuracy of the calibration.

4.0 MAINTENANCE

The water quality meter will be rented for the duration of each brief field effort. Therefore, little field maintenance will be required. For any maintenance other than the routine cleaning, calibrating, or battery charging, the instrument should be returned to the vendor and a replacement sent immediately to the job site.

4.1 Meter Storage

For this field effort, the meter storage will be short term, [i.e. over night or between work shifts (2-day break)]. During these breaks, the meter will be charged. One-half inch of tap or distilled water will be placed in the meter calibration cup and the cup threaded onto the sonde. The key for short-term storage of probes is to use a minimal amount of water so the calibration cup will remain at 100 percent humidity. The water level must be low enough so that none of the probes are actually immersed. Proper storage of the sonde between usage will extend its life and will also ensure that the unit is ready for use as quickly as possible for the next application.

Multi-parameter short term storage key points:

- Use enough water to provide humidity but not enough to cover the probe surfaces.
- Make sure the storage vessel is sealed to minimize evaporation.
- Check periodically to make certain that water is still present.

4.2 Probe Cleaning

- Rinse the probe thoroughly with potable water.
- Wash the probe in a mild solution of Liquinox and water and wipe with paper towels and/or cotton swabs.
- Rinse and soak the probe in deionized water.
- If stronger cleaning is required, consult the equipment manual.

Note: Reagents that are used to calibrate and check the water quality meter may be hazardous. Review the health and safety plan, appropriate Appendix of the equipment manual, and Material Safety Data Sheets (MSDSs), all of which are on file in the field trailer.

5.0 ATTACHMENTS

1. Equipment Calibration Log

ATTACHMENT 1
EQUIPMENT CALIBRATION LOG

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : _____

SITE NAME: _____

PROJECT No.: _____

INSTRUMENT NAME/MODEL: _____

MANUFACTURER: _____

SERIAL NUMBER: _____

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

STANDARD OPERATING PROCEDURE
SOP-13

FIELD PORTABLE X-RAY FLUORESCENCE ANALYSIS OF SOIL AND SEDIMENT SAMPLES USING THE INNOV-X ALPHA SERIES INSTRUMENT

1.0 PURPOSE

This procedure is for the semiquantitative analysis of metallic lead particles and chemical compounds of lead in soil using a field portable x-ray fluorescence (FPXRF) spectrometer. This procedure is based on the United States Environmental Protection Agency (USEPA)-approved XRF field screening method for elemental analysis (Method 6200).

2.0 SCOPE, APPLICATION, AND LIMITATIONS

2.1 Scope of Procedure

Analysis of any other elements beside lead using FPXRF may require changes to this Standard Operating Procedure (SOP), and are therefore outside the scope of this SOP.

Although it is possible to use FPXRF to measure analytes in situ, this SOP requires removal of a soil sample from its native environment prior to analysis. By removing, drying, and homogenizing the sample prior to analysis, more precise and accurate results are obtained.

2.2 Analyst Training

Use of this method is restricted to personnel both trained and knowledgeable in the operation of the Innov-X alpha series XRF instrument or under the supervision of a trained and knowledgeable individual. Proper training for the safe operation of the instrument should be completed by the analyst prior to analysis. This training may be obtained directly from INNOV-X, an INNOV-X instrument distributor or lessor, or another trained Tetra Tech person.

3.0 ACRONYMS AND ABBREVIATIONS

FPXRF: Field portable x-ray fluorescence.
mg/kg: milligrams per kilogram.
MDL: Method detection limit.
PQL: Practical quantitation limit.
QC: Quality control.
RPD: Relative percent difference.
USGS: United States Geological Survey.
XRF: X-ray fluorescence.

4.0 RESPONSIBILITIES

Analyst/Chemist - Responsible for all aspects of sample preparation and analysis including equipment maintenance. Also responsible for maintaining chain-of-custody of samples after receipt from sampling personnel.

5.0 PROCEDURES

5.1 Safety

5.1.1 Radiation Safety

Radiation safety practices for the INNOV-X instrument can be found in the operator's manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer.

An additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply. The danger of electric shock is as substantial as the danger from radiation but is often overlooked because of its familiarity.

5.1.2 Protective Equipment

Analysts must wear disposable plastic gloves whenever sample aliquots are being transferred from one vessel to another. Consult the health and safety plan for other protection requirements.

5.2 Apparatus and Materials

Apparatus and materials consist of the following:

INNOV-X Alpha Series FPXRF spectrometer with data processing unit (iPAQ) pocket personnel computer):

INNOV-X Alpha Series XRF instrument manual to match the INNOV-X Alpha Series instrument.

Aluminum drying pans or aluminum foil: Sized suitably to hold as much as 50 grams of sample and fit into the drying oven.

Calibration verification check sample: A National Institute of Standards and Technology (NIST) or other Standard reference material (SRM) that contains lead in a concentration range that is compatible with the project objectives to verify the accuracy of the instrument. SRMs can be obtained from the NIST, the U.S. Geological Survey (USGS), the Canadian National Research Council, and the national bureau of standards in foreign nations. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories. Acceptable limits for SRM percent recoveries are usually provided with the SRM. In their absence, a limit of ± 30 percent will be used as a guideline.

Instrument Blank: May be silicon dioxide, a Teflon block, a quartz block, "clean" sand, or lithium carbonate.

Lead calibration check standard: Supplied by the FPXRF manufacturer.

Method blank material for performing method blank checks: May be lead-free silica sand or lithium carbonate that undergoes the same preparation procedure as the samples.

Battery charger.

Polyethylene sample cups: 31 millimeters (mm) to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

X-ray window film: Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 micrometers (µm) thick.

Sample containers: glass or plastic to store samples.

Sieves: 60-mesh Stainless steel, Nylon, or equivalent for preparing soil and sediment samples if necessary.

Trowels: for collecting soil samples.

Plastic bags: used for collection and homogenization of soil samples. May also be used as sample presentation device.

Drying oven: standard convection or toaster oven, for soil samples that require drying.

Rolling pin (optional): Wooden rolling pin for crushing samples.

5.3 Sample Collection, Preservation, and Handling

Samples shall be provided to the FPXRF analyst in plastic bags. The analyst is responsible for maintaining chain-of-custody of all samples until all analyses have been successfully completed. No sample preservation is necessary. All samples shall be handled in accordance with sample handling SOPs in effect for the field event.

5.4 Preventive Maintenance

Refer to the instrument manual for specific manufacturer's recommendations.

5.5 Instrument Start-Up

5.5.1 Ensure the pocket PC (iPAQ) is plugged into the FPXRF instrument body and install a fully charged battery into the instrument.

5.5.2 Press the ON/OFF button on the base of the pistol grip of the instrument. If the iPAQ does not automatically power up, press the Power button in the to right corner of the iPAQ.

5.5.3 Tap the Microsoft icon at the upper left corner of the iPAQ.

5.5.4 Chose START.

5.5.5 Tap "Soil Mode" on the menu or choose Mode (bottom of screen) and then choose Soil Mode from the drop down menu.

5.5.6 Allow the instrument to warm up (approximately 3 minutes).

5.5.7 Release the manual trigger lock.

5.5.8 Standardize the instrument in accordance with Section 5.6.

5.6 Standardization/Calibration Check

It is not possible to start an analysis if the instrument has not been standardized. To verify proper calibration of the instrument it is necessary to periodically standardize it using the automated standardization procedure. This must be done anytime the instrument is restarted and every 4 hours of operation, although re-standardization may be done at any other time (e.g., when instrument drift is suspected).

5.6.1 Click the standardization piece (supplied with the instrument) on the front of the instrument, verifying that the solid portion of the standard completely covers the analysis window.

5.6.2 Select "Tap here to Standardize" or select *File* → *Standardize*. The red light on top of the instrument will blink indicating that the instrument is producing x-rays and the shutter is open. The amber light on the rear of the instrument will also be illuminated and a status bar will appear to display the progress of the standardization.

5.6.3 Upon successful standardization the message "Successful Standardization" will appear along with the instrument resolution. In this case tap "ok" to dismiss the completion message. If problems are encountered, either follow the prompts that appear and/or repeat the standardization. Contact the FOL if problems persist. Take note of any error messages that appear as they may be useful if the instrument manufacturer must be contacted. Additional assistance is also available in the manufacturer's instrument manual.

5.7 Quality Control

The quality control (QC) program includes analysis of blanks calibration verification checks, duplicate analyses, and field duplicate samples. For all the above areas, any identified problems and corrective

action must be documented in the instrument run log, analysis narrative report, and instrument maintenance log or standards log (as applicable). Identical operating conditions will be used for each sample.

5.7.1 Laboratory Blanks

Two types of blank samples shall be analyzed for FPXRF analysis: instrument blanks and method blanks.

5.7.1.1 At the beginning of each day, at the end of each day, and after every 20th sample or when potential contamination of the instrument is suspected, analyze an instrument blank to verify that no contamination exists in the spectrometer or on the probe window.

If the lead concentration in the blank exceeds the method detection limit (MDL), see Section 5.9.3) check the probe window and other potentially contaminated instrument components for contamination. If contamination is not causing the elevated blank readings, restandardize the instrument according to manufacturer's instructions.

5.7.1.2 After every 20th sample analyze a method blank. If the method blank lead concentration exceeds the practical quantitation limit (PQL, see Section 5.9.4), identify the cause of the elevated lead concentration and reanalyze all samples since the last acceptable method blank.

5.7.2 Calibration Verification Checks

5.7.2.1 After performing each blank check (Section 5.7.2), analyze a calibration verification check sample to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest.

5.7.2.2 If the measured lead percent recovery (See Section 5.9.1) is less than 60 percent or greater than 135 percent, reanalyze the check sample. If the value continues to fall outside this acceptance range, the instrument should be restandardized according to the manufacture instructions and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

5.7.3 XRF Duplicate Samples

XRF duplicate samples are two portions of the same sample that have been prepared and homogenized together, and then split and analyzed in the same manner by the XRF analyst.

5.7.3.1 Analyze an XRF duplicate at a frequency of 1 per 20 or once per day, whichever is more frequent.

5.7.3.2 If the computed RPD (See Section 5.9.2) exceeds 50 percent reanalyze both samples. If the RPD again exceeds 50 percent RPD consider whether the high degree of imprecision is caused by sample heterogeneity or other causes. This assessment may be aided by repeating the analysis of a sample that was analyzed previously. If the observed imprecision is attributed to sample heterogeneity, increase the number of readings made per sample to try to limit the imprecision and repeat the analyses. If this does not correct the problem notify the FOL.

5.8 Sample Analysis

Note:

This section provides sample analysis instructions, assuming that appropriate instrument start-up and calibration checks have been completed. The longer the instrument count time, the lower the detection limits and the less uncertainty there is with a recorded result. Count time is user-selectable through the instrument's software. Because the XRF data will be used in a screening capacity to make preliminary decisions concerning the soil concentrations relative to 400 mg/kg, it is not necessary to obtain a high degree of accuracy or precision with the instrument. Therefore, count times should be limited to less than 60 seconds unless an usually high degree of precision is expected. To change the count time, select Options → Setup Testing and enter the same value (in seconds) to minimum and maximum count times.

Note:

Section 5.7 identifies the appropriate frequencies for conducting various QC sample analyses and the associated acceptance limits and corrective actions for potentially unsuitable conditions. The specified QC analysis frequencies are minimum frequencies. More frequent QC sample analyses are permitted, especially when diagnosing quality problems.

5.8.1 Ensure that calibration checks and blanks have been analyzed according to Sections 5.6, 5.7.1, and 5.7.2. Count times shall be at least 30 seconds but generally less than 60 seconds.

5.8.2 Acquire enough soil sample to fill an 8-ounce jar and separate from it all particles greater than the size of a pea.

5.8.3 Homogenize the remaining finer grained portion of the sample by simple mixing until it appears as uniform in texture and composition as practicable. Mixing may be done in a beaker or other suitable lead-free container. If the sample is moist and has high clay content, it may be kneaded in a plastic bag. Mixing shall continue for at least two minutes to ensure that the sample is well mixed. To aid mixing, the sample may be placed into a thick-walled (3 mil or thicker) gallon-sized freezer bag (e.g., ZipLoc®) and rolled flat with a rolling pin to break up large chunks of dirt.

5.8.4 Place approximately 20 to 50 grams (one U.S. nickel weighs about 5 grams) in a suitable container (e.g., aluminum drying pan) for drying.

5.8.5 Dry the homogenized sample from Step 5.8.4 for approximately 20 to 30 minutes in the oven at a temperature not greater than 150°C (a setting of approximately 300°F). If the sample is not visibly dry after this initial drying time, place the sample back into the oven until the sample is dry.

5.8.6 Re-homogenize the dried sample aliquot in a beaker or other suitable lead-free container to obtain a well mixed soil sample. Mixing shall continue for at least one minute.

5.8.7 Place a portion (approximately 1.5 cubic inches) of the dried, homogenized sample aliquot into the instrument manufacturer's recommended sample cup (e.g., a 31.0-mm polyethylene sample cup (or equivalent) or place it in a thin-walled (1.0 mil or thinner) plastic sandwich bag (e.g., ZipLoc®).

5.8.8 If using a disposable plastic sample cup, ensure the cup is at least three-quarters full and cover with mylar (or other) film per the manufacturer's recommendations.

5.8.9 Present the sample to the instrument in Soil Mode.

5.8.10 Perform a single pull of the trigger to start the count. The count time shall be the same as was used for the calibrations, calibration checks, and blank analyses. The message "Test in progress" will appear on the instrument and the red light on top of the instrument and will illuminate.

5.8.11 When the predetermined count period has expired the message "Test complete" will appear on the instrument. A slight delay may also be incurred during which time the message "calculating" may appear to indicate that results are being computed.

5.8.12 Record the displayed results for lead concentration in mg/kg and the associated error on Figure 1.

CAUTION

Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases by the square of the distance from the radioactive source. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample surface should be flat and smooth to provide a good contact surface.

5.8.13 Remix the sample in the plastic bag (or rotate the sample cup approximately one-third of a turn) then acquire another measurement by repeating Steps 5.8.10 and 5.8.11. Record the result and associated error on Figure 1.

5.8.14 Repeat Steps 5.8.10 and 5.8.11. Record the result and associated error on Figure 1 (XRF Field Form).

5.8.15 Based on the degree of precision demonstrated by the three individual measurements, determine whether additional readings should be acquired on the sample. This determination shall be based on professional judgment of the FPXRF analyst and shall consider the degree of precision observed during calibration checks and previous sample analyses. The objective will be to ensure that the average reading reported for each sample is representative of the true sample concentration. If the analyst feels that non-representative readings are being obtained the analyst shall correct the analytical system or notify the FOL prior to continuing with analyses.

5.8.16 Ensure that measured results are reported to the following standards

- Results < 1000 mg/kg (or parts per million) are reported to two significant figures and results > 1000 mg/Kg are reported to three significant figures.
- All values < MDL shall be reported as the MDL and flagged with the letter “U”.
- All values > MDL and < PQL shall be reported as is and flagged with the letter “B”.

5.9 Calculations

5.9.1 Percent Recovery: The equation for determining percent recovery of calibration verification check standards and standard reference materials is:

$$\%R = \frac{\text{Experimental Concentration}}{\text{Certified or Known Concentration}} \text{ X } 100\,\%$$

5.9.2 Relative Percent Difference: The equation for determining relative percent difference for laboratory and field duplicate samples is:

$$RPD = \frac{|\text{Amount in Sample 1} - \text{Amount in Sample 2}|}{0.5\,(\text{Amount in Sample 1} + \text{Amount in Sample 2})} \text{ X } 100\,\%$$

5.9.3 Method Detection Limit (MDL): Because the analyses governed by this SOP are semi-quantitative, the manufacturer-specified detection limit will be reported as the MDL unless the specified detection limit is less than 20 mg/kg. Care will taken to ensure that the appropriate count time is consistent with the reported detection limit. However, no value less than 20 mg/kg will be reported as an MDL.

5.9.4 Practical Quantitation Limit (PQL): Multiply the MDL by 3 to obtain the PQL: PQL = MDL*3

6.0 CORRELATION EVALUATION

To predict lab concentrations based on XRF results a correlation analysis will be performed on the Ordinary Least Squares (OLS) Regression model. The R-Square statistic will be used to measures how well the regression line fits the data (i.e. how well the XRF data can predict the fixed based laboratory results).

First evaluate the OLS assumptions:

1) Determine that there is a linear relationship between the XRF results and fixed based laboratory results.
2) Determine that the residuals of the OLS are normally distributed.
3) Determine that the residuals of the OLS do not display non-constant variance.

If the assumptions of the OLS model are violated transform the data so the assumptions of the model are met.

After demonstrating that the assumptions are valid, compute the OLS model.
From the OLS model compute the R-Square statistic:

$$\left(r = \frac{1}{n-1} \sum \left(\frac{x_i - \bar{x}}{s_x} \right) \left(\frac{y_i - \bar{y}}{s_y} \right) \right)^2$$

7.0 REFERENCES

Innov-X Systems, Inc. Innov-X Systems X-Ray Fluorescence Spectrometers Instruction Manual. Woburn, MA. June 2002.

Stephen Shefsky, NITON Corporation. Comparing Field Portable X-Ray Fluorescence (XRF) To Laboratory Analysis of Heavy Metals In Soil. Presented at the International Symposium of Field Screening Methods for Hazardous Wastes and Toxic Chemicals. Las Vegas, Nevada. January 29-31, 1997.

USEPA (U.S. Environmental Protection Agency), Method 6200: Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment, Office of Solid Waste, Washington, D.C. January 1998.

USEPA, Region I, Northeast Waste Management Officials' Association (NEWMOA) Technology Review Committee Advisory Opinion. Innovative Technology: X-Ray Fluorescence Field Analysis. September 21, 1999.

FIGURE 1

FIELD PORTABLE XRF SAMPLE ANALYSIS LOG SHEET

Page___ of ___

Project Site Name: ______________________ Run Length/Duration: ______________________
Project No.: ______________________ Measurement Units: ______________________
Analyzed By: ______________________ Reference/Calibration: ______________________

SAMPLING AND FIELD PORTABLE XRF RESULTS DATA:

SAMPLE ID	ANALYSIS DATE	ANALYSIS TIME	ELEMENT ANALYSIS	RESULT RUN 1	RESULT RUN 2	RESULT RUN 3	RESULTS AVERAGE	ERROR RUN 1	ERROR RUN 2	ERROR RUN 3	ERROR AVERAGE

STANDARD OPERATING PROCEDURE

SOP-14

LARGE WATER BODY SEDIMENT SAMPLING

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for sediment sampling in large water bodies from a motor powered work boat.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following field forms and equipment are required for sediment sampling.

Sediment Sample Log Forms: See Attachment to this SOP.
Writing utensil (preferably black pen with indelible ink)
Indelible marker
Bound field logbook
Stainless steel trowels
Large stainless steel bowls
Aluminum Foil
Clamshell dredge 15 pound or 25 pound dredge with rope: If the 25 pound dredge is needed a winch and boom will be required
Global Positioning System (GPS): Used to locate and mark sampling locations.
Labeled sample containers: See SOP-02 for sample identification procedures. Sample containers are certified clean by the laboratory supplying the containers.
Decontamination equipment: See SOP-04 for decontamination equipment
Sealable polyethylene bags
Shipping containers (containing ice)
Disposable medical-grade gloves (e.g., latex, nitrile)
Otter plastic gloves (e.g., nitrile)
Chain-of-Custody Form
Personal floatation devices (life jackets)
18-ft wide-bodied, flat-bottomed boat powered by an outboard motor

3.0 SEDIMENT SAMPLE LOCATION SELECTION

Sediment samples shall be collected throughout a study area in a grid like pattern or at locations established by the FOL. The sample locations x-y coordinates will be electronically loaded in to a hand held global positioning system. These coordinates are then used to navigate to and locate the sampling locations using the global positioning system.

4.0 SEDIMENT SAMPLING PROCEDURES

4.1 The work boat will be loaded with all necessary equipment in order to find sampling locations, collect the samples, and clean the sampling equipment, minimizing the number of times required to return to shore.

4.2 The sampler will wear clean, disposable medical-grade gloves with outer nitrile type gloves. Attach the clamshell dredge to a tie line or rope, spread the sampling jaws and place the spring loaded keeper pin into the jaw arms of the dredge, thus locking the jaws open. Lower the dredge into the water, and down to the river bottom in one smooth motion allowing the dredge to sink steadily to the river bottom. The jaws will automatically close when the retrieval is initiated. Retrieve the dredge with sediment and before opening place the dredge into a clean stainless steel bowl. Carefully open the jaws and allow the dredge to empty the sediment in to the stainless steel bowl. Use the trowel to homogenize the sediment in the stainless steel bowl. Use the same trowel to scoop the homogenized sediment into the requisite labeled sample container(s).

4.3 Record the sample time (using military time) on the Sediment Sample Log Form and sample container labels. Record all other information required on the labels as specified by SOP-01.

4.4 Place the labeled sample container into a sealable polyethylene bag and then place the bag holding the sample container into a cooler containing ice.

4.5 Record date, sampling site, site conditions, location map, and other information (e.g., depth to the river bottom) on the Sediment Collection Log Sheet. Enter the sample information onto the Chain-of-Custody Form in accordance with SOP-03.

4.6 Using the GPS unit, survey in the sample location in order to relocate the sampling site in the future.

5.0 ATTACHMENTS

1. Soil and Sediment Sample Log Sheet

ATTACHMENT 1

SOIL AND 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 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):

STANDARD OPERATING PROCEDURE
NUMBER SOP-15

COMPOSITE SAMPLING FOR SOIL AND SEDIMENT

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for collecting composite surface soil and sediment samples using the hand auger technique to support the field investigations at NSF Indian Head and the NSF Indian Head – Stump Neck Annex facility.

The basis of rationale for collecting composite surface soil and sediment samples is to limit the variability of analytical data that is expected to be caused by the heterogeneous nature of explosives releases to soil.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following field forms and equipment are required for composite soil and sediment sampling.

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

Indelible marker

Stainless steel auger buckets

Stainless steel extension rods

Cross handle

Plastic storage bags

Sample tags

Shipping containers (containing ice)

Disposable plastic trowels or Stainless steel trowels

Stainless steel mixing bowls

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

Field Forms: Soil and Sediment Sample Log (SOP-05 and 08), Soil Boring Log (SOP-10).

Electronic label maker

3.0 SAMPLING PROCEDURES

Hand augering will be employed to collect soil cores (see SOP-05). The sampler will wear clean, disposable, medical-grade gloves.

3.1 SOIL COMPOSITE SAMPLING PROCEDURES

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

3.1.2 Turn the hand auger sampler into the ground to a depth of 2 feet. The 0- to 2-foot depth soil interval is considered to be the surface soil.

3.1.3 Log the soil core on the Boring Log Sheet (see SOP-10).

3.1.4 Repeat steps 3.1.1–3.1.3 in the designated sampling area for a specific sample until the desired number of discrete soil cores are collected.

3.1.5 Place equal amounts of the soil cores for a given sample in a stainless-steel mixing bowl and homogenize using a disposable dedicated plastic trowel. Alternatively, mix the samples in a large disposable Ziploc bag. Place the composite soil sample into the laboratory supplied sampling container, print, and affix electronically printed label.

3.1.6 For samples that are to be submitted for laboratory analysis, place the labeled sample container into a plastic storage bag and then place the plastic storage bag holding the sample container into a cooler containing ice.

3.1.7 Complete the required information on the electronic Soil and Sediment Sample Log Sheet (copy attached at the end of this SOP). Note the location of each aliquot in the "OBSERVATIONS/NOTES" section of the Soil and Sediment Sample Log Sheet. Only the notes added within the sample analysis block on the Soil and Sediment Sample Log Sheet will be visible on the Chain-of-Custody (COC) Form for review by laboratory personnel.

3.1.8 Decontaminate the hand auger bucket and the bowl and spoon, if employed, in the field between each composite sample location.

3.2 SEDIMENT COMPOSITE SAMPLING PROCEDURES

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

3.2.2 Remove sediment sample from a depth of 0- to 6-inches.

3.2.3 Repeat steps 3.2.1–3.2.2 in the designated sampling area for a specific sample until the desired number of discrete sediment sample locations are collected.

3.2.4 Place equal amounts of the sediment for a given sample in a stainless-steel mixing bowl and homogenize using a decontaminated stainless steel spoon or dedicated disposable towel. Alternatively, mix the samples in a large disposable Ziploc bag. Place the composite sediment sample into the laboratory supplied sampling container, print, and affix electronically printed label.

3.2.5 For samples that are to be submitted for laboratory analysis, place the labeled sample container into a plastic storage bag and then place the plastic storage bag holding the sample container into a cooler containing ice.

3.2.6 Complete the required information on the electronic Soil and Sediment Sample Log Sheet (copy attached at the end of this SOP). Note the location of each aliquot in the "OBSERVATIONS/NOTES" section of the Soil and Sediment Sample Log Sheet. Only the notes added within the sample analysis block on the Soil and Sediment Sample Log Sheet will be visible on the COC Form for review by laboratory personnel.

3.2.7 Decontaminate the hand auger bucket and the bowl and spoon, if employed, in the field between each composite sample location.

4.0 ATTACHMENTS

1. Soil and Sediment Sample Log Sheet

ATTACHMENT 1

SOIL AND SEDIMENT SAMPLE LOG SHEET

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

COMPOSITE SAMPLE DATA:

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

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

APPENDIX B

LABORATORY STANDARD OPERATING PROCEDURES

APPENDIX B

TABLE OF CONTENTS

LABORATORY STANDARD OPERATING PROCEDURES

KATAHDIN ANALYTICAL SERVICES

CA-213	Analysis of Semivolatile Organic Compounds by: SW 846 8270 SIM
CA-502	Preparation of Aqueous Samples for Extractable Semivolatile Analysis
CA-512	Preparation of Sediment/Soil Samples by Sonication Using Method 3550 for Subsequent Extractable Semivolatile Analysis
CA-604	Acid Digestion of Aqueous Samples by USEPA Method 3010 for ICP Analysis of Total or Dissolved Metals
CA-605	Acid Digestion of Solid Samples by USEPA Method 3050 for Metals Analysis by ICP-AES and GFAA
CA-627	Trace Metals Analysis by ICP-MS using USEPA Method 6020
CA-709	pH Concentration Measurements in Soil Matrices – SW 846 Method 9045
CA-737	Cation Exchange Capacity of Soils using USEPA Method 9081
CA-741	Determination of Total Organic Carbon in Solids using the USEPA Region 2 Lloyd Kahn Method
SD-902	Sample Receipt and Internal Control
SD-903	Sample Disposal

ANALYTICAL LABORATORY SERVICES, INC.

09-8330B Soil	Ultrasonication of Solids for the Analysis of Explosives by EPA Method 8330B (HPLC)
09-8330W	Solid Phase Extraction of Water for the Analysis of Explosives by EPA Method 8330A (HPLC)
1B-8330	Nitroaromatics and Nitroamines by HPLC with Ultraviolet Detection
19-Waste Disposal	Waste Disposal
19-Rec/Han	Standard Operating Procedure for Sample Receipt/Sample Handling
19-COC	Standard Operating Procedure for Chain of Custody Entry

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:

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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Figure 3 LCS/Matrix Spike Component List
Figure 4 Appendix Ix LCS/Matrix Spike Component List
Figure 5 Copy of EPA Region I Memo Regarding Combination of Extracts for 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

TITLE: **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

TABLE 1, continued

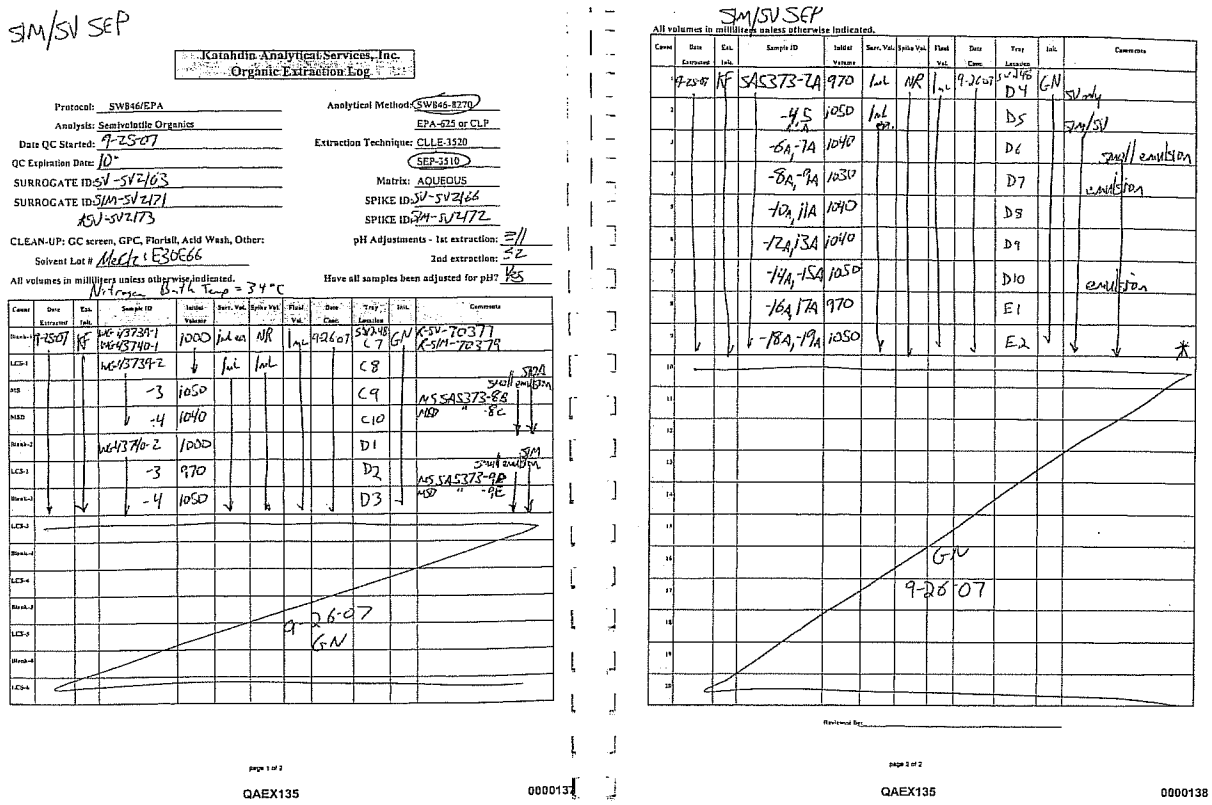
SUMMARY OF METHOD MODIFICATIONS (METHOD 625)

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

TITLE: **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

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

Reviewed By:

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QAEX135 0000138

TITLE: **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 2

SOLVENT/REAGENT LOT CHECK LOGBOOK

SOLVENT:

LOT#:

DATE RECEIVED:

DATE CONCENTRATED:

CONCENTRATED BY:

PREP METHOD:

TRAY LOCATION:

ANALYZED BY:

PASS/FAIL:

TITLE: **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 3

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	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-512
Revision History
Cover Page
Page 1

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

Prepared By: Mike Thomas Date: 09/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: [illegible] Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10·24·00

General Manager: [illegible] Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10·24·00	10/24/00
02	Addition of compounds to Figure 2.	DN	3·28·02	3·28·02
03	Definitions added to section 1.1. Wording was added or changed to clarify sections 4, 5, 6, 7, 8 + 9. Minor changes throughout. New figures.	HRC	11.08.04	11.08.04
04	Updated Sect. 5.0 with current spike solutions prep. Removed section on medium level soil extraction. Replaced figure 3 and 4 with current LCS/MS spike components. Minor corrections to sect. 1.3, 4.24, 6.0 and 7.12. Updated logbook	LAD	04/06	04/06
05	Many changes made throughout, including but not limited to, waste information, updated spikes and surrogates, added SIM LCS/D and MS/D information, updated Table 1. Please refer to the QAM/SOP change form filed w/ SOP in QA for a detailed list of changes.	LAD	09/07	09/07

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

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KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-512-05**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS.**

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TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for analysis of extractable semivolatile organic compounds. This SOP is specifically applicable to EPA Method 3550.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training”.

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab

notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical 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 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.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

2.0 SUMMARY OF METHOD

A 30 gram portion of sediment/soil is mixed with anhydrous powdered sodium sulfate and extracted with 1:1 methylene chloride/acetone (v/v) using an ultrasonic probe. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory.

At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis. Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with methylene chloride. Brand names and catalog numbers are included below for illustration purposes only.

4.1 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.

4.2 Sonicator ultrasonic processor XL – Misonix (or equivalent) equipped with dual titanium 3/4" horn extenders for extracting two samples at a time.

4.3 Powder funnels, 100 mm diameter, 35 mm stem

4.4 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL
Evaporative flask - 500 mL
Snyder column - 3-ball macro

4.5 Filter paper, 7.0 cm, Whatman #4

4.6 Vacuum filtration flask - 500 mL Erlenmeyer

4.7 Buchner funnel, porcelain, Coors® with 85 mm plate diameter (or equivalent)

4.8 Beakers - 400 mL

4.9 Boiling chips - approximately 12 mesh, silicon carbide (carborundum or equivalent). Soxhlet extract overnight in methylene chloride.

4.10 Water bath - eight position concentric ring bath, or equivalent, equipped with a calibrated thermometer. The bath should be used in a hood.

4.11 Balance - capable of accurately weighing ± 0.1 g.

4.12 Vials and caps – 1.8 mL with PTFE/silicone septa and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.

4.13 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.14 Pasteur pipets - disposable, 5 ¾ ".

4.15 Nitrogen evaporation apparatus.

4.16 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

5.0 REAGENTS

5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, certified by the manufacturer/vendor as purified heating to 400°C prior to receipt by the laboratory. Solvent rinse immediately prior to use by rinsing three times with pesticide grade methylene chloride. (Jost Chemical anhydrous powder, catalog #2797 or equivalent, and Jost Chemical granular crystals, catalog #2796 or equivalent).

5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated by lot prior to use by concentration of approximately 400 mL to 1.0 mL followed by GC/MS analysis.

5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand.

5.4 Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound	Conc.
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.6 Base/Neutral and Acid (SVOA) Matrix Spike/Lab Control Sample Spiking Solution - Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 50 ug/mL for base/neutrals and 100 ug/mL for acids. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check

samples indicates a problem.

5.7 Base/Neutral and Acid (SVOA APPENDIX IX) Matrix Spike/Lab Control Sample Spiking Solution. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 100 µg/mL for each compound. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem

5.8 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2 ug/mL for base/neutral. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL in methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3550 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

Some solid samples may need to be cleaned up to reduce matrix interferences. The cleanup procedure employed is gel permeation chromatography (GPC). The organic department manager should be consulted to determine if a particular sample should be subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs.

Sign chain-of-custody when removing and replacing samples in storage locations, and fill out the sample preparation/extraction log with the necessary information before starting the extraction. Prerinse all glassware three times with methylene chloride.

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7.1 Decant and discard any water layer on a sediment sample. Mix with a stainless steel spatula to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, and rocks, and note actions taken in the appropriate extraction logbook.

7.2 The following steps should be performed rapidly to avoid loss of the more volatile extractable. Weigh out a 30.0 ± 0.05 g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.1 g in appropriate extraction logbook. Add between 30 g and 60 g of anhydrous powdered sodium sulfate as required for producing a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.3 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare a method blank, weigh out one 30.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Add 60 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.

7.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30.0 ± 0.05 g portion of purified sand in a labeled 400 mL beaker. Add 30 g sodium sulfate and mix well. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30.0 ± 0.05 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.1 g in appropriate extraction logbook. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis.

7.6 Record all weights to one decimal place in the extraction logbook.

7.7 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the appropriate base/neutral and acid surrogate spiking solution listed below using the pre-rinsed 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution.

7.6.1 If the request is for SVOA or SVOA Appendix IX, use the SVOA surrogate solution (sect. 5.4).

7.6.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5).

7.6.3 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.

7.7 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 the matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse the syringe with solvent when spiking is completed.

7.7.1 If the request is for SVOA, add 1 mL of SVOA Spiking Solution (sect 5.6).

7.7.2 If the request is for SIM, add 1 mL SIM Spiking solution (sect 5.8).

7.7.3 If the request is for SVOA and SIM, add 1mL of SVOA Spiking Solution and 1 mL SIM Spiking solution (sect 5.6 and 5.8).

7.7.4 If the request is for SVOA Appendix IX, add 1mL of SVOA Spiking Solution and 1 mL of SVOA Appendix IX Spiking solution (sect 5.6 and 5.7).

7.8 To assure optimum operation and maximum energy output, the sonicators <u>must</u> 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.

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. Position the beaker in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4 inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.

7.12 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 the flask, funnel and filter with methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask and Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered.

7.14 Repeat the extraction two more times (sec 7.11 – 7.14) using 100 mL portions of 1:1 methylene chloride: acetone. Before each extraction, make certain that the sodium sulfate is still free-flowing and not a consolidated mass. As required, break up large lumps with the spatula. Decant the extraction solvent into the Buchner funnel after each sonication. On the final sonication, pour the entire sample contents into the Buchner funnel and rinse thoroughly with methylene chloride to complete the quantitative transfer of the extract. Use the vacuum pump to pull all the extract into the flask

CONCENTRATION OF LOW LEVEL EXTRACTS

7.15 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 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.

7.16 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.

7.17 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.

7.18 If samples are not to be GPC'd, follow Steps 7.19 through 7.24 to concentrate extracts to final volume of 1 mL. Otherwise proceed to GPC cleanup procedure as described in the current revision of Katahdin SOP CA-513.

7.19 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.

7.20 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-extraction must occur immediately.** Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.21 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.

7.22 When the apparent volume reaches slightly less than 1 mL, remove the concentrator tube and allow it to cool.

7.23 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.

7.24 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extraction logbook the box number and "tray location" of the individual extract vials.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (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 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.

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10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3550B, USEPA SW-846, Third Edition, Update III, December 1996

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TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-512-05	METHOD 3550, current revision
Apparatus/Materials	1) short stem funnels	1) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) extract dried using Na_2SO_4 in short stem funnels 2) place sonicator horns ½ way between the surface of the solvent and the sediment layer 3) no apparatus height specification for concentration on water bath 4) water bath at 75-85 deg C 5) sample removed from water bath when volume reaches ~6 mL	1) extract dried using Na_2SO_4 in drying columns 2) place sonicator horns ½ inch below the solvent surface but above sediment layer 3) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4) water bath at 80-90 deg C 5) sample removed from water bath when volume reaches 1-2 mL
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC - MDL		

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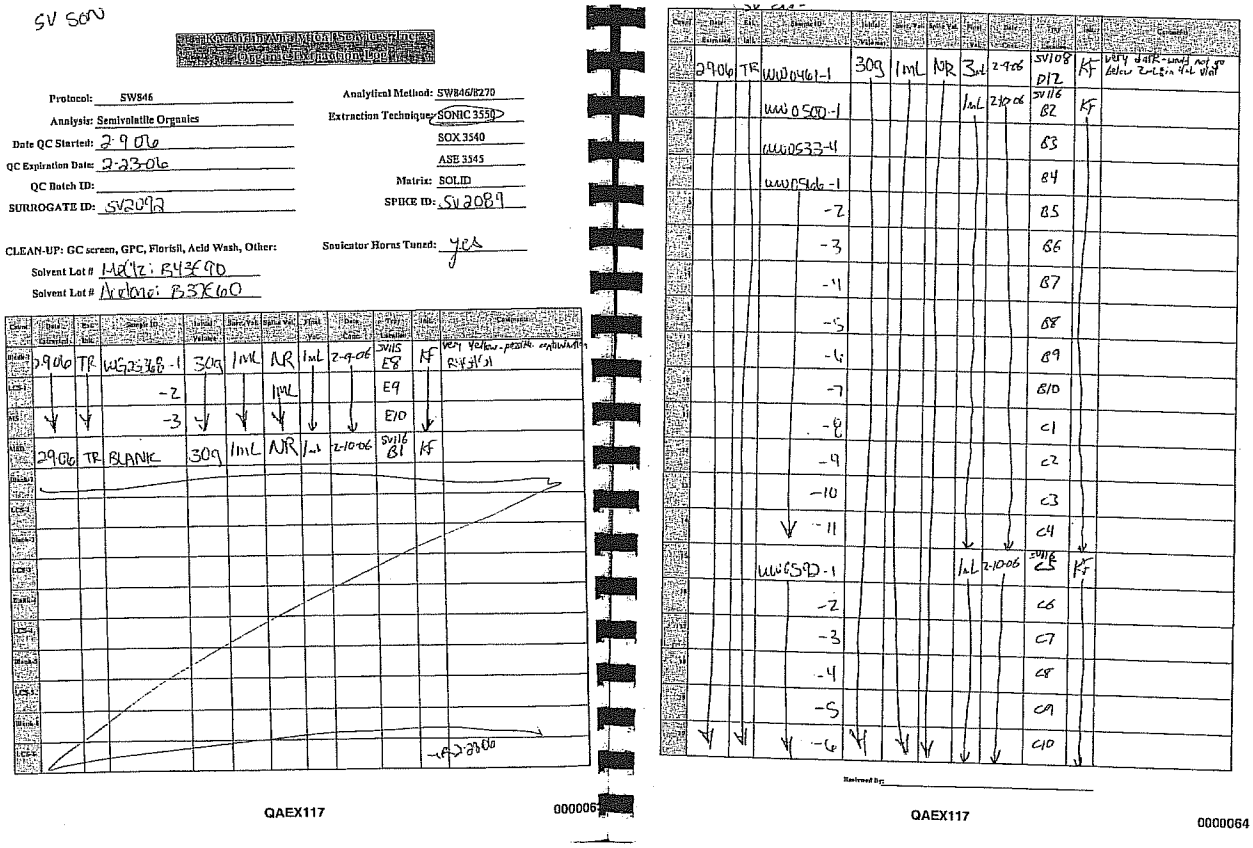
FIGURE 1

EXAMPLE OF LOGBOOK PAGE


SV SON

Protocol: SW846
Analysis: Semivolatile Organics
Date QC Started: 2-9-06
QC Expiration Date: 2-23-06
QC Batch ID:
SURROGATE ID: SV2092
Analytical Method: SW846/8270
Extraction Technique: SONIC 3550
SOX 3540
ASE 3545
Matrix: SOLID
SPIKE ID: SV2089
CLEAN-UP: GC screen, GPC, Florisil, Acid Wash, Other:
Sonicator Horns Tuned: yes
Solvent Lot # Hexane: B43E90
Solvent Lot # Acetone: B35E60

Date Extracted	Tech. Initials	Sample ID	Initial Volume	Dry Wt.	Spike Vol.	Final Volume	Date Analyzed	Instrument	Analyst Initials	Comments
2906	TR	WS23368-1	30g	1mL	NR	1mL	2-9-06	SV115 E8	KF	very yellow-peaks explosion? Rinsed
		-2			1mL			E9		
		-3						E10		
2906	TR	BLANK	30g	1mL	NR	1mL	2-10-06	SV116 B1	KF	
Blank										
LCS										
Blank										
LCS										
Blank										
LCS										
Blank										
LCS										
Blank										
LCS										
Blank										
LCS										
Blank										
LCS										
Blank										
LCS										

-KF 2-23-06

QAEX117 0000063

Date Extracted	Tech. Initials	Sample ID	Initial Volume	Dry Wt.	Spike Vol.	Final Volume	Date Analyzed	Instrument	Analyst Initials	Comments
2906	TR	WW0461-1	30g	1mL	NR	3mL	2-9-06	SV108 D12	KF	very dark - could not go below 2mL in final vial
		WW0500-1				1mL	2-10-06	SV116 B2	KF	
		WW0533-1						B3		
		WW0546-1						B4		
		-2						B5		
		-3						B6		
		-4						B7		
		-5						B8		
		-6						B9		
		-7						B10		
		-8						C1		
		-9						C2		
		-10						C3		
		-11						C4		
		WW659D-1				1mL	2-10-06	SV116 C5	KF	
		-2						C6		
		-3						C7		
		-4						C8		
		-5						C9		
		-6						C10		

Reviewed By:

QAEX117 0000064

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	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 SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

TITLE: 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**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
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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Table 2 Summary of Method Modifications
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Figure 2 Preparation of Matrix Spikes, LCSs, and Spiking Solutions: Method 3010
Figure 3 Element Concentrations in Matrix Spikes, LCSs, and Spiking Solutions: Method 3010

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

QA Officer: Deborah J. Nadeau Date: 1.24.01

General Manager: Duncan F. Lufkin Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3050B	Format changes, added pollution prevention, added MSD, added spiking instruction tables.	DN	1.24.01	1/24/01
02 3050B	Removed all references/procedures devoted to GFAA. Added use of digestates for ICP-MS analysis. Revised standard solution names + concs. in Tables 3 + 4 to reflect current practice.	DN	8.29.02	8.29.02

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.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

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.**

Recipient: _____ Date: _____

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

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Table 2 Summary of Method Modifications – Method 3050
Table 3 Preparation of Matrix Spikes and Spiking Solutions
Table 4 Element Concentrations in ICP-AES Matrix Spikes and Their Component Spiking Solutions
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

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**
.

Recipient: _____Date:_____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-627-02**, titled **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

Recipient: _____Date:_____

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of sub-ppb (ug/L) concentrations of a large number of elements in water samples and in waste extracts or digests. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are required.

ICP-MS has been applied to the determination of over 60 elements in various matrices. Analytes for which EPA has demonstrated the acceptability Method 6020 in a multi-laboratory study on solid wastes are listed as "analytes" in Table 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

TITLE: pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.

Prepared By: Wet Chemistry Date: 8/96

Approved By:

Group Supervisor: Keith Tangney Date: 021301

Operations Manager: John C. Burton Date: 2/13/01

QA Officer: Deborah J. Nadeau Date: 2·13·01

General Manager: Dennis P. Lufkin Date: 2/12/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03 9045C	Format changes, added pollution prevention, database and operation of Accumet pH meter and calibration.	DN	2·13·01	2·13·01
04 9045C	Addition to Scope and Application to include reference for 9040B use when aqueous phase is >20%	DN	8·27·02	8·27·02
05 9045C	added KIMS Minor changes throughout added wording to sect. 6 New fig. 1 and 2	LAD	120104	120104
06 9045C	Added SW-846 reference. Minor formatting changes throughout.	LAD	03/07	03/07

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

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

I acknowledge receipt of copy _____ of document **CA-709-05**, titled **pH Concentration Measurements in Soil Matrices - SW846 Method 9045**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **CA-709-05**, titled **pH Concentration Measurements in Soil Matrices - SW846 Method 9045**.

Recipient: _____ Date: _____

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and techniques followed by Katahdin Analytical Services, Inc. personnel to determine the pH of soils and waste samples in accordance with SW846 method 9045 (current promulgated revision). Method 9045 is an electrometric procedure for measuring pH in soils and waste samples. Wastes may be solids, sludges, or non-aqueous liquids. If water is present, it must constitute less than 20% of the total volume of the sample. If the aqueous phase is greater than 20%, pH determination should be performed in accordance with EPA method 9040 (current promulgated revision). Refer to the current revision of Katahdin SOP CA-708, pH Concentration Measurements in Aqueous Samples.

The procedures in this SOP are applicable to all non-CLP pH measurements performed for all soil matrices analyzed in the laboratory.

1.1 Definitions

pH - A measure of the acidity or alkalinity of a solution, defined as -log [H^+].

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of pH in solids by EPA Method 9045. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the determination of pH concentration measurements in solid matrices to read and understand this SOP, to adhere to the procedures outlined, and to properly document theirdata in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for pH data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method

has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety datasheets 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 preparation of samples must be disposed of in accordance with the Katahdin Chemical Hygiene Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

A representative aliquot of sample, measured in grams, is mixed with an equivalent volume of laboratory reagent grade water, measured in mL. The solution is allowed to settle, and the pH of the standing water (decanted) is determined electrometrically.

3.0 INTERFERENCES

3.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode. Strong acid solutions, with a true pH of <1, may give incorrectly high pH measurements.

3.2 Temperature fluctuations will cause measurement errors.

3.3 Errors will occur when the electrodes become coated with an oily material. See section 7.18 for special cleaning instructions.

4.0 APPARATUS AND MATERIALS

4.1 pH meter, Accumet Model 20 or equivalent with Automatic Temperature Compensation (ATC)

4.2 Glass beakers, 25 mL and 400 mL

4.3 25 mL dose cups

4.4 Teflon coated stir-bars

4.5 Stir-bar retriever

4.6 Magnetic stirplate

4.7 Shaker, 12 place

4.8 Analytical balance, capable of weighing to 0.1 g

5.0 REAGENTS

5.1 Buffer solutions (pH 4.0, 6.0, 7.0, 8.0, 10.0, 12.0)

5.2 Laboratory reagent grade water (Lab Water)

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected in soil jars and stored at 4 (±2) ^{o}C until analyses. Since there are no published holding times for this method, sample analysis should be performed as soon as possible.

7.0 PROCEDURES

SAMPLE PREPARATION

7.1 Mix samples thoroughly. Discard any foreign objects such as sticks, leaves and rocks. Decant any standing liquid. Using the balance, weigh out 20.0 g of sample into a 400 mL glass beaker. Record weight in pH logbook (Figure 1).

7.2 Add 20 mL of Laboratory reagent grade water to the sample. Cover the top of the beaker with parafilm.

7.3 Place the sample on the shaker and allow it to shake, at medium speed, for one hour.

7.4 After one hour, remove the sample from the shaker and allow it to settle for one hour.

7.5 After one hour, decant the standing liquid into a 25 mL beaker. If no standing liquid is present, add sufficient laboratory reagent grade water to result in standing water, cover with parafilm, and repeat steps 7.3 through 7.5.

7.6 Record total volume of laboratory reagent grade water added to sample in pH logbook. If volume of laboratory reagent grade water (in mL) added to sample exceeds the initial gram weight of the sample, flag sample data in pH logbook with reason for addition of excess laboratory reagent grade water (eg. minimum volume of water required in order to cover pH probe).

CALIBRATION OF PH METER

7.7 NORMAL RANGE CALIBRATION (pH range 3.5 - 10.5)

7.7.1 Meter should be calibrated daily. As described in the following steps, conduct a two-point calibration with pH buffers 4 and 10. Perform a calibration check using pH 7 buffer. The source/lot number of each solution at the time of analysis must be recorded in the logbook (Figure 1).

7.7.2 Dispose of buffer solutions used the previous day (see Waste Disposal, section 1.4). Put about 20 mL of the appropriate buffer solution into new dose cups. Place a tiny stir bar in each new cup.

7.7.3 Rinse probe (i.e., electrode) with laboratory DI laboratory reagent grade water. Gently blot dry with kimwipe.

7.7.4 Place pH 4 buffer on stir plate. Turn stir plate on so that the stir bar spins without creating a vortex. Push *1* key (meaning add a standard). Then push *4* (calibrate). Record the value in the pH logbook.

7.7.5 Remove pH 4 buffer. Rinse probe. Blot dry.

7.7.6 Repeat step 7.7.4 with the pH 10 buffer. Record the value in the pH logbook. Remove pH 10 buffer. Rinse and dry probe.

7.7.7 With the pH 7 buffer, repeat step 7.7.4, but DO NOT press any keys as this reading is a calibration check. Record reading in pH logbook. Results must be within 0.05 pH units of the true value for analysis to proceed.

NOTE: If buffer readings are not within 0.05 pH units of expected values (4.00, 7.00 and 10.00) the electrode may need cleaning. Rerun and enter in pH lab notebook that meter was recalibrated with the pH 4 and 10 buffers, with the pH 7 buffer used as a calibration check. Also record any maintenance performed.

7.8 LOW RANGE CALIBRATION

7.8.1 If pH of a sample as is less than 3.5, the instrument must be recalibrated using buffers that bracket the expected pH of the sample, as described below. The source/lot number and temperature of each solution at the time of analysis must be recorded in the logbook (Figure 1).

7.8.2 Rinse probe (i.e., electrode) with laboratory reagent grade water.

7.8.3 . Gently blot dry with kimwipe.

7.8.4 Place pH 2 buffer on stir plate. Turn stir plate on so that the stir bar spins without creating a vortex. Push *1* key (meaning add a standard). Then push *4* (calibrate). Record the value in the pH logbook.

7.8.5 Remove pH 2 buffer. Rinse probe. Blot dry.

7.8.6 Repeat step 7.8.3 with the pH 7 buffer. Record the value in the pH logbook. Remove pH 7 buffer. Rinse and dry probe.

7.8.7 With the pH 4 buffer, repeat step 7.8.3, but DO NOT press any keys as this reading is a calibration check. Record reading in pH logbook. Results must be within 0.05 pH units of the true value for analysis to proceed.

NOTE: If buffer readings are not within 0.05 pH units of expected values (2.00, 4.00 and 7.00) the electrode may need cleaning. Rerun and enter in pH lab notebook that meter was recalibrated with the pH 2 and 7 buffers, with the pH 4 buffer used as a calibration check. Also record any maintenance performed.

7.9 HIGH RANGE CALIBRATION

7.9.1 If pH of a sample as determined in Sections 7.10 through 7.17 is greater than 10.5, the instrument must be recalibrated using buffers that bracket the expected pH of the sample, as described below. The source/lot number and temperature of each solution at the time of analysis must be recorded in the logbook (Figure 1).

7.9.2 Rinse probe (i.e., electrode) with laboratory reagent grade water. Gently blot dry with kimwipe.

7.9.3 Place pH 7 buffer on a stirplate. Turn stir plate on so that the stir bar spins without creating a vortex. Push *1* key (meaning add a standard). Then push *4* (calibrate). Record the value in the pH logbook.

7.9.4 Remove pH 7 buffer. Rinse probe. Blot dry.

7.9.5 Repeat step 7.9.3 with the pH 12 buffer. Record the value in the pH logbook. Remove pH 12 buffer. Rinse and dry probe.

7.9.6 With the pH 10 buffer, repeat step 7.9.3, but DO NOT press any keys as this reading is a calibration check. Record reading in pH logbook. Results must be within 0.05 pH units of the true value for analysis to proceed.

NOTE: If buffer readings are not within 0.05 pH units of expected values (7.00, 10.00 and 12.00) the electrode may need cleaning. Rerun and enter in pH lab notebook that meter was recalibrated with the pH 7 and 12 buffers, with the pH 10 buffer used as a calibration check. Also record any maintenance performed.

ANALYSIS OF SAMPLES

7.10 Sample analysis may proceed once the meter has been calibrated for the day with two buffers that bracket the expected pH of the sample, and after checking the calibration with a mid-range solution between the two buffers used for calibration of the meter (refer to sections 7.7, 7.8, and/or 7.9 as applicable).

7.11 For the range being used, rerun the appropriate calibration check point as the laboratory control sample (LCS) for the analytical batch. An LCS is required at the beginning of every batch of twenty or fewer samples.

7.12 Record date, time and initials for this analytical session.

7.13 The decanted samples should be equilibrated to room temperature prior to analysis (i.e., at the same temperature as the calibration buffers, ±2 °C). A more accurate pH

reading will be achieved when the buffers and the samples are at the same temperature. However, the Accumet® pH meter is equipped with automatic temperature compensation (ATC) for when samples and buffers are not at the same temperature. Refer to the Accumet® Model 20 pH/Conductivity Meter operating Instructions, #300143.3 (Revision C) for information on the ATC probe.

7.14 Pour about 25 ml of the supernatant into a clean dose cup. Place a tiny stir bar in cup. Place on stir plate, turn on stir plate and immerse probes.

7.15 When meter locks, record value displayed.

7.16 Rinse probe with laboratory reagent grade water and blot dry between samples.

7.17 Place probe in pH 4 buffer solution to store until next analysis.

CLEANING PROCEDURE

7.18 Only if an electrode becomes coated with an oily material that will not rinse free, the electrode can either (refer to instrument manual):

- be cleaned with an ultrasonic bath, or
- be washed with detergent, rinsed several times with laboratory reagent grade water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with laboratory reagent grade water.

REPORTING OF RESULTS

7.19 All pH measurements less than 10.0 are to be reported using two significant figures.

Examples: 2.46 = 2.5
6.32 = 6.3
9.94 = 9.9

7.20 All pH measurements which are at or greater than or round up to 10.0 are to be reported to three significant figures.

Examples: 9.95 = 10.0
12.25 = 12.3
13.76 = 13.8
11.95 = 12.0

7.21 When a sample duplicate is analyzed, both the original result and duplicate result are recorded in the pH logbook; however, the original sample result is to be reported to the client.

7.22 After completion of each test, the logbook must be signed and dated by the person performing the test. All unused lines are to be "z-ed" out and initialed and dated.

7.23 The sample data results, with any appropriate notations, are entered into KIMS by the analyst. A batch sheet is generated (Figure 2). Raw data and batch sheets are reviewed for completeness and accuracy by the Inorganic Department Manager or other qualified designee.

7.24 All batch sheets and copies of the raw logbook data are filed with the Inorganic Department Manager for approximately 3 months, for reference by analysts. Prior data are archived.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, remaining sample volume and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

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

8.1 One sample duplicate is to be analyzed per batch or every 10 sample analyses.

8.1.1 Acceptance criteria for duplicates is a difference of less than or equal to 20% relative percent difference between sample and duplicate results.

8.1.2. If criterion is not met, check calibration and reanalyze sample in duplicate.

8.2 One Laboratory Control Sample (LCS) is to be analyzed per batch or every 20 samples.

8.2.1 The LCS must be within 90-110% recovery for analysis to proceed.

8.2.2 If criteria are not met, recalibrate.

9.0 METHOD PERFORMANCE

Refer to method 9045.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update III, December 1996, Method 9045C.

LIST OF TABLES AND FIGURES

Table 1 QC Requirements
Table 2 Summary of Method Modifications
Figure 1 Example of pH - Soils Logbook Page
Figure 2 Example of Batch Sheet for pH

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

TABLE 1

QC REQUIREMENTS

Analytical Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW9045	PH (soil)	2-point calibration with pH buffers with a midrange cal. check	Once per day	± 0.05 pH units for every buffer	If calibration is not achieved, check meter, buffer solutions, and probe; replace if necessary; repeat calibration
		LCS	One per batch of twenty or fewer samples	90-110%R	Correct problem, recalibrate
		Sample duplicate	One sample duplicate per every ten field samples	RPD ≤20	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD is still unacceptable, report original result with notation or narration.

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES- SW 846 METHOD 9045.**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-709-06	METHOD SW846 9045, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures	1) Shake, at medium speed, for one hour. 2) Add more liquid after shaking and settling if there is no standing liquid left. 3) All buffers and samples are analyzed at room temperature. PH meter is equipped with automatic temperature compensation.	1) Continuously stir the suspension for five minutes. 2) No guidance for samples with no standing liquid left. 3) Report both pH and temperature at the time of analysis.
QC – Spikes		
QC – LCS		
QC - Accuracy/Precision		

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

FIGURE 1

EXAMPLE OF pH LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES, INC.

CORROSIVITY pH
pH SOIL

SW 846 9045A SOW OLM01.8

CALIBRATION STDS:	CALIBRATED TO:	LOT NO:
pH 7.00	7.00	Sol 2217
pH 4.00	4.00	Sol 2014
pH 5.00		
pH 10.00	10.01	Sol 2216
pH 8.00		
pH 12.00	12.00	Sol 2176
WG 35229 R58891		

LAB SAMPLE ID	ANALYSIS TIME	SAMPLE VOL (mL)	SAMPLE WEIGHT(g)	pH	REPORTED pH
LCS WG 35229-1	1150	20.0	20.000	7.00	7.0
WW6750-1B	1550	20.0	20.333	9.27	9.3
DUP WG 35229-2	1552	20.0	20.352	9.37	9.4
L -2B	1554	20.0	20.418	10.76	10.8
WW6821-1B	1556	20.0	20.316	10.04	10.0
L -2B	1558	2000	20.178	7.40	7.4

ANALYST: Date 12.22.06
DATE: 12-22-06
CHECKED BY: DATE: 12/28/06

TITLE: **pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

FIGURE 2

EXAMPLE OF BATCH SHEET FOR pH

WET CHEMISTRY BATCH REPORT
Dec 27 2006, 04:11 pm
Batch: WG35229

Parameter: pH(Soil)

Date Analyzed: 22-DEC-06

Analyst Initials: JF

Prep Date: 22-DEC-06

Prep Method: SW846 9045C

Prep Chemist: JF

Sample	Samp Type	Method	Initial Amt.	Final Amt.	Rpt. DF	Result	Rpt Result	TS(%)	PQL	MDL	Adj PQL	RPD	%Rec
WG35229-1	LCS	SW846 9045C	20g	20mL	1	7	7.0 pH	NA	.1	.1	.1		100
WG35229-2	DUP	SW846 9045C	20.352g	20mL	1	9.37	9.4 pH	NA	.1	.1	.1	1	
WW6780-1	SAMP	SW846 9045C	20.333g	20mL	1	9.27	9.3 pH	NA	.1	.1	.1		
WW6780-2	SAMP	SW846 9045C	20.418g	20mL	1	10.76	11. pH	NA	.1	.1	.1		
WW6821-1	SAMP	SW846 9045C	20.316g	20mL	1	10.04	10. pH	NA	.1	.1	.1		
WW6821-2	SAMP	SW846 9045C	20.178g	20mL	1	7.4	7.4 pH	NA	.1	.1	.1		

Comments:

WG35229-1 WW6780-1
WG35229-2 WW6780-1

Entered by: _____ Date: 12-27-06 Accepted by: _____ Date: 12/28/06

TITLE: **CATION EXCHANGE CAPACITY of SOILS USING USEPA METHOD 9081**

Prepared By: George Brewer Date: 03/16/05

Approved By:

Department Manager: George Brewer Date: 03/16/05

Operations Manager: Deborah J. Nadeau Date: 3·16·05

QA Officer: Leslie Dimond Date: 03-16-05

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date

TITLE: CATION EXCHANGE CAPACITY OF SOILS USING USEPA METHOD 9081

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-737-00**, titled **CATION EXCHANGE CAPACITY OF SOILS USING USEPA METHOD 9081**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-737-00**, titled **CATION EXCHANGE CAPACITY OF SOILS USING USEPA METHOD 9081**.

Recipient: _____ Date: _____

TITLE: CATION EXCHANGE CAPACITY OF SOILS USING USEPA METHOD 9081

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure is to describe the procedures used by Katahdin Analytical Services, Inc. technical personnel for the determination of the cation exchange capacity of soils using USEPA Method 9081.

Method 9081 is applicable to most soils, including calcareous and noncalcareous soils.

a. Definitions:

CEC = cation exchange capacity

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of cation exchange capacity of soils by Method 9081. 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 analysis of cation exchange capacity by Method 9081 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 Management Plan and follow appropriate procedures such as wearing safety glasses and gloves when working with chemicals or near an instrument. Food or drink are not allowed in the laboratory. Each

analyst should possess a respirator and be trained in its proper use and should know the location and use of all safety equipment.

1.4 Pollution Prevention / Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Spent soil extracts and reagents should be disposed of in the satellite waste container for corrosive wastes (labeled "Waste Stream A") that is located in the Metals Sample Prep lab. Spent isopropanol should be disposed in the satellite waste container for flammable wastes (labeled "Waste Stream O"). 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

A soil sample is mixed with an excess of sodium acetate solution, resulting in an exchange of the added sodium cations for the matrix cations. Subsequently, the sample is washed with isopropyl alcohol. An ammonium acetate solution is then added which replaces the adsorbed sodium with ammonium. The concentration of displaced sodium is then determined by inductively-coupled plasma atomic emission spectroscopy (USEPA Method 6010).

3.0 INTERFERENCES

Refer to the SOP for ICP analysis for potential interferences that may occur during analysis.

4.0 APPARATUS AND MATERIALS

4.1 Analytical balance capable of weighing to 0.01 g

4.2 50-mL disposable graduated polyethylene centrifuge tubes with screw caps

4.3 Disposable polystyrene specimen cups

4.4 Mechanical shaker – the separatory funnel shaker in the Organics Extraction laboratory is suitable

4.5 Centrifuge with rotor allowing use with 50-mL disposable centrifuge tubes

4.6 50-mL graduated cylinder, polyethylene or polypropylene

4.7 100 mL capacity graduated polyethylene or polypropylene containers, e.g. 4 oz. Corning Snap-Seal containers

5.0 REAGENTS

5.1 Laboratory grade reagent water,

5.2 Isopropyl alcohol, 99% purity

5.3 Sodium acetate (NaOAc), 1.0 N: Dissolve 136 g of sodium acetate trihydrate ($NaC_2H_2O_2 \cdot 3H_2O$) in laboratory grade reagent water and dilute to 1000 mL. The pH of this solution pH 8.2.

a. Ammonium acetate (NH_4OAc), 1 N: Dilute 114 mL of glacial acetic acid (99.5%) with laboratory grade reagent water to a volume of approximately 1 L. Then add 138 mL of concentrated ammonium hydroxide (NH_4OH) and add laboratory grade reagent water to obtain a volume of about 1980 mL. Check the pH of the resulting solution and add more NH_4OH, as needed, to obtain a pH of 7. Dilute the solution to a volume of 2 L with laboratory grade reagent water.

b. Cation exchange resin of known cation exchange capacity, such as Dowex HCR-W2, for use as a reference material.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples should be collected in glass or polyethylene containers and stored at 4 (±2) ° C. until analysis.

The USEPA has not specified a holding time for this analysis.

7.0 PROCEDURE

7.1 Place a labeled centrifuge tube in a disposable specimen cup to hold it upright. Place the specimen cup on the balance and tare the balance. Weigh 4 g (wet weight) of a medium- or fine-textured soil or 6 g (wet weight) of a coarse textured soil into the centrifuge tube. Record the sample weight to the nearest 0.01 g in the CEC Sample Preparation Logbook (Figure 1).

a. Using the graduated cylinder, add 33 mL of 1.0 N NaOAc solution, cap the tube, and shake on the mechanical shaker for 5 minutes. (Note: Samples are processed on the separatory funnel shaker by attaching 4 or 5 tubes together with rubber bands, and attaching the group of tubes to the shaker.) Remove from the mechanical shaker and centrifuge until the liquid is clear (approx. 5 minutes).

b. Decant the liquid and dispose of it the satellite waste container for corrosive wastes (Waste Stream "A").

c. Repeat Steps 7.2 and 7.3 three more times.

d. Using the graduated cylinder, add 33 mL of 99% isopropyl alcohol, cap the tube, and shake on the mechanical shaker for 5 minutes. Remove from the mechanical shaker and centrifuge until the liquid is clear (about 5 minutes).

e. Decant the liquid and dispose of it in the satellite waste container for flammable waste (Waste Stream "O").

f. Repeat Steps 7.5 and 7.6 two more times.

g. Using the graduated cylinder, add 33 mL of NH_4OAc solution, cap the tube, and shake on mechanical shaker for 5 minutes. Remove from the mechanical shaker and centrifuge until liquid is clear (about 5 minutes).

h. Decant the liquid into a Corning Snap-Seal container.

i. Repeat Steps 7.8 and 7.9 two more times, collecting all of the supernatant liquid from the sample in the same Snap-Seal container.

j. Dilute the liquid in the Snap-Seal container to the 100 mL graduation. Cap the container and invert several times to mix.

k. Analyze the extract obtained from each sample for sodium by inductively-coupled plasma atomic emission spectroscopy (USEPA Method 6010). Refer to the current revision of Katahdin Analytical Services SOP CA-608 for detailed instructions.

CALCULATION OF FINAL RESULTS

I. Calculate the cation exchange capacity (CEC) of each soil sample as follows:

$$\text{CEC (meq/100 g dry wt.)} = \frac{Na \times FV \times 100}{W \times 22.99 \times TS}$$

Where:

Na = Measured sodium concentration of extract (mg/L)
FV = Final volume of extract (L)
W = Initial wet weight of sample (g)
TS = Decimal percent solid content of sample, measured on a separate aliquot
22.99 = Atomic weight of sodium (22.99 mg sodium = 1 meq sodium)

Report cation exchange capacity down to the Practical Quantitation Limit (PQL) for the analysis. The reporting limit for cation exchange capacity is based on the PQL for sodium by ICP analysis, 1.0 mg/L. A sodium concentration of 1.0 mg/L (in the extract) calculates to a cation exchange capacity of 0.11 meq/100 g dry weight of sample, assuming that 4.0 g of sample (100% solids) are extracted with a final extract volume of 100 mL. A lower sample weight, a lower sample solids content, or a higher final extract volume will result in a higher sample-specific PQL for CEC.

7.14 Sample preparation information, measured sample concentrations, and quality control data are entered manually into the Katahdin Information Management System for calculation and reporting. Refer to the current revision of Katahdin SOP CA-762 ("Wet Chemistry Data Entry and Review Using Katahdin Information Management System") for further information.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 9081 requires the laboratory to perform specific quality control checks to assess laboratory and method performance. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are listed in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts, and do not cover all possible situations. If any of the QC checks are outside the acceptance criteria listed in Table 1, all associated samples must be evaluated against all of the QC. In some cases sample results that are associated with failing QC samples may be reported with narration, and in other cases reanalysis may be required. It may be necessary to consult the client regarding project-specific Data Quality Objectives to determine the appropriate corrective action for a QC failure. The Department Manager, Operations Manager, and/or Quality Assurance Officer should be consulted for guidance in these matters as required.

8.1 A Preparation Blank (PB), consisting of 4.0 g of laboratory grade reagent water carried through the same process as associated samples, is prepared with each batch of twenty or fewer samples. The measured cation exchange capacity of the PB must be less than the Practical Quantitation Level (PQL) for the analysis. If the PB fails, results may not be reported from the preparation batch, and all associated samples must be reextracted, with the following exception. If the result for a PB is greater than or equal to the PQL, associated sample results that are greater than or equal to ten times the measured PB concentration may be reported.

8.2 A Laboratory Control Sample (LCS), consisting of cation exchange resin of known CEC carried through the same process as associated samples, is prepare with each batch of twenty or fewer samples. The measured CEC of the LCS must fall within 80% to 120% of the known value. If the LCS fails, results may not be reported from the preparation batch, and all associated samples must be reextracted.

8.3 One field sample in ten must be analyzed in duplicate. The relative percent difference (RPD) between duplicates must be less than or equal to 20%. If the duplicate RPD exceeds this control limit, the CEC result of the associated sample should be flagged on the Report of Analysis.

1) METHOD PERFORMANCE

No data provided.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA, SW-846, Third Edition and updates, Method 9081.

Trace Metals Analysis By ICP-AES Using EPA Method 6010, Katahdin SOP CA-608, current revision.

LIST OF TABLES AND FIGURES

Table 1 QC Requirements
Table 2 Summary of Method Modifications
Figure 1 Example of Cation Exchange Capacity Preparation Logbook Page

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Cation Exchange Capacity (CEC) of soils / USEPA Method 9081	Preparation Blank (PB)	One per prep batch of 20 or fewer samples	CEC < PQL	1) Investigate source of contamination 2) If associated sample CEC < 10 times blank concentration, reprep and reanalyze sample 3) If associated sample CEC ≥ 10 times blank concentration, report measured sample CEC
	Laboratory Control Sample (LCS)	One per prep batch of 20 or fewer samples	80% - 120% recovery	Reprep and reanalyze associated samples
	Sample Duplicate	One sample duplicate per ten or fewer samples	RPD ≤20	Narrate or flag associated sample result
	MDL study	Once per year	Ideally PQL = at least 3 times the MDL.	Repeat MDL or raise PQL

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-737-00	USEPA Method 9081
Apparatus/Materials	Extracts brought to final volume in disposable graduated plastic container	Extracts brought to final volume in volumetric flasks

TITLE: CATION EXCHANGE CAPACITY OF SOILS USING USEPA METHOD 9081

FIGURE 1

EXAMPLE OF CEC SAMPLE PREPRATION LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES, INC.
Cation Exchange Capacity of Soils - USEPA Method 9081

Reagent	Lot No.	Manufacturer/ Catalog No.	CEC True Value (meq/100 g)
1.0 N Sodium Acetate	MR 1234		
1.0 N Ammonium Acetate	MR 1235		
Isopropyl Alcohol	SWL1176	Acros	
Cation Exchange Resin	32 x 126	Powex HCR-W2	≈50.

Katahdin Sample No.	Sample Wet Wt. (g)	CE Resin Added (g)	Extract Final Vol. (mL)
Prep Blank	4.00		100
LCS	4.00	1.03	100
WV9999-1	4.12		100
-1 Dup	4.03		100
-2	4.31		100
-3	4.16		100
-4	4.22		100
-5	4.04		100

01/19/05

Sample Preparation Date:	Prepared By: 01/19/05

TITLE: **DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD**

Prepared By: Greg Lull Date: 7/2002

Approved By:

Group Supervisor: Keith Tanguay Date: 091102

Lab Operations Mgr: Joel C. Burton Date: 9/11/02

QA Officer: Deborah J. Nadeau Date: 9.11.02

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Sect. 1-clarified CCB, Sect. 2-clarified phosphoric acid conc. Sect. 7- reworded software and instr. start up, added TS to calc., removed steps not currently in practice. Sect. 8 -updated LCS and CCV information. Updated Table 1	LAD	01/07	01/07

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD

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

I acknowledge receipt of copy ___ of document SOP **CA-741-01**, titled **Determination of Total Organic Carbon in Solids Using the EPA Region II Lloyd Kahn Method.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP **CA-741-01**, titled **Determination of Total Organic Carbon in Solids Using the EPA Region II Lloyd Kahn Method.**

Recipient: _____ Date: _____

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD

1.0 SCOPE AND APPLICATION

This SOP describes the procedures used by Katahdin Analytical Services, Inc. technical personnel to determine Total Organic Carbon (TOC) in solids in accordance with EPA Region II Lloyd Kahn method.

This method is applicable to sediment, sludges, and soil samples. The detection limit for this method is 100 µg C and a method PQL of 400 ug/g.

1.1 Definitions/Acronyms

TC – Total carbon
IC – Inorganic Carbon
TOC – Total Organic Carbon

Method Blank - A deionized water sample that is carried through the entire analytical procedure in the same manner as a sample.

LCS/ICV - Laboratory Control Sample/ Initial Calibration Verification. One LCS/ICV per batch is prepared from a separate source from the CCV and calibration curve standards. LCS/ICV verifies the calibration curve.

CCV - Continuing Calibration Verification. The CCV is made from the same source as the calibration. One CCV is run every ten samples.

CCB - Continuing Calibration Blank. The CCB is an empty sample boat with no reagents added. One CCB is run every ten samples.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of TOC in solids by the Lloyd Kahn Method. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of TOC in solids by the Lloyd Kahn method to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD

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.

Wastes generated during the preparation of samples must be disposed of in adherence with the Katahdin Hazardous Waste Management 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

Total Carbon (TC) is measured utilizing a carbonaceous analyzer with a boat sampling module and 900°C furnace attached. The resulting combustion converts carbon to carbon dioxide (CO_2) in the presence of oxygen. The amount of CO_2 derived from a sample is directly

TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD

proportional to the concentration of carbonaceous material in the sample and is then measured by a non-dispersive infrared detector (NDIR).

To determine **Total Organic Carbon** (TOC), however, carbonate and bicarbonate ions contributing to the TC result must be accounted for. This is achieved by adding 1:1 phosphoric acid to the sample and combusting it at 103° C for 10 minutes to remove any **Inorganic Carbon** (IC) before analyzing the sample. The **Total Carbon** result then equals the **Total Organic Carbon.**

3.0 INTERFERENCES

Volatile organics in the sediment may be lost in the decarbonation step resulting in a low bias.

4.0 APPARATUS AND MATERIALS

4.1 Shimadzu model TOC-Vcph with NDIR.

4.2 SSM-5000A 970°C furnace with boat sampling module.

4.3 Mettler AE 100 balance (accurate to 0.1 mg) or equivalent.

4.4 Ceramic boats.

4.5 Drying oven capable of maintaining 103-105°C

4.6 Oxygen gas

5.0 REAGENTS

5.1 Dextrose Solid (40% Carbon by weight = 400,000 ug/g C)

Calibration Standards:

0.0 mg - Calibration Blank
1.0 mg - 400ugC
5.0 mg - 2000ugC
10.0 mg - 4000ugC
20.0 mg - 8000ugC
60.0 mg - 24,000ugC

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(Upper range limit for TC is 30,000ugC)

5.2 Dextrose Solid (40% Carbon by weight = 400,000 ug/g C) used as LCS run at 8000-12000ugC (20-30mg). Standard must be a source different from CCV source.

5.3 Sodium Carbonate, anhydrous (11.3% Carbon by weight = 113,000 ug/g C)

Calibration Standards:

0.0 mg	- Calibration Blank
3.5 mg	- 400ugC
17.7 mg	- 2000ugC
35.3 mg	- 4000ugC
70.7 mg	- 8000ugC
212.0 mg	-24,000ugC

(Upper range limit for IC is 25,000 ug C)

5.4 1:1 Phosphoric acid / DI water solution

5.5 Sodium Hydrogen Carbonate (14.28% Carbon by weight = 142,857ug/gC) used as LCS run at 4000ugC level (28 mg).

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Collect sediments in a glass jar with Teflon or aluminum foil. Cool and maintain at 4° (±2) C. Analyze within 14 days.

7.0 PROCEDURES

SET UP AND CALIBRATION

7.1. Turn on TOC-Vcph analyzer, SSM-5000A furnace, and oxygen supply and let warm up for one hour or until the temperature readouts show 970° C for the TC and 200° C for the IC.

7.2. Start the TOC-V software program by clicking the TOC-V icon on the desktop and selecting sample table editor. Click the "new" icon followed by "sample run". Choose SSM-5000A from the pull down menu. This activates a sample field spreadsheet in which calibrations, controls, and samples can be inserted and run. Turn on the TOC aqueous and soil instruments and the oxygen. Click the "connect" icon and choose

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"Settings on PC". This will start the gas flows through both units and will activate the NDIR. Wait for the TC furnace to read 970°C before beginning analysis. Also place any sample boats that will be used in the furnace for several minutes to bake off any remaining residue.

7.3. Determine whether TC or IC will be analyzed for the run. A 6 point curve (for either TC or IC) must be run quarterly to verify the calibration. Using a calibrated analytical balance, the standards listed in section 5.1 for TC or in section 5.3 for IC should be used. The instrument must recover ± 10 % of these true values. The calibration may also be updated as necessary as demonstrated by failure of the Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV). An ICV is run at the beginning of every sample sequence and a CCV is run every 10 samples. To verify baseline stability and demonstrate any cross contamination issues, a boat blank or Continuing Calibration Blank (CCB) is run at the beginning of a sample sequence and every ten samples as well. A laboratory control sample (LCS) and duplicate (LCSD) are run at the beginning of a sample sequence and every 20 samples as a secondary source calibration check.

7.4. The value of the calibration checks must fall within the control limits (80-120% recovery). If not, rerun the sample up to two more times. If the calibration check is still out of the acceptable recovery range, recalibrate the instrument and repeat the procedure. If the problem persists, remake the standard and repeat the procedure.

ANALYSIS

7.5. Using a calibrated analytical balance, weigh out 50-200mg of the sample in a tared sample boat. Record the weight in the TOC soil logbook (Figure 1).

7.6. If the sample is being analyzed for TC, the boat is inserted into the TC boat port. After placing the boat in the port and closing the cover, allow the instrument to reestablish the baseline (the atmospheric CO_2 that enters the closed system will create an initial small peak) by waiting 2 minutes or until the "baseline fluctuation" warning is back to "ready".

7.7. Enter the sample information into the spreadsheet by clicking the insert sample icon. Select "tc method soils" as the method and click next. Type in the sample name, followed by next twice, then finish. Click the start icon and enter the file name for the sample run. The instrument will then prompt for the sample weight to be entered. The instrument will prompt to push boat into sampling position.

7.8. If the sample is being analyzed for IC, the boat is inserted into the IC boat port. After placing the boat in the port and closing the cover, allow the instrument to restabilize just as in the TC analysis. Once the "baseline fluctuation" warning is back to a ready

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status, and before inserting the boat, apply between 0.2- 0.5 ml of the 1:2 phosphoric solution using the pump bottle attached to the IC port. Insert the boat immediately and allow the inorganic carbon to evolve and be evaluated.

7.9. To view the sample peak in real time, click view sample window in the menu bar at the top of the screen. This will show the NDIR activity as CO_2 evolves.

7.10. After integration, back the boat out of the furnace to the cooling position and then to the sample preparation position as prompted by the software. When sample run is complete, print sample table (Figure 2) and sample results (Figure 3).

CALCULATIONS AND REPORTING OF RESULTS

7.11. Calculate the TC or IC (whichever is being performed) concentration using the following equation:

$$\frac{\text{Abs C value (instrument reading) in ug}}{\text{Sample Weight (g)}} \times \frac{100}{\%TS} = \text{TC or IC result in ug/g C}$$

7.12. Workgroup samples and get run ID. Enter true values for the LCS and MS and save. Go back to the spreadsheet and enter "LLOYD" in the comments section for the samples you wish to report. Change QC to match workgroup. Data is then exported by selecting the ASCII export option from the file menu. Select "save as" and choose parsefiles on LVSlims. Select TOC and type file name. Click save and wait for data to export, then review data in wetrev.

7.13. A batch sheet is generated (Figure 4). Raw data, calibrations, and batch sheets are reviewed for completeness and accuracy by the Inorganic Department Manager or other qualified designee.

7.14. 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 for retrieval.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

See below and refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed below and in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The

corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

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

8.1 An ICB is analyzed at the beginning of the run and a CCB is analyzed every ten samples thereafter. The ICB and CCBs are boat blanks. The boat should be analyzed empty.

8.2 Analyze an LCS and LCSD (20-30 mg of Dextrose = 8000-12000ugC for TC) / (28 mg Sodium Hydogen Carbonate = 4000 ug C for IC) with each batch of 20 samples. Acceptance criteria is 80-120% of expected value.

8.3 Run a CCV (20-30 mg Dextrose = 8000-12000 ug C for TC) / (35.3 mg Hydrogen Carbonate = 4000 ug C for IC) every 10 samples and at the end of each batch. Acceptance criteria is 80-120% of expected value. Run a CCB or boat blank every 10 samples. Results for this blank must not be greater than the reporting limit (PQL).

8.4 Run a duplicate every 20 samples. Run a matrix spike every 10 samples by weighing out the sample and adding 10 mg of dextrose to it for the TC spike or 35.3 mg of hydrogen carbonate for the IC spike. The recovery can be determined by calculating the theoretical yield from the sample result based on the weight as compared to the native result and adding 4000 ug C that was added from the spike component. The actual yield divided by the theoretical yield will give the recovery.

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 abovezero. The MDL's are determined annually per type of instrument filed with the Inorganic Department

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Manager and with the QAO. The MDL standards are prepared following the procedures outlined in this SOP.

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 EPA 9060 and the Lloyd Kahn method for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Determination of Total Organic Carbon in Sediment, Lloyd Kahn, USEPA Region II, 7/88.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update III, December 1996, Method 9060.

TOC-V series SSM-5000A user's manual.

Installation and Operation of Shimadzu's Solid Sample Module.

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Figure 3 Example of TOC Soil Instrument Results Printout
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TABLE 1

QC REQUIREMENTS – LLOYD KAHN

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS	One per prep batch	80-120% recovery	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
ICV/CCV	ICV at the beginning of the analysis and one after every 10 samples: same conc. as LCS/ICV	80-120% recovery	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reanalyze all samples back to last acceptable CCV recovery
Sample Quadruplicate	One every twenty samples.	≤ 30% RPD and < 3 times the standard deviation	(1) If lab QC in criteria and matrix interference suspected, flag data (2) Else, reanalyze
Matrix spike	One MS per ten samples	75-125% recovery	(1) If LCS in criteria and matrix interference suspected, flag data (2) Else, reanalyze
Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file
MDL study	Once per year	Ideally, PQL = 3 to 5 X the MDL.	Repeat MDL

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TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-741-01	METHOD LLOYD KAHN
Apparatus/Materials		
Reagents	Glucose solid for TC calibration (Dextrose for LCS) Sodium Carbonate for IC calibration (Sodium Hydrogen Carbonate for LCS)	Potassium Hydrogen Phthalate solution used for calibration.
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

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FIGURE 1

EXAMPLE OF TOC SOLIDS LOGBOOK PAGE

Katahdin Analytical Services, Inc.

Carbon Analysis of Solid Samples - Shimadzu TOC-V_{CPH} / SSM-5000A

Analysis Type and Method (Check One)

____Total Carbon (SW846 9060M)
____Total Inorganic Carbon (SW846 9060M)
____Total Organic Carbon (SW846 9060)
____Other (Specify):
X Total Organic Carbon (Lloyd Kahn)

Spiking Information	
LCS Spike Source ID / Compound:	Dextrose SWL1628
CCV Spike Source ID / Compound:	SWL449
MS Spike Source ID / Compound:	SWL1628

Calibration Information	
Calibration Date:	082206
Calibration Analyst:	AmB

KATAHDIN Sample Number	Sample Wt. (mg)	Sample Type * (Circle One)	Spike Added (mg)
Blank		Wet Dry	
LCS		Wet Dry	29.3 RS41720
1 WW4746-7	191.5	Wet Dry	
2 -7Dup	190.7	Wet Dry	
3 -7Dup	190.0	Wet Dry	
-7Dup	190.6	Wet Dry	
-8	166.8	Wet Dry	
-8MS	164.8	Wet Dry	9.9 TV=3960
-9	183.2	Wet Dry	
-10	190.8	Wet Dry	
WW4774-1	168.8	Wet Dry	
-2	176.1	Wet Dry	
CCV		Wet Dry	26.0 TV10400
CCB		Wet Dry	
		Wet Dry	
		Wet Dry	
		Wet Dry	
		Wet Dry	
		Wet Dry	
		Wet Dry	
		Wet Dry	
		Wet Dry	

* "Wet" = field-moist sample (as received). "Dry" = oven-dried sample.

Analyst: AmB	Analysis Date: 091606
Reviewer:	Review Date:

QAWL400

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FIGURE 2

EXAMPLE OF TOC SOLIDS INSTRUMENT PRINTOUT

	Analysis	Sample Name	Dilutio	Result	Comment	Status	Action	Date / Time
1	SSM-TC	Blank	1.000	SSM-TC:0.000 u		Completed		09/16/06 03:54:48
2	SSM-TC	LCS	1.000	SSM-TC:11408		Completed		09/16/06 04:05:21
3	SSM-TC	WW4746-7	1.000	SSM-TC:7.795 u		Completed		09/16/06 04:15:24
4	SSM-TC	WW4746-7D	1.000	SSM-TC:2.308 u		Completed		09/16/06 04:22:10
5	SSM-TC	WW4746-7D	1.000	SSM-TC:10.76 u		Completed		09/16/06 04:33:49
6	SSM-TC	WW4746-7D	1.000	SSM-TC:10.26 u		Completed		09/16/06 04:46:24
7	SSM-TC	WW4746-8	1.000	SSM-TC:0.8961		Completed		09/16/06 04:53:53
8	SSM-TC	WW4746-8M	1.000	SSM-TC:4050 u		Completed		09/16/06 05:06:32
9	SSM-TC	WW4746-9	1.000	SSM-TC:19.84 u		Completed		09/16/06 05:16:14
10	SSM-TC	WW4746-10	1.000	SSM-TC:71.33 u		Completed		09/16/06 05:27:15
11	SSM-TC	WW4774-1	1.000	SSM-TC:4.253 u		Completed		09/16/06 05:35:56
12	SSM-TC	WW4774-2	1.000	SSM-TC:21.96 u		Completed		09/16/06 05:43:31
13	SSM-TC	CCV	1.000	SSM-TC:9766 u		Completed		09/16/06 05:56:03
14	SSM-TC	CCB	1.000	SSM-TC:0.000 u		Completed		09/16/06 06:03:06
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								

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FIGURE 3

EXAMPLE OF TOC SOIL INSTRUMENT RESULTS PRINTOUT

AMB 01/24/07 01:53:34 PM lloyd091606.132

Instr.Information

System	SSM-5000A
Detector	Combustion
Catalyst	Regular Sensitivity
Cell Length	short

Sample

Sample Name:	Blank
Sample ID:	<Untitled>
Origin:	tc method SOILS.met
Chk. Result	

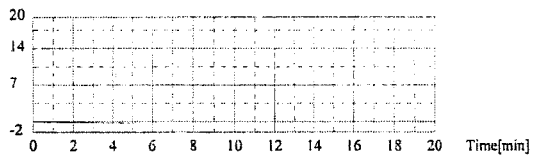
Type	Anal.	Dil.	Density	Result
Unknown	SSM-TC	1.000	1.000mg/uL	SSM-TC:0.000 ug

1. Det

Anal.: SSM-TC

No.	Area	CNV	Abs C	Conc.	Weight	Volume	Ex.	Cal. Curve	Date / Time
1	0.000	0.000	0.000ug	0.000ug	100.0mg	100uL		tccal082206a.2006_08_22_15_58_36.cal	09/16/06 03:54:48 PM

Mean Area	0.000
Mean CNV	0.000
Mean Conc.	0.000ug



Sample

Sample Name:	LCS
Sample ID:	<Untitled>
Origin:	tc method SOILS.met
Chk. Result	

Type	Anal.	Dil.	Density	Result
Unknown	SSM-TC	1.000	1.000mg/uL	SSM-TC:11408 ug

1. Det

Anal.: SSM-TC

1/8

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FIGURE 4

EXAMPLE OF TOC SOLIDS BATCH SHEET

WET CHEMISTRY BATCH REPORT
Sep 20 2006, 03:32 pm
Batch: WG32156

Parameter: TOC In Soil

Date Analyzed: 18-SEP-06

Analyst Initials: CP

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
WG32156-1	MBLANK	LLOYDKAHN	100mg	200.00ug	1	0	U400 ug/g	NA	400	85	400		
WG32156-2	LCS	LLOYDKAHN	22.2mg	200.00ug	1	401576.57658	400000 ug/g	NA	400	85	400		100
WG32156-3	DUP	LLOYDKAHN	186.1mg	200.00ug	1	329.64731	U500 ug/g	NA	400	110	500	NC	
WG32156-4	MS	LLOYDKAHN	177.3mg	200.00ug	1	33309.59213	33000 ug/g	NA	400	110	500		114
WW4746-1	SAMP	LLOYDKAHN	164.7mg	200.00ug	1	786.64612	790 ug/g	86.	400	99	470		
WW4746-3	SAMP	LLOYDKAHN	171.6mg	200.00ug	1	599.57589	600 ug/g	81.	400	100	490		
WW4746-4	SAMP	LLOYDKAHN	102.3mg	200.00ug	1	1360.58295	1400 ug/g	88.	400	96	450		
WW4746-5	SAMP	LLOYDKAHN	125.5mg	200.00ug	1	877.6473	880 ug/g	91.	400	93	440		
WW4746-6	SAMP	LLOYDKAHN	158.4mg	200.00ug	1	426.98726	U500 ug/g	80.	400	100	500		
WW4774-10	SAMP	LLOYDKAHN	155.4mg	200.00ug	1	1518.4076	1500 ug/g	78.	400	110	510		
WW4774-9	SAMP	LLOYDKAHN	182.1mg	200.00ug	1	248.95448	U500 ug/g	79.	400	110	500		
WW4840-1	SAMP	LLOYDKAHN	138.5mg	200.00ug	1	1121.12466	1100 ug/g	83.	400	100	480		
WW4840-10	SAMP	LLOYDKAHN	133.8mg	200.00ug	1	1264.58905	1300 ug/g	81.	400	100	500		
WW4840-11	SAMP	LLOYDKAHN	151.6mg	200.00ug	1	539.39017	540 ug/g	89.	400	95	450		
WW4840-12	SAMP	LLOYDKAHN	157.3mg	200.00ug	1	371.4859	U450 ug/g	89.	400	95	450		
WW4840-13	SAMP	LLOYDKAHN	132.3mg	200.00ug	1	911.67713	910 ug/g	80.	400	100	500		
WW4840-2	SAMP	LLOYDKAHN	137.2mg	200.00ug	1	666.21547	670 ug/g	81.	400	100	490		
WW4840-3	SAMP	LLOYDKAHN	131.5mg	200.00ug	1	1364.60047	1400 ug/g	84.	400	100	480		
WW4840-4	SAMP	LLOYDKAHN	140.8mg	200.00ug	1	1886.46341	1900 ug/g	91.	400	100	490		
WW4840-5	SAMP	LLOYDKAHN	156.2mg	200.00ug	1	444.89933	U520 ug/g	76.	400	110	520		
WW4840-6	SAMP	LLOYDKAHN	180.0mg	200.00ug	1	421.3455	U510 ug/g	79.	400	110	510		
WW4840-7	SAMP	LLOYDKAHN	141.4mg	200.00ug	1	599.5476	600 ug/g	81.	400	100	500		
WW4840-8	SAMP	LLOYDKAHN	162.6mg	200.00ug	1	1138.64589	1100 ug/g	80.	400	100	500		
WW4840-9	SAMP	LLOYDKAHN	173.4mg	200.00ug	1	930.27902	930 ug/g	80.	400	100	500		

Comments:

WG32156-1	WW4774-9
WG32156-2	WW4774-9
WG32156-3	WW4774-9
WG32156-4	WW4774-9

Entered by: CP Date: 9/20/06 Accepted by: KAT Date: 092506

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: SD-902
Revision History
Cover Page
Page 1

TITLE: **SAMPLE RECEIPT AND INTERNAL CONTROL**

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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

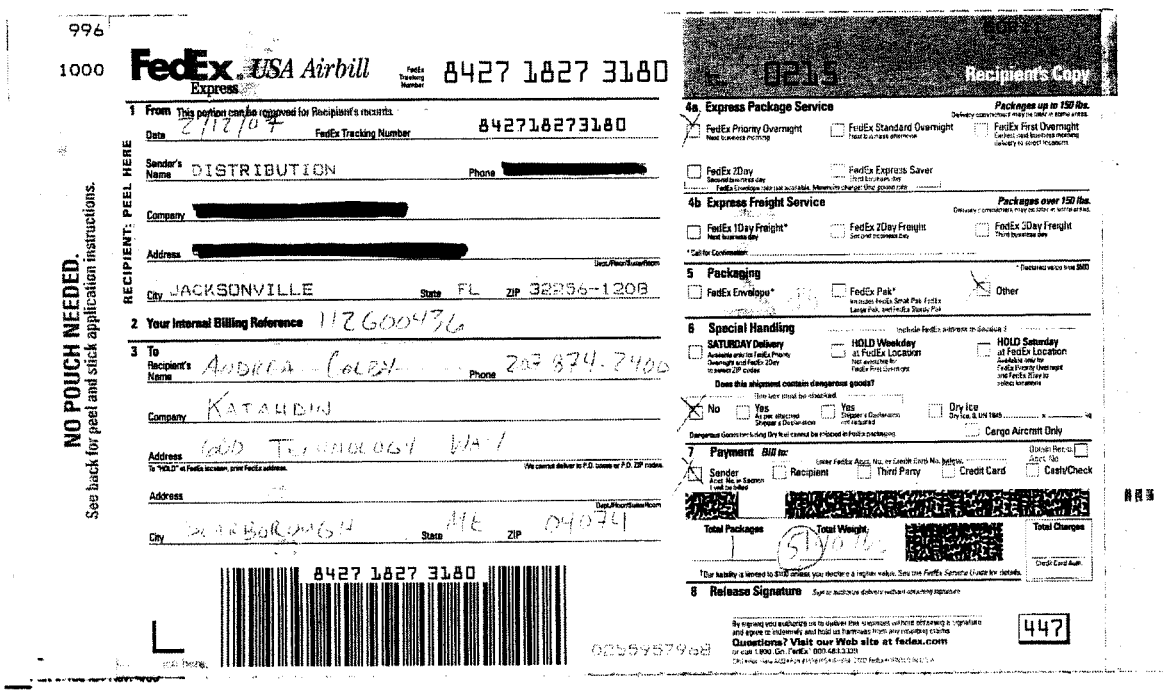
KAS Lab Sample ID	Measured Turbidity (NTU)	Turbidity Date	Turbidity Analyst	Preservation Date	Preservation Time	Preservation Analyst
WW6757-9A	0.22	12-19-06		12/14/06	~~1830~~ 1400	DM
↓ -10A	0.18	↓		↓	↓	↓
WW6758-1A	4.80	↓	↓	↓	↓	↓
WW6776-1A	0.68	12-20-06		12/20/06	1000	DM
WW6855-1A	0.92	12-26-06		12/26/06	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

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KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: SD-903
Revision History
Cover Page
Page 1

TITLE: **SAMPLE DISPOSAL**

Prepared By: _____ Date: 2/01

Approved By:

Group Supervisor: _____ Date: _____

Operations Manager: _____ 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

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

Document Title: Ultrasonication of Solids for the Analysis of Explosives by EPA Method 8330B (HPLC)

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICE, INC. (ALSI)

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Justin Hall, Date
GC Department Supervisor

_____ _____
Evan Cooper, Ph.D. Date
Validator

Annual Review:

_____ _____
Reviewed By Date Reviewed

_____ _____
Approved By Date Approved

_____ _____
Reviewed By Date Reviewed

_____ _____
Approved By Date Approved

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Annual Review (continued):

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Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

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1 Scope and Application

1.1 This Standard Operating Procedure is adapted from U. S. EPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Method 8330B, Revision 2, October 2006.

1.2 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

1.3 This extraction procedure is appropriate for those samples requiring analysis by EPA Method 8330B, Revision 2, October 2006. The analytes cited in Section 1 of the SOP 1B-8330 are the analytes currently validated and acceptable for this procedure.

1.4 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

2.1 Solid samples are ground and extracted, using acetonitrile in an ultrasonic bath, and filtered, in preparation for HPLC analysis.

3 Interferences

3.1 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics in particular must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials.

3.2 Soap residue on glassware may cause degradation of certain compounds. All glassware shall be rinsed carefully with deionized water to avoid the problem.

3.3 Interferences co-extracted from the samples will vary considerably from source to source.

3.4 Light decomposes various target analytes, in particular, tetryl. Precautions shall be taken as to not allow excessive amounts of light to reach sample.

3.5 Heat causes some analytes, in particular, tetryl, to decompose rapidly. Samples shall not be exposed to temperatures above room temperature.

4 Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound shall be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available.

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4.2 ALSI maintains material safety data sheets (MSDS) on all chemicals used in this procedure. ALSI recommends that all individuals performing this SOP familiarize themselves with the MSDS associated with the procedure prior to SOP performance. MSDS are available to all staff and are located in hard copy in the QA reference library and electronically on the ALSI server in the MSDS folder.

4.3 All solvents and reagents used in this procedure shall be handled in a fume hood wearing a fully buttoned lab coat and chemical resistant gloves.

4.2 Solids containing a high amount of explosive analytes may be present as finely ground grayish-white material. Extreme caution shall be used with these samples. Samples containing large amounts of grayish-white material, or lumps of material that have a chemical appearance or are known to have come from a site containing high amounts of explosives shall be suspect and not ground as described in 09-8330B Grinding.

5 Apparatus and Materials

5.1 Ultrasonic Disrupter – Bronson 2510, 80 watt, or equivalent, equipped with floating tray, and a temperature-controlled bath.

5.2 Vortex Mixer.

5.3 Balance – capable of weighing ± 0.01 g.

5.4 Syringes – gastight, appropriate sizes.

5.5 Syringes – disposable 10-mL (0.90 mm x 25 mm).

5.6 Syringe Filters – Spartan #00760, 0.45-µm Teflon (PTFE) membrane, 25 mm diameter, or equivalent.

5.7 Pasteur Pipets – glass disposable.

5.8 Culture Tubes – glass, disposable with Teflon-lined (PTFE) screw caps, 16 x 125 mm.

5.9 VOA Vials – 20-mL, pre-cleaned glass.

5.10 Vials – amber, glass 2-dram.

5.11 Weigh Boats – aluminum.

5.12 Volumetric Pipets – 5-mL, 10-mL, Class A.

5.13 600-micron sieve – stainless steel.

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6 Reagents

NOTE: Unless otherwise noted in this section, all chemicals are stored at room temperature and labeled with an expiration date of five years from receipt. Manufacturer's labeled expiration dates, when less than five years, take precedent over all other expiration dates.

6.1 Acetonitrile: HPLC grade purchased from VWR Catalog #BJ015-4 or equivalent.

6.2 Methanol (MeOH): HPLC grade purchased from VWR Catalog #EM-MX0475-1 or equivalent.

6.3 Surrogate Stock Solution – 1-chloro-3-nitrobenzene (1000 µg/mL) in acetonitrile, Ultra Scientific part #IST-630, or equivalent. Store at -10ºC to -20ºC protected from light.

6.4 LCS/matrix spike stock solution – Cerilliant Method 8330 Stock Standard P/N ERE-021, or equivalent. This standard contains all 14 of the explosive compounds listed in EPA method 8330A, all at 200 µg/mL in acetonitrile. Store at -10 to -20°C protected from light.

6.5 Additional Compounds LCS/matrix spike stock solutions: The compounds Nitroglycerin (NG), Pentaerythritol tetranitrate (PETN), and 3,5-Dinitroaniline (3,5-DNA), may also be analyzed by this method. The stock standards for these compounds are purchased as separate single component solutions:

6.5.1 NG Primary Stock Solution: Cerilliant Trinitroglycerin Standard P/N T-002, or equivalent. This standard contains NG at 1000 µg/mL in acetonitrile. Store from -10°C to -20°C protected from light.

6.5.2 PETN Primary Stock Solution: Cerilliant PETN Standard P/N P-037, or equivalent. This standard contains PETN at 1000 µg/mL in acetonitrile. Store from -10°C to -20°C protected from light.

6.9.3 3,5-DNA Primary Stock Solution: Restek 3,5-DNA P/N 31661, or equivalent. This standard contains 3,5-DNA at 1000 µg/mL in acetonitrile. Store at -10°C to -20°C protected from light.

6.6 Sand - Ottawa 20-30 mesh. Fisher Scientific, Part #523-50 (1999). Muffle furnace at 400ºC for four hours. Cool and store in a glass bottle at room temperature. Label with an expiration date of 180 days.

6.7 VOA vials, 20-mL, purchased from VWR, or equivalent.

7 Glassware Cleaning

7.1 Remove surface residuals immediately after use by rinsing with tap water or the last

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solvent used.

7.2 Soak the glassware in hot water and Liquinox detergent to float most particulate material from glassware. Use a brush to scrub the glassware to aid in the removal of residual materials.

7.3 Hot water rinse the glassware.

7.4 If the glassware used was in contact with high level samples and/or there is an apparent residue remaining on the glassware after the first three steps, it will be necessary to soak the glassware in a strong oxidizing agent to destroy traces of residual compounds. The oxidizing agent currently used is Chem Solv 2157, manufactured by Mallinckrodt and distributed by Baxter Scientific (part #2157-INY, 1995). Chem Solv 2157 is a safer alternative to chromic acid solutions typically suggested for cleaning glassware. The manufacturer's instructions for the safe handling of Chem Solv 2157 shall be stored with each container and reviewed before its use. Glassware suspected or known to have been in contact with high-level samples shall soak for 1 hour in the Chem Solv solution. Rinse with copious amounts of tap water and resubmit for normal cleaning procedure (Section 7.2).

7.5 Rinse the glassware thoroughly with deionized water to remove remaining materials and any metallic deposits.

7.6 Dry the glassware in an oven at 130ºC ± 30ºC for a minimum of 30 minutes.

7.7 Allow the glassware to cool for a minimum of 30 minutes.

7.8 Wrap glassware openings with aluminum foil if stored for later use.

7.9 Flush all glassware immediately before use with the extraction solvent being used for the application.

8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 Initial Demonstration of Capability: This demonstration must be successfully performed by each analyst prior to being considered proficient to analyze samples by this method. Ongoing proficiency must be established annually as specified in the QA Plan, under Technical Training,

8.2.1 Four QC check samples will be prepared: Spike four 10.0 g samples of reagent sand with 20.0 µL of the Spike Solution. Take the samples through all steps of the extraction as described in this SOP.

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8.2.2 Analyze samples by HPLC as described in ALSI SOP 1B-8330.

8.2.3 An average recovery range of 70-130% recovery will be used as guidance to assess laboratory performance. See ALSI SOP 1B-8330 for calculation of percent recovery. A precision control limit of 30% RSD for all four check samples will be used as guidance to assess laboratory performance. When sufficient data is gathered, in house control limits will be used to assess laboratory performance.

8.2.4 If one or more analytes do not meet this criterion, identify and correct source of problem and report test for those analytes that initially failed.

8.3 A method blank must be run with each batch of samples prepared. It is imperative that the blanks be subjected to exactly the same analytical procedures as those used in the actual samples. This includes grinding, the addition of the surrogate standards, and the use of sodium sulfate and other chemicals used in the extraction procedures.

8.4 A matrix spike and matrix spike duplicate or a matrix spike and duplicate must be extracted with each batch. If insufficient sample is available to perform a matrix spike or duplicate, a comment must be placed in the extraction log.

8.4.1 Samples selected for duplicate and matrix spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

8.5 A laboratory control sample (LCS) must be extracted with every batch. The LCS is prepared similarly to a method blank but is spiked with the LCS spiking solution.

8.6 The size of any extraction batch cannot exceed 20 samples.

8.7 MDL studies must be performed according to ALSI SOP 99-MDL or the reference method, whichever is more frequent.

9 Sample Collection, Preservation and Handling

9.1 Soil samples shall be collected as per method 8330B, through the use of multi-increment sampling. No preservation is recommended. Upon receipt, samples are air dried, unexposed to light, to a constant weight. If samples are not dried immediately, they may be stored in the dark at 4ºC or lower. After air drying the sample, it can be held at ambient temperature or cooler.

9.2 It is recommended and is also the practice at ALSI that samples are dried and extracted within 14 days of collection. However, it has been demonstrated that the dried/ground

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sample remains stable for up to two months.

9.3 Sample extracts must be analyzed within 40 days of extraction. All extracts shall be stored protected from light above the freezing point of water up to 6ºC.

9.4 See SOP 20-Field Services Sampling Plan for collection procedures.

10 Procedure

10.1 Sample drying and grinding--Refer to SOP 09-8330B Grinding.

10.2 Sample Homogenization –

10.2.1 Once the sample has been dried and completely ground, an aliquot for extraction may be removed. This can be accomplished by the following steps.

10.2.1.1 Lay the entire sample out on the original tray it was dried on so that it is 1-2 cm thick. Caution needs to be taken to not spread the dust which could in turn cause analyte loss.

10.2.1.2 Place an appropriately labeled 20-mL VOA vial on a balance. Tare the balance.

10.2.1.3 Using a clean, dry, acetonitrile rinsed spatula, obtain 10 grams of the sample, using at least 30 different increments (portions ~0.3g) from randomly chosen locations collecting each in the VOA vial.

10.3 Sample Extraction

10.2.1 Record the actual weight in extraction logbook.

10.2.2 Add 10 µL of working surrogate solution to the surface of all samples, blanks, and LCS's.

10.2.3 Add 10 µL of the necessary spiking solution(s) (6.4 and/or 6.5) to all matrix spikes and LCS.

10.2.4 Using an acetonitrile rinsed, Class A, 20-mL volumetric pipet, add 20.0 mL of acetonitrile to all samples, blanks, and LCS's. Cap all tubes with clean PTFE-lined caps.

10.2.5 Vortex swirl each tube for one minute. (In larger batches, this step may take considerable time. Precautions shall be taken as to not subject tubes to excessive light or heat.)

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10.2.6 Place all tubes into the floating tray of the ultrasonic bath. (Samples shall be upright during sonication; a rubber band may be needed to hold all the tubes together and upright over the entire sonication process.) Cover the entire top of sonicator with aluminum foil so as not to allow any light to reach the samples.

10.2.7 Set the ultrasonic bath to the "run continuously" position, recording the time in the logbook. Samples are sonicated for 18 hours. (During the sonication, bath temperature must stay in a 15°C - 25ºC temperature range. To ensure this, a chilling apparatus needs to be in place. Occasional monitoring of the bath temperature needs to be done in order to ensure the water stays within this range.)

10.2.8 After 18 hours of sonication, turn the bath off and record the time. Remove the samples from the bath and place in a tube rack and allow to settle for 30 minutes.

10.3 Filtering of Supernatant

10.3.1 Assemble a 10-mL disposable syringe without the PTFE filter. Draw entire supernatant into syringe. (Depending on sample matrix, if any residual substrate is left on the syringe tip, it will need to be rinsed with a small amount of acetonitrile.) Invert syringe and attach a 0.45-µm PTFE filter.

10.3.2 Discard first 1 mL and retain remainder in a PTFE-capped 2-dram amber vial labeled with all appropriate COC information and LIMS ID. For dirtier samples, the syringe filter may need to be replaced before the entire sample is pushed through.

11 Calculations

11.1 Not applicable.

12 Reporting Results

12.1 Pre-extraction Entries (Batching).

12.1.1 In the "batching" function of the LIMS select the "appropriate" RULE under the EXTR Queue.

12.1.2 Select the desired samples for the extraction batch by placing a check beside the COC# number. Press the "build batch" button.

12.1.3 Preview the "notes" section of the batch schedule to identify client specified QC. Determine which COC#s have additional sample available for the required sample matrix QCs' and add them to the batch using the "Add QC" button.

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12.1.4 Verify all desired samples are in the batch and appropriate QCs' have been added. Save the batch. Record the HBN# in the extraction logbook.

12.2 Post Extraction Entries (Posting) and Data Review

12.2.1 Once extracts are vialed and initial sample volumes and extract final volumes have been recorded in the extraction logbook, the initial and final volumes must be posted in the LIMS.

12.2.2 In the "operations" function of the LIMS select one of the posting options.

12.2.3 Enter the technician's initials beginning the extraction and the date and time the extraction was initiated as recorded in the extraction logbook. Times must be recorded in hours and minutes. Seconds shall be recorded as "00".

12.2.4 Enter the appropriate initial and final volume in the LIMS. When entering data into Horizon LIMS, do not round off results; Horizon will automatically perform rounding appropriate to the method. Verify all entries are correct and "save" the entries.

12.2.5 Date and initial in the extraction logbook in the "approved" entry at the bottom of the page.

12.2.6 Review the labeling on all vials and deliver to the GC Department for storage at a temperature above the freezing point of water up to 6°C until analysis.

12.2.7 A second technician must verify all initials, dates, times, and volumes have been correctly transferred from the logbook to the LIMS. Date and initial in the extraction logbook in the "reviewed" entry at the bottom of the page.

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

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15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

16 Troubleshooting

16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the instrumentation used in this method.

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APPENDIX A

Work continued from page:____

HPLC EXTRACTION

Work continued to page:____

EPA Method: _____ Surrogate ID: _____

ALSI SOP: _____ Surrogate Amount: _____

Extraction:	Date	Time	Tech	Sonication:	Time On	Time Off	Total Hours Sonicated	Tech

Line #	Sample ID	Sample Amount	QC Type	Spike ID	Spike Amount	Final Volume	Elution Time	Sample Specific Comments (Include weigh data for drying, if applicable.)
1.)								
2.)								
3.)								
4.)								
5.)								
6.)								
7.)								
8.)								
9.)								
10.)								
11.)								
12.)								
13.)								
14.)								
15.)								

Reagent Identification(s): _____

_____

Approved By:_____
Date Approved:_____

Page #:____

Revised 2/1/05

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SOP Change History Sheet

Section No.	Section	Reason for Change

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SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

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Document Title: Solid Phase Extraction of Water for the Analysis of Explosives by EPA Method 8330A (HPLC)

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICE, INC. (ALSI)
Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

_____ _____
Helen MacMinn, Date
Quality Assurance Manager

_____ _____
Justin Hall, Date
GC Department Supervisor

_____ _____
Evan Cooper, Ph.D. Date
Validator

Annual Review:

_____ _____
Reviewed By Date Reviewed

_____ _____
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Annual Review (continued):

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
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1 Scope and Application

1.1 This Standard Operating Procedure is adapted from U. S. EPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Method 8330A, Revision 1, January 1998.

1.2 This document states the laboratory's policies and procedures established in order to meet requirements of all certification/accreditations currently held by the laboratory, including the most current NELAC Standards.

1.3 This extraction procedure is appropriate for those samples requiring analysis by EPA Method 8330A, Revision 1, January 1998. The analytes cited in Section 1 of SOP 1B-8330 are the analytes currently validated and acceptable for this procedure.

1.4 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

2.1 A measured volume of water, usually 1 liter, is passed through an SDB-RPS disk and eluted with acetonitrile.

3 Interferences

3.1 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics in particular must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials.

3.2 Soap residue on glassware may cause degradation of certain compounds. All glassware shall be rinsed carefully with deionized water to avoid the problem.

3.3 Interferences co-extracted from the samples will vary considerably from source to source.

3.4 Light decomposes various target analytes, in particular, tetryl. Precautions shall be taken as to not allow excessive amounts of light to reach sample.

3.5 Heat causes some analytes, in particular, tetryl, to decompose rapidly. Samples shall not be exposed to temperatures above room temperature.

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4 Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound shall be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available.

4.2 ALSI maintains material safety data sheets (MSDS) on all chemicals used in this procedure. ALSI recommends that all individuals performing this SOP familiarize themselves with the MSDS associated with the procedure prior to SOP performance. MSDS are available to all staff and are located in hard copy in the QA reference library and electronically on the ALSI server in the MSDS folder.

4.3 All solvents and reagents used in this procedure shall be handled in a fume hood wearing safety glasses, a fully buttoned lab coat, and chemical resistant gloves.

5 Apparatus and Materials

5.1 Vacuum pump – equipped with trap, sufficient capacity to maintain 30 inches of mercury.

5.2 Filtration apparatus – glass or Teflon-lined. Filtration surface shall accommodate 47-mm disks. An addition reservoir with a minimum capacity of 250 mL is recommended along with connecting clamps. Fritted glass support and 1.5 L vacuum trap.

5.3 Solid phase extraction disks – 47-mm SDB-RPS, a PTFE membrane with surface entrapped particles of sulfonated styrene divinyl benzene that have reversed phase modification, 3M Empore P/N 2241, or equivalent. Each lot of extraction disks shall be evaluated prior to use with actual samples. This is done by the extraction of a method blank, to verify that the disks do not elute any interfering compounds.

5.4 Collection tube – glass 25 x 250 mm tube.

5.5 Tweezers – Teflon-coated.

5.6 Pipets – glass 10-mL with 0.1 mL graduations.

5.7 Syringes – gastight, appropriate sized.

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5.8 VOA vials, 2- mL pre-cleaned glass.

5.9 Pasteur pipets – glass disposable.

5.10 Vials – amber 2-dram.

5.11 Graduated cylinder, Class A, TC, 1-L.

6 Reagents

NOTE: Unless otherwise noted in this section, all chemicals are stored at room temperature and labeled with an expiration date of five years from receipt. Manufacturer's labeled expiration dates, when less than five years, take precedent over all other expiration dates.

6.1 Reagent Water – A Filson Water Purification System is used which provides analyte-free >16.0 megaohm deionized water on demand.

6.2 Acetonitrile – HPLC grade, Burdick & Jackson P/N AH015-4, or equivalent.

6.3 Methanol – HPLC grade, Burdick & Jackson P/N AH230-4, or equivalent.

6.4 Surrogate Stock Solution – 1-chloro-3-nitrobenzene (1000 µg/mL) in acetonitrile, Ultra Scientific part #IST-630, or equivalent. Store at -10 to -20°C protected from light.

6.6 LCS/matrix spike stock solution – Cerilliant Method 8330 Stock Standard P/N ERE-021, or equivalent. This standard contains all 14 of the explosive compounds listed in EPA method 8330A, all at 200 µg/mL in acetonitrile. Store at -10 to -20°C protected from light.

6.7 Nitroglycerin LCS/matrix spike – Cerilliant Trinitroglycerin Standard P/N T-002, or equivalent. This standard contains nitroglycerin at 1000 µg/mL in acetonitrile. Store at -10 to -20°C protected from light. NOTE: nitroglycerin should only be spiked when specifically requested by a client. It is not a routine part of 8330A analysis.

6.8 Filter Aid – 3M Empore P/N FA400, or equivalent.

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7 Glassware Cleaning

7.1 Remove surface residuals immediately after use by rinsing with tap water or the last solvent used.

7.2 Soak the glassware in hot water and liquinox detergent to float most particulate material from glassware. Use a brush to scrub the glassware to aid in the removal of residual materials.

7.3 Hot water rinse the glassware.

7.4 If the glassware used was in contact with high level samples and/or there is an apparent residue remaining on the glassware after the first three steps, it will be necessary to soak the glassware in a strong oxidizing agent to destroy traces of residual compounds. The oxidizing agent currently used is Chem Solv 2157, manufactured by Mallinckrodt and distributed by Baxter Scientific (part #2157-INY, 1995). Chem Solv 2157 is a safer alternative to chromic acid solutions typically suggested for cleaning glassware. The manufacturer's instructions for the safe handling of Chem Solv 2157 shall be stored with each container and reviewed before its use. Glassware suspected or known to have been in contact with high level samples shall soak for 1 hour in the Chem Solv solution. Rinse with copious amounts of tap water and resubmit for normal cleaning procedure (Section 7.2).

7.5 Rinse the glassware thoroughly with deionized water to remove remaining materials and any metallic deposits.

7.6 Dry the glassware in an oven at 130 +/-30°C for a minimum of 30 minutes.

7.7 Allow the glassware to cool for a minimum of 30 minutes.

7.8 Wrap glassware openings with aluminum foil if stored for later use.

7.9 Flush all glassware immediately before use with the extraction solvent being used for the application.

7.10 Glassware cleaning filtration apparatus.

7.10.1 The filtration apparatus itself is the only nonconsumable used in the extraction process. With the reservoir removed, rinse the filtration surface

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with 3-5 mL of acetonitrite. Pull a vacuum on the surface until dry.

7.10.2 Attach the reservoir and rinse the walls down with approximately 15 mL of acetonitrite. Pull a vacuum on the unit until all surfaces are visibly dry.

7.10.3 Samples that leave a visible residue may require a cleaning and drying of the filtration apparatus as described in sections 7.1 – 7.9.

8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 A method blank must be run with each batch of samples prepared. It is imperative that the blanks be subjected to exactly the same analytical procedures as those used in the actual samples. This includes the addition of the surrogate standards and other chemicals used in the extraction procedures.

8.3 A matrix spike and matrix spike duplicate or a matrix spike and duplicate must be extracted with each batch. If insufficient sample is available to perform a matrix spike or duplicate, a comment must be placed in the extraction log.

8.3.1 Samples selected for duplicate and matrix spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

8.4 A laboratory control sample (LCS) must be extracted with every batch. The LCS is prepared similarly to a method blank but is spiked with the LCS spiking solution.

8.5 The size of any extraction batch cannot exceed 20 samples.

8.6 Initial Demonstration of Capability: This demonstration must be successfully performed by each analyst prior to being considered proficient to analyze samples by this method. Ongoing proficiency must be established annually as specified in the QA Plan, under Technical Training,

8.6.1 Four QC check samples will be prepared: Spike four 1000 mL samples of

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reagent water with 10.0 µL of the Spike Solution. Take the samples through all steps of the extraction as described in this SOP.

8.6.2 Analyze samples by HPLC as described in ALSI SOP 1B-8330.

8.6.3 An average recovery range of 70-130% recovery will be used as guidance to assess laboratory performance. See ALSI SOP 1B-8330 for calculation of percent recovery. A precision control limit of 30% RSD for all four check samples will be used as guidance to assess laboratory performance. When sufficient data is gathered, in house control limits will be used to assess laboratory performance.

8.6.4 If one or more analytes do not meet this criterion, identify and correct source of problem and report test for those analytes that initially failed.

8.7 MDL studies must be performed according to ALSI SOP 99-MDL or the reference method, whichever is more frequent.

9 Sample Collection, Preservation and Handling

9.1 Containers used to collect samples must be specially cleaned 1-Liter amber glass bottles. These sample bottles shall have screw caps with Teflon lining. A minimum of 1-Liter of sample is necessary, and three 1-Liter bottles for MS/MSD processing.

9.2 All samples shall be stored above the freezing point of water up to 6°C until extraction. No preservation is recommended.

9.3 Samples must be extracted within 7 days of collection.

9.4 Sample extracts must be analyzed within 40 days of extraction. All extracts shall be stored protected from light above the freezing point of water up to 6ºC.

9.5 See SOP 20-Field Services Sampling Plan for collection procedures.

10 Procedure

10.1 Examine the sample closely. A water sample may contain a significant amount of suspended solids or sediment. This extraction procedure is designed for water samples that are relatively free of sediment and suspended solids. If a sample

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contains a sediment layer larger than 0.5 cm, then the client must be notified that this sediment will not be part of the analysis. They should be given the option to have the sediment analyzed by the solids procedure, 09-8330S. For excessive sediment layers > 0.5 cm, the liquid layer must be carefully decanted into a separate clean bottle for analysis by this method. The original sample bottle with the sediment should be retained until the client is satisfied that analysis of the sediment is unnecessary.

10.2 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. It may be necessary to decant sufficient sample in order to allow for the addition of 5 mL methanol as described later in this procedure. If that is the case, the bottle should be marked after decantation.

10.3 Add 1000 mL of reagent water to 2 (two), l-liter, glass bottles. Label one as the blank and the other as the LCS. The bottles must have sufficient headspace to allow for the addition of 5 mL of methanol.

10.4 Using a 10-mL graduated pipet, add 5 mL of methanol to all blanks, matrix spikes and samples. Cap all bottles and invert several times to mix.

10.5 Using a 25-µL syringe, add 5.0 µL of the surrogate stock solution to all samples, blanks and LCS's.

10.6 Using a 25-µL syringe, add 5.0 µL of the working spiking solution to all matrix spikes and the LCS.

10.7 If the analysis of nitroglycerin is specifically requested for a sample, then the associated matrix spikes and LCS must be spiked with nitroglycerin. IF NITROGLYCERIN IS NOT SPECIFICALLY REQUESTED DO NOT SPIKE WITH NITROGLYCERIN. Using a 25-µL syringe, add 5.0 µL of the nitroglycerin spiking solution to all matrix spikes and LCS.

10.8 Cap all bottles and invert several times to mix.

10.9 Ensure the filtration apparatus has been cleaned and dried as described in Section 7.

10.10 This SOP utilizes standard 47-mm diameter extraction disks. All solvent volumes used in washing and eluting disks are appropriate only with this size. This SOP is not applicable if other size disks are used.

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10.11 Assemble the filtration apparatus while also inserting a large 25 mm x 250 mm collection tube inside the flask so rinsing solvents will be collected directly into it through the filter support. Attach the flask to a vacuum hose from the trap which is connected to the pump.

10.12 Using clean tweezers, place an extraction disk onto the frit surface. Center the disk and make sure no wrinkles are present if flexible Teflon disks are used. Replace the reservoir carefully on top of apparatus so the sides are even with frit base and clamp into place.

10.13 If samples in the batch contain significant suspended solids, then the use of filter aid is recommended. In this event, carefully add approximately 40 g of filter aid to the surface of the extraction disk. Distribute the filter aid carefully so as to create a uniform layer of about 1.0 cm depth. When using filter aid, it is important to slowly add liquids down the disk reservoir so as to avoid disturbing the filter aid bed.

10.14 Precondition the disk by adding 10 mL of an acetonitrite using a 10-mL graduated pipet then applying low vacuum, draw about half of the solvent through the disk. Stop the vacuum to allow the disk to soak for about a minute, then start again to draw the remaining solvent through the disk turning up the vacuum to full so the disk is taken to what appears to be complete dryness. Turn vacuum back down to low then stop the pump.

10.15 Using a 10-mL graduated pipet, add 15 mL of acetonitrile to the disk, and pull it through the disk with vacuum, being careful to maintain a minimal liquid layer (1-2 mm) above the disk. The disk MUST NOT go to dryness from this point on until the disk is eluted post sample addition.

10.16 Add another 15 mL of acetonitrile to the disk, and pull it through with vacuum as in step 10.15 above.

10.17 Add 30 mL of reagent water to the disk.

10.18 Pull a slow vacuum until a minimal layer of water remains on the disk.

10.19 Remove the collection tube from the flask. Invert the sample bottle to mix and begin to pour the sample into the reservoir.

10.20 Adjust the vacuum to 8 inches of Hg. It should take about 6 minutes for clean

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samples to be pulled through the disk.

10.21 Once the entire sample has passed through the disk, dry the disk at 20 inches of Hg for 3 minutes.

10.22 Remove the filtered water sample from the flask.

10.23 Place a 20-mL VOA vial labeled with the COC# and LIMS ID under the elution tip.

10.24 Add exactly 5 mL of acetonitrile directly to the surface of the disk using a Class A graduated 10-mL pipet. It is critical that no more than 5 mL of acetonitrile be added to the disk. The final extract volume is targeted for 5 mL and any volume that results in an excess of 5 mL will result in raised reporting limits.

10.25 Carefully pull half of the solvent through the disk. Allow the disk to soak for 1 minute. Pull the remaining solvent through at 20 inches of Hg. Allow the disk to elute for 30 seconds at 20 inches of Hg.

10.26 Remove the 20-mL VOA vial containing the extract and cap immediately.

10.27 Pour the extract into an amber 2-dram vial. Label the vial with the COC # and the LIMS ID.

10.28 Check the volumes of all sample bottles by refilling with water to the mark and then pouring the contents into a 1-L graduated cylinder.

11 Calculations

11.1 Not applicable.

12 Reporting Results

12.1 Pre-extraction Entries (Batching).

12.1.1 In the “batching” function of the LIMS select the “appropriate” RULE under the EXTR Queue.

12.1.2 Select the desired samples for the extraction batch by placing a check beside the COC# number. Press the “build batch” button.

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12.1.3 Preview the "notes" section of the batch schedule to identify client specified QC. Determine which COC#s have additional sample available for the required sample matrix QCs' and add them to the batch using the "Add QC" button.

12.1.4 Verify all desired samples are in the batch and appropriate QCs' have been added. Save the batch. Record the HBN# in the extraction logbook.

12.2 Post Extraction Entries (Posting) and Data Review

12.2.1 Once extracts are vialed and initial sample volumes and extract final volumes have been recorded in the extraction logbook, the initial and final volumes must be posted in the LIMS.

12.2.2 In the "operations" function of the LIMS select one of the posting options.

12.2.3 Enter the technician's initials beginning the extraction and the date and time the extraction was initiated as recorded in the extraction logbook. Times must be recorded in hours and minutes. Seconds shall be recorded as "00".

12.2.4 Enter the appropriate initial and final volume in the LIMS. When entering data into Horizon LIMS, do not round off results; Horizon will automatically perform rounding appropriate to the method. Verify all entries are correct and "save" the entries.

12.2.5 Date and initial in the extraction logbook in the "approved" entry at the bottom of the page.

12.2.6 Review the labeling on all vials and deliver to the GC Department for storage at a temperature above the freezing point of water up to 6°C until analysis.

12.2.7 A second technician must verify all initials, dates, times, and volumes have been correctly transferred from the logbook to the LIMS. Date and initial in the extraction logbook in the "reviewed" entry at the bottom of the page.

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13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

16 Troubleshooting

16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the instrumentation used in this method.

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Appendix A

Work continued from page:____

HPLC EXTRACTION

Work continued to page:____

EPA Method: _____ Surrogate ID: _____

ALSI SOP: _____ Surrogate Amount: _____

Extraction:	Date	Time	Tech	Sonication:	Time On	Time Off	Total Hours Sonicated	Tech

Line #	Sample ID	Sample Amount	QC Type	Spike ID	Spike Amount	Final Volume	Elution Time	Sample Specific Comments (Include weigh data for drying, if applicable.)
1.)								
2.)								
3.)								
4.)								
5.)								
6.)								
7.)								
8.)								
9.)								
10.)								
11.)								
12.)								
13.)								
14.)								
15.)								

Reagent Identification(s): _____

_____

Approved By:_____
Date Approved:_____

Page #:____

Revised 2/1/05

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SOP Change History Sheet

Section No.	Section	Reason for Change
Revision 2: 11/16/07		
Spelling, grammar, and formatting changes may have been made throughout SOP for clarity, correctness, and site conformity.		
Temperature ranges updated to read "above the freezing point of water to 6°C."		
1.3, 1.4	Scope and Application	Added sections
3.4, 3.5	Interferences	Added sections
4.2, 4.3	Safety	Added sections
5	Apparatus and Materials	Updates to materials, manufacturer and part numbers
6	Reagents	Added NOTE
6.4	Reagents	Removed section
6.5, 6.6	Reagents	Updated to current practice
7.6	Glassware Cleaning	Removed section
7.7, 7.8	Glassware Cleaning	Added sections
8.6, 8.7	Quality Control	Added sections
9.1, 9.4, 9.5	Sample Collection…	Added sections
10	Procedure	Numerous revisions made throughout section
12	Reporting Results	Updated section from AMS to Horizon LIMS
15	Definitions	Added section
16	Troubleshooting	Added section
Appendix A		Added section

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SOP Change History Sheet (continued)

Section No. ***Revision 3: 01/29/08***	**Section**	**Reason for Change**
5.3	Apparatus and Materials	Added verification verbiage per DoD audit

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SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

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Document Title: Nitroaromatics and Nitroamines by HPLC with Ultraviolet Detection

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICE, INC. (ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

_____ _____
Helen MacMinn, Date
Quality Assurance Manager

_____ _____
Justin Hall, Date
GC Department Supervisor

_____ _____
Evan Cooper, Ph.D. Date
Validator

Annual Review:

_____ _____
Reviewed By Date Reviewed

_____ _____
Approved By Date Approved

_____ _____
Reviewed By Date Reviewed

_____ _____
Approved By Date Approved

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Annual Review (continued):

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

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Approved By	Date Approved

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1 Scope and Application

1.1 This standard operating procedure is adapted from EPA Method 8330B, Rev. 2, October 2006, SW 846, "Test Methods for Evaluating Solid Waste". Method Detection Limits (MDLs) are available on the ALSI network and are maintained and updated by QA Department. The detection limits for a specific sample may differ from those listed due to the nature of interferences in a particular sample matrix.

1.2 This method is used to determine nitroaromatics and nitramines in a variety of solid and liquid matrices. This method is applicable to nearly all types of samples regardless of water content, including ground water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils and sediments. The following compound can be determined by this method:

ANALYTE	ABBREVIATION	CAS#
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (Octogen)	HMX	2691-41-0
Hexahydro-1,3,5-trinitro-1,3,5-triazine (Hexogen)	RDX	121-82-4
1,3,5-Trinitrobenzene	1,3,5-TNB	99-35-4
1,3-Dinitrobenzene	1,3DNB	99-65-0
Methyl-2,4,6-trinitrophenylnitramine	Tetryl	479-75-8
Nitrobenzene	NB	98-95-3
2,4,6-Trinitrotoluene	2,4,6-TNT	118-96-7
4-Amino-2,6-dinitrotoluene	4-Am-DNT	1946-51-0
2-Amino-4,6-dinitrotoluene	2-Am-DNT	35572-78-2
2,4-Dinitrotoluene	2,4-DNT	121-14-2
2,6-Dinitrotoluene	2,6-DNT	606-20-2
2-Nitrotoluene	2-NT	88-72-2
3-Nitrotoluene	3-NT	99-08-1
4-Nitrotoluene	4-NT	99-99-0
Nitroglycerin	NG	55-63-0
Pentaerythritol tetranitrate	PETN	78-11-5
3,5-Dinitroaniline	3,5-DNA	618-87-1

1.3 This is a high performance liquid chromatography (HPLC) method applicable to the determination of the compounds listed above.

1.4 This method is restricted for use by or under the supervision of analysts experienced in the use of HPLC systems and who have demonstrated the ability to generate acceptable results using the procedure described in this document.

1.5 This document states the laboratory's policies and procedures established in order to meet the requirements of all certifications/accreditations currently held by the laboratory including the most recent NELAC standards.

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1.6 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

2.1 A measured volume/weight of sample is processed using one of the following procedures: a salting-out extraction, Solid-phase extraction, ultrasonic bath extraction, or a high level direct injection. An aliquot of the extract/sample is injected into a HPLC and the compounds are detected by the ultraviolet (UV) detector.

3 Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis by running method blanks.

3.2 2,4-DNT and 2,6-DNT elute at similar retention times (retention time difference of 0.2 minutes). A large concentration of one isomer may mask the response of the other isomer. If it is not apparent that both isomers are present (or are not detected), an isomeric mixture shall be reported.

3.3 Tetryl decomposes rapidly in methanol/water solutions, as well as with heat. All aqueous samples expected to contain tetryl shall be diluted with acetonitrile prior to filtration and acidified to pH <3. All samples expected to contain tetryl shall not be exposed to temperatures above room temperature.

3.4 Degradation products of tetryl appear as a shoulder on the 2,4,6-TNT peak. Peak heights rather than peak areas shall be used when tetryl is present in concentrations that are significant relative to the concentration of 2,4,6-TNT.

4 Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound shall be treated as a potential health hazard. From this viewpoint exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory maintains a current awareness file of the OSHA regulations regarding the safe handling of the chemicals specified in this method.

4.2 ALSI maintains material safety data sheets (MSDS) on all chemicals used in this procedure. ALSI recommends that all individuals performing this SOP familiarize themselves with the MSDSs associated with the procedure prior to SOP performance. MSDS are available to all staff and are located in hard copy in the QA reference library and electronically on the ALSI server in

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the MSDS folder.

4.3 When handling any chemicals in this procedure, including sample extracts, it is recommended that gloves and safety glasses be used as personal protective equipment (PPE).

5 Apparatus and Materials

5.1 High Performance Liquid Chromatography (HPLC) apparatus

5.1.1 HPLC1 – Dionex Ultimate 3000 or equivalent.

5.2 HPLC Columns

5.2.1 Preferred columns, used when nitroglycerin is not analyzed:

5.2.1.1 PRIMARY: Dionex Acclaim Explosives E1, P/N 064305, used with a guard column, Dionex Acclaim Explosives E1, P/N 064303, or equivalent.

5.2.1.2 CONFIRMATION: Dionex Acclaim Explosives E2, P/N 064309, used with a guard column, Dionex Acclaim Explosives E2, P/N 064307, or equivalent.

5.2.2 Alternate Primary Column: Merck Purospher RP-18e column; 5 µm particle size; length 25 cm; i.d. 4.6 mm; Merck P/N 1.50169.0001 or Agilent P/N 79925PE-584, or equivalent

5.3 Data system: Data is acquired using PE Nelson Turbochrom. All data is imported to the Hewlett Packard Chemserver and processed using Target software

5.4 Autosampler: Dionex WPS-3000 Autosampler or equivalent.

5.5 Detectors: Ultraviolet detection: Dionex VWD-3400 Variable Wavelength UV/Vis Detector with multi-channel and multi-wavelength data collection, or equivalent.

5.6 Degasser: automated Teflon membrane vacuum degassing equipment, built-in with the Dionex Ultimate 3000 system, or equivalent.

5.7 Microsyringes: Hamilton gas tight, various sizes purchased from Supelco.

5.8 Analytical balance: capable of reading to 0.0001 g.

5.6 Membrane Filtration Apparatus using 0.2 µm, 47 mm, membrane filter; PALL Supor-200, P/N 60301, or equivalent.

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6 Reagents

NOTE: Unless otherwise noted in this section, all chemicals are stored at room temperature and labeled with an expiration date of five years from receipt. Manufacturer's labeled expiration dates, when less than five years, take precedent over all other expiration dates.

6.1 Acetonitrile: HPLC grade purchased from VWR Catalog #BJ015-4 or equivalent

6.2 Reagent Water – A Filson Water Purification System is used which provides analyte-free >16.0 megaohm deionized water on demand.

6.3 Methanol (MeOH): HPLC grade purchased from VWR Catalog #EM-MX0475-1, or equivalent.

6.4 Calcium chloride, $CaCl_2$ - Reagent grade, Fluka P/N 21098, or equivalent. Prepare an aqueous solution containing 5.0 g/L of calcium chloride. Label with an expiration date of 180 days.

6.5 1:1 Acetonitrile:$CaCl_2$ Solution: Mix 250 mL of Acetonitrile with 250 mL of $CaCl_2$ solution, expiration period of 6 months.

6.6 Surrogate Stock Solution – 1-chloro-3-nitrobenzene (1000 µg/mL) in acetonitrile, Ultra Scientific part #IST-630, or equivalent. Store at -10°C to -20°C protected from light.

6.7 Primary Stock Solution: Cerilliant Method 8330 Stock Standard P/N ERE-021, or equivalent. This standard contains all 14 of the explosive compounds listed in EPA method 8330A, all at 200 µg/mL in acetonitrile. Store at -10°C to -20°C protected from light.

6.7.1 Calibration Stock Solution (2000 µg/L): Mix 2.0 µL of Surrogate Stock and 10.0 µL of Primary Stock Solution, with 990 µL acetonitrile, prepare as needed for calibration.

6.8 Second Source Solution: Ultra Combined Stock Solution P/N NAIM-833E, or equivalent. This standard contains all 14 of the explosive compounds listed in EPA method 8330A, all at 1000 µg/mL in acetonitrile. Store at -10°C to -20°C protected from light.

6.8.1 Second Source Intermediate Stock Standard (20 µg/mL): Mix 20.0 µL of Surrogate Stock, 20.0 µL of Second Source Solution, with 960 µL acetonitrile. Store at -10°C to -20°C protected from light, label with an expiration date of 180 days.

6.8.1.1 Second Source Calibration Check Standard (100 µg/L): Fortify 1.00 mL of a 1:1 mixture of acetonitrile and $CaCl_2$ solution with 5.0 µL of Second Source Intermediate Stock Standard. Prepare daily as needed.

6.9 Additional Compounds: The compounds Nitroglycerin (NG), Pentaerythritol tetranitrate (PETN), and 3,5-Dinitroaniline (3,5-DNA), may also be analyzed by this method. The stock

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standards for these compounds are purchased as separate single component solutions:

6.9.1 NG Primary Stock Solution: Cerilliant Trinitroglycerin Standard P/N T-002, or equivalent. This standard contains NG at 1000 µg/mL in acetonitrile. Store from -10°C to -20°C protected from light.

6.9.2 NG Second Source Solution: Restek Nitroglycerin P/N 31498, or equivalent. This standard contains NG at 1000 µg/mL in methanol. Store at -10°C to -20°C protected from light.

6.9.3 PETN Primary Stock Solution: Cerilliant PETN Standard P/N P-037, or equivalent. This standard contains PETN at 1000 µg/mL in acetonitrile. Store from -10°C to -20°C protected from light.

6.9.4 PETN Second Source Solution: Restek PETN P/N 31600, or equivalent. This standard contains PETN at 1000 µg/mL in acetonitrile. Store at -10°C to -20°C protected from light.

6.9.5 3,5-DNA Primary Stock Solution: Restek 3,5-DNA P/N 31661, or equivalent. This standard contains 3,5-DNA at 1000 µg/mL in acetonitrile. Store at -10°C to -20°C protected from light.

6.9.6 3,5-DNA Second Source Solution: Restek 3,5-DNA P/N 31661, of a different manufacturer's lot from the Primary Standard (6.9.5), or equivalent. This standard contains 3,5-DNA at 1000 µg/mL in acetonitrile. Store at -10°C to -20°C protected from light.

6.9.7 Calibration Stock Solution with Additional Compounds: Adjust the Calibration Stock Solution (6.7.1) as necessary to incorporate 10.0 mg/mL of the additional compounds. As an example, a Calibration Stock Solution with the addition of Nitroglycerin would be prepared as follows: Mix 2.0 µL of Surrogate Stock, 10.0 µL of Primary Stock Solution, and 10.0 µL of Nitroglycerin Primary Stock Solution with 980 µL acetonitrile, prepare as needed for calibration.

6.9.8 Second Source Intermediate Stock Standard with Additional Compounds: Adjust the Second Source Intermediate Stock Standard (6.8.1) as needed to analyze for any or all of the additional compounds. As an example, a Second Source Intermediate Stock Standard (6.8.1) with addition of Nitroglycerin would be prepared as follows: Mix 20.0 µL of Surrogate Stock (6.6), 20.0 µL of Second Source Solution (6.8), and 20.0 µL of Nitroglycerin Second Source Stock Solution, with 940 µL acetonitrile. Store at -10°C to -20°C protected from light, label with an expiration date of 180 days.

6.10 Sand - Ottawa 20-30 mesh. Fisher Scientific, part # 523-50 (1999) or equivalent. Muffle

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furnace at 400ºC for four hours. Cool and store in a glass bottle at room temperature. Label with an expiration date of 180 days.

7 Instrument Calibration

7.1 An external standard procedure is used for the 8330 analysis. A valid calibration of all target compounds and the surrogate must be in place prior to sample analysis. The calibration must consist of at least five points for linear calibration models, and of at least six points for non-linear calibration models. The points are generated by varying the injection volume for the appropriate Calibration Stock Solution. The following table shows a typical set of injections:

Table 1

Injection Volume (µL) of Calibration Stock Solution (6.8.1 or 6.10.1)	Equivalent Concentration Based On 100 µL Sample Injection (µg/L)	Equivalent Concentration for Additional Compounds Based On 100 µL Sample Injection (mg/L)
1.0	20.0	0.100
2.5	50.0	0.250
5.0	100	0.500
12.5	250	1.25
25.0	500	2.50
50.0	1000	5.00
100	2000	10.0

7.2 With the Target Software, use the data to prepare a calibration using the external standard calibration technique. Two general calibration types are available on the target software.

7.2.1 Averaged Response Factor: This calibration model is acceptable if the averaged response factor over the calibration range is constant (20% RSD or less). If the RSD is greater than 20%, or if it does not represent the calibration data well, proceed to Section 7.2.2.

7.2.2 Prepare a calibration curve using a linear regression model. When using a linear regression calibration curve, do not include the origin or force the calibration through the origin. The linear regression calibration curve will be valid if the coefficient of determination (r^2) is 0.99 or greater.

7.2.3 If the criteria specified above in Section 7.2.1 or 7.2.2 are not met, sample analysis will not begin until corrective action is taken resulting in acceptable %RSD or coefficient of determination. The following are suggestions on types of corrective action that may be

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pursued:

7.2.3.1 If a specific calibration level is the cause of the unacceptable calibration, re-inject the standard.

7.2.3.2 A calibration level for an analyte may be removed if it does not represent the practical quantitation limit for the analyte and as long as the calibration consists of the minimum number of calibration levels specified in Section 7.1. Calibration points shall not be removed from the middle of the initial calibration without technical justification.

7.2.3.3 If the above steps do not correct the problem, prepare another calibration standard and repeat the calibration. If the new calibration is still unacceptable, instrument maintenance may be necessary.

7.3 The initial calibration must be verified by the analysis of a second source calibration check standard at the beginning of each 12-hour shift or every ten field samples, which ever is more frequent. If the response for each analyte is ±20% of the response obtained during the initial calibration then the initial calibration is considered still valid and the analyst may proceed with sample analysis. Should the standard injection not meet the ±20% criteria, the following step shall be taken:

7.3.1 Prepare a fresh second source calibration check standard, inject and analyze it. If it meets the ±20% criteria, then the initial calibration is considered valid and the analyst may proceed with sample analysis. Should this second standard injection not meet the ±20% criteria, the following step shall be taken:

7.3.2 If it is possible to identify a specific source that could inhibit a successful check standard injection, then correct and document this source, and re-inject the second source calibration check standard. If it meets the ±20% criteria, then the initial calibration is considered valid and the analyst may proceed with sample analysis. Should this second standard injection not meet the ±20% criteria, the following step shall be taken:

7.3.3 Recalibrate the instrument.

7.4 Verify the initial calibration following the analysis of a group of samples to ensure the instrument is still in control. The result shall be ±20% of the expected result. If this criterion is not met, re-inject a calibration verification standard. If the criterion is still not met, the following actions will be taken with the samples analyzed prior to the failing calibration verification standard.

7.4.1 If the bias is low, re-analyze the samples under a valid calibration.

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7.4.2 If the bias is high, and there were detections in a field sample, re-analyze that field sample.

7.4.3 If the bias was high and analytes were not detected in the field sample, data quality is not impacted. Do not re-analyze the samples. Report to the client without qualification.

8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 Initial Demonstration of Capability. Each analyst must successfully perform this demonstration before being considered proficient to analyze samples by this method. Ongoing proficiency must be established annually as specified in the QA Plan, under Technical Training,

8.2.1 Four QC check samples will be prepared:

8.2.1.1 Soil: Spike four 2.0 g samples of reagent sand with 10.0 µL of the Primary Stock Solution, and 10.0 µL of the Surrogate Stock Solution, also add 10.0 µL of the Nitroglycerin Primary Stock Solution if that compound is also needed. Take the samples through all steps of the extraction as described in SOP 09-8330S.

8.2.1.2 Water: Spike four 1000 mL samples of reagent water with 5.0 µL of the Primary Stock Solution, and 5.0 µL of the Surrogate Stock Solution, also add 5.0 µL of the Nitroglycerin Primary Stock Solution if that compound is also needed. Take the samples through all steps of the extraction as described in SOP 09-8330W.

8.2.2 Analyze samples by HPLC as described in Section 10.

8.2.3 An average recovery range of 70-130% recovery will be used as guidance to assess laboratory performance. A precision control limit of 30% RSD for all four check samples will be used as guidance to assess laboratory performance. When sufficient data is gathered, in house control limits will be used to assess laboratory performance.

8.2.4 If one or more analytes do not meet these criteria, identify and correct source of problem and report test for those analytes that initially failed.

8.3 Laboratory Method Blank. A method blank is prepared and analyzed with each extraction batch of 20 samples or less.

8.3.1 Evaluate an instrument blank (acetonitrile) to determine if the contamination is post

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extraction related.

8.3.2 If Section 8.3.1 does not reveal a problem, evaluate all the samples in the extraction batch. If there are samples without detections of the analyte in question, data was not impacted and may be reported without qualification.

8.3.3 If samples do have detections of the analyte, and the detection of the analyte in the method blank is ≤ 10% of the detection in the field sample, data may be reported as acceptable.

8.3.4 If the samples have detections, and the detection in the method blank is greater than 10% of the detection in the sample, the following steps will be taken:

8.3.4.1 If additional sample is available and the sample is still within holding time, the sample will be re-extracted.

8.3.4.2 If additional sample is not available, or the sample is past its holding time, the appropriate customer service representative will be notified, and the data will be qualified to the end user.

8.3.5 DoD requirements: If the method blank concentration is greater than or equal to ½ the reporting limit AND is greater than $^{1}/_{10}$ the sample concentration, the source of contamination must be investigated and measures taken to minimize or eliminate the problem and affected samples reanalyzed. If reanalysis is not possible, data shall be reported with a qualifying statement.

8.4 Assessing Surrogate Recovery: The surrogate concentration in samples will be 5.0 µg/L based on a 1 liter initial volume, or in the case of solid samples 5.0 mg/kg. Until sufficient data has been acquired to calculate matrix and extraction specific control charts, surrogate recovery limits will be 65% to 135%. When sufficient data has been acquired (>20 data points), determine the average and standard deviation of the data points. Control limits will be the average of ± 3 standard deviations.

8.4.1 When surrogate recovery from a sample is outside the established control limits, and the method blank and laboratory control sample is in control, the following steps shall be taken:

8.4.1.1 Check calculations and volumes used for spiking.

8.4.1.2 In this situation, sample matrix may be the cause of the out of control recovery in the field sample. Examine the sample chromatogram for other indications of matrix affect. Re-extract the sample or qualify as suspect due to sample matrix.

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8.4.2 If surrogate recovery is out of control in the method blank and laboratory control sample, the following actions shall be taken:

8.4.2.1 Check calculations and volumes used for spiking.

8.4.2.2 Re-validate the surrogate spike solution used to spike the samples. If this indicates a problem, re-evaluate recoveries based on the actual concentration of the spike solution. Additionally, dispose of the spike solution.

8.4.2.3 Check instrument performance. If the instrument is not running correctly, make the appropriate adjustments, or perform the appropriate instrument maintenance. Re-calibration may be necessary before sample analysis can resume.

8.4.2.4 If the above do not indicate a problem, and re-analysis of the blank and / or laboratory control sample do not result in acceptable surrogate recovery the following actions will be taken:

8.4.2.4.1 If surrogate recoveries for the samples are in control, no further actions will be necessary.

8.4.2.4.2 If possible, re-extract all samples with out of control surrogate recoveries.

8.4.2.4.3 If re-extraction is not possible, contact the appropriate customer service person to notify client. Qualify the samples.

8.5 Assessing Laboratory Performance - One laboratory control sample (LCS) will be extracted and analyzed with each group of 20 field samples or one per extraction batch. The laboratory control sample is prepared according to the appropriate prep SOP 09-8330W for waters, 09-8330S for soils, and analyzed as described in Section 10.0. Until sufficient data is acquired to establish in house control limits, the recoveries must be within the ranges 70 to 130 %. However, for an LCS associated with a United States Department of Defense (DoD) sample, the current approved DoD QSM recovery limits will be acceptable (see Table 5 and Table 6, in the Appendix). If a recovery for an analyte is out of control, the following actions shall be taken:

8.5.1 Check calculations and volumes used for spiking.

8.5.2 Re-validate the spike solution used to make the LCS. If this indicates a problem, re-evaluate recoveries based on the actual concentration of the spike solution. Additionally, dispose of the spike solution.

8.5.3 If the above steps do not indicate a problem, the following actions shall be taken:

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8.5.3.1 If spike recovery for the matrix spike is acceptable, sample data is acceptable and reported to the end user without qualification.

8.5.3.2 If spike recovery for the matrix spike is also unacceptable, samples will be re-extracted if additional sample is available and if the sample is still within holding time.

8.5.3.3 If additional sample is not available, contact the appropriate customer service representative. Data will be reported to the end user as suspect.

8.6 Assessing Analyte Recovery (Matrix Spike & Matrix Spike Duplicate): The laboratory must add a known concentration to a minimum of 5% of the routine samples or one sample per batch, whichever is greater. The sample selected for spiking will be done randomly. The same sample will be spiked a second time for precision data. Alternatively, when a field sample is known to have significant levels of target analyte, the field sample will be extracted in duplicate rather than a matrix spike duplicate. Spiked samples will be fortified at the same level as the laboratory control sample. Samples selected for matrix spike and matrix spike duplicate analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike or matrix spike duplicate may indicate a problem with sample composition and shall be reported to the client whose sample produced the poor recovery.

8.6.1 Until sufficient data is acquired to establish in house control limits, the recoveries must be within the ranges 70 - 130%, unless the project involved is for the Department of Defense (DoD). DoD matrix spike recoveries are evaluated under the DoD LCS Control Limits (Appendix A). If a recovery for an analyte is out of control, the following actions shall be taken:

8.6.1.1 If analyte recovery in a spiked field sample is outside laboratory control limits, and the same analyte is in control in the laboratory control sample, and the specific matrix interference is identified, sample results for that analyte in the unfortified matrix shall be reported with a qualifying statement.

8.6.1.2 If analyte recovery in a spiked field sample is outside laboratory control limits, and the same analyte is in control in the laboratory control sample, and the specific matrix interference is unknown, reanalyze the sample and matrix spike to determine matrix effect or analytical error.

8.6.2 Precision: Until sufficient data is acquired to generate precision control limits, 30% RPD will be the upper precision control limit when comparing results between the matrix spike and the matrix spike duplicate. The same precision criterion applies to results in a duplicate pair when a sample duplicate is extracted in place of a matrix spike duplicate.

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8.7 Retention Time Windows

8.7.1 Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for compound identification in this method. Retention time studies will be conducted annually or after major instrument maintenance, including the changing of a column.

8.7.2 Three injections of the second source calibration check standard are made over the course of a 72 hour period.

8.7.3 Calculate the mean and standard deviation of the three absolute retention times for each component. The width of the retention time window for each analyte and the surrogate is defined as ± 3 times the standard deviation of the mean absolute retention time.

8.7.4 Establish the center of the retention time window for each analyte and the surrogate by using the absolute retention time for each analyte and the surrogate from the calibration verification standard at the beginning of each analytical batch.

8.8 MDL studies must be performed according to SOP 99-MDL or the reference method, whichever is more frequent.

9 Sample Collection, Preservation and Handling

9.1 Containers used to collect samples must be specially cleaned 1-liter amber glass bottles. The sample containers shall have screw caps with Teflon lined septa. Plastic containers or lids may not be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic. A minimum of one liter is necessary for this analysis.

9.2 The samples must be iced or refrigerated above the freezing point of water up to 6°C from the time of collection until extraction.

9.3 All aqueous samples must be extracted within 7 days.

9.4 Solid and concentrated waste samples must be extracted within 14 days.

9.5 Sample extracts must be analyzed within 40 days of extraction. Store extracts protected from light above the freezing point of water up to 6°C.

10 Procedure

10.1 Sample Preparation: Samples must be prepared by one of the following methods prior to HPLC analysis.

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Matrix	Methods
Water	3535A SPE, 8330A Salting out
Solid	8330A Soil sonication

10.2 To achieve maximum sensitivity with this method, the extract must be 5.0 mL.

10.3 HPLC Analysis

10.3.1 The HPLC operating conditions used for the initial calibration will be used for sample analysis.

10.3.2 HPLC conditions:

10.3.2.1 Injection Volume is 100 µL.

10.3.2.2 Mobile Phase/ Column Conditions:

- Preferred Primary Column (5.2.1.1) is used isocratically with 44.0% methanol in water, and column at 32.0º C.
- Alternate Primary Column for Nitroglycerin (5.2.2) is used with a gradient as shown in Table 2, and column at 30.0º C.
- Preferred Confirmation Column (5.2.1.2) is used isocratically with 48.0% methanol in water and column at 28.0º C.

Table 2
Primary Analysis Gradient Program

Time (min)	**Flow (mL/min)**	**%H_2O**	**%MeOH**	**Curve**
Initial	1.00	90	10	*
5.0	1.00	90	10	11 (hold)
30.0	1.00	20	80	6 (linear)
40.0	1.00	20	80	11 (hold)
45.0	1.00	90	10	6 (linear)

10.3.2.3 UV detection wavelength is dependent on the compounds analyzed. Some compounds perform best at certain wavelengths as shown in Table 3. The detector and data system are capable of simultaneously collecting only two wavelengths. As Table 3 shows, if nitroglycerin is not desired, then the best pair of wavelengths is 235 and 265 nm. If nitroglycerin is desired, then 218

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and 265 is preferred.

Table 3

UV Detection Wavelengths

UV Detection Wavelength	Primary Compounds for Wavelength	Secondary Compounds for Wavelength
218 nm	NG, PETN	HMX, RDX, 1,3,5-TNB, 1.3-DNB, Tetryl, NB, 2,4,6-TNT, 4-Am-2,6-DNT, 2-Am-4,6-DNT,
235 nm	HMX, RDX, 1,3,5-TNB, 1.3-DNB, Tetryl, 2,4,6-TNT, 4-Am-2,6-DNT, 2-Am-4,6-DNT, 2,6-DNT, 2,4-DNT, 3,5-DNA	NB
265 nm	NB, 2-NT, 4-NT, 3-NT, Surrogate	1,3-DNB, Tetryl, 2,4,6-TNT, 4-Am-2,6-DNT, 2-Am-4,6-DNT, 2,6-DNT, 2,4-DNT, 3,5-DNA

10.3.3 Qualitative Analysis: As previously discussed, the UV detector will be the primary detector used for identification and quantitation of target compounds. Primary analysis, using either of the primary columns, will be used for tentative identification and all quantitation of target compounds. Tentative identification of an analyte occurs when a peak from a sample extract fall within the retention time window of a compound of interest.

Confirmation Techniques:

10.3.4 Confirmation: All positive detections will be confirmed by analysis on a dissimilar column. The confirmation column will provide good qualitative confirmation if the analyte is found in the retention time windows on both the primary and the confirmation columns. Quantitation may be adversely impacted due to co-elution of analytes on the confirmation column. Verify the confirmation by comparing the result for an analyte for the primary column to the result for that analyte on the confirmation column, RPD should be less than or equal to 40%.

10.3.4.1 If RPD is less than or equal to 40%, then the primary result confirms, and the higher of the two values should be reported, unless chromatographic issues such as coelution preclude this. Any such issues must be documented in the data if the higher value is not reported.

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10.3.4.2 If RPD is greater than 40%, then the higher of the two values shall be reported with a comment (comment acts as the J-flag for DoD samples).

10.3.4.3 If a result from the primary column is not confirmed by the confirmation column, since the analyte peak detected on the primary column is not found in the retention window for that analyte on the confirmation column, then the result will not be reported, except for DoD samples, where the primary result will be reported with a qualifying comment (comment acts as the Q-flag for DoD samples.)

10.3.5 Quantitative Analysis: As described in Section 7, all compounds will have a valid calibration curve. All calculations will be performed with the Target software. Generally, the Target software will be capable of automatic integration for all analyte and the surrogate peaks. This is preferred. If it becomes necessary to perform manual integrations of the data, then these manual integrations will adhere to SOP 99-Integration, and they will be properly noted in the data.

10.3.6 Linear Range: If the peak area for any analyte exceeds the linear range of the system, either by exceeding the calibration range, or by exceeding the maximum detector response, then dilute the extract and reanalyze.

11 Calculations

11.1 LCS Recovery:

% Recovery = (Cm/Cn)x100

where: Cm = measured concentration of LCS

Cn = spiking concentration

11.2 Spike Recovery:

% Recovery = [(Cs-Cu)/Cn]x100

where: Cs = measured concentration of spiked sample aliquot

Cu = measured concentration of unspiked sample aliquot

Cn = spiking concentration

11.3 Precision (RPD):

% Recovery = [(Cs-Cu)/Cn]x100

where: Cs = measured concentration of spiked sample aliquot

Cu = measured concentration of unspiked sample aliquot

Cn = spiking concentration

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12 Reporting Results

12.1 All raw data used for reporting results must be dated and initialed by the qualified laboratory personnel performing first and second review.

12.2 Horizon LIMS results are reported to three significant figures but limited to the number of decimal places in the reporting limit for the individual compound or analyte.

12.3 When entering data into the Horizon LIMS do not round off results: Horizon will automatically round off to 3 significant figures after all internal calculations are completed.

12.4 Report the actual result in the Horizon LIMS. The reporting limit is at or above the lowest calibration standard.

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

16 Troubleshooting

16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the instrumentation used in this method.

16.2 The Chromeleon software that controls the instrument used for this procedure provides a fully searchable database of all instrument parameters, including peak areas, pump pressure, column temperature, and more. This database has trending capability. This routine recording of

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instrument parameters is an excellent reference for properly maintaining the instrument as well as an invaluable trouble-shooting tool.

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Appendix A

DoD QSM Control Limits

Based on QSM Revision 3, January 2006

TABLE 4

DoD QSM LCS Control Limits for 8330 Water Matrix[a]

Compound	Lower Control Limit (%)	Upper Control Limit (%)	Lower ME Limit (%)	Upper ME Limit (%)
HMX	80	115	75	120
RDX	50	160	35	180
1,3,5-TNB	65	140	50	150
1,3-DNB	45	160	30	175
Tetryl	20	175	10	200
2,4,6-TNT	50	145	35	160
NB	50	140	35	155
4-Am-DNT	55	155	40	170
2-Am-DNT	50	155	35	170
2,4-DNT	60	135	50	145
2,6-DNT	60	135	50	150
2-NT	45	135	30	150
3-NT	50	130	35	145
4-NT	50	130	35	145

TABLE 5

DoD QSM LCS Control Limits for 8330 Solid Matrix[a]

Compound	Lower Control Limit (%)	Upper Control Limit (%)	Lower ME Limit (%)	Upper ME Limit (%)
HMX	75	125	65	135
RDX	70	135	65	145
1,3,5-TNB	75	125	65	135
1,3-DNB	80	125	70	135
Tetryl	20	175	10	200
2,4,6-TNT	50	145	35	160
NB	50	140	35	155
4-Am-DNT	55	155	40	170
2-Am-DNT	50	155	35	170
2,4-DNT	60	135	50	145
2,6-DNT	60	135	50	150
2-NT	45	135	30	150
3-NT	50	130	35	145
4-NT	50	130	35	145

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UNCONTROLLED DOCUMENT

Appendix B Sample Logbook Page

Work continued from page:____

HPLC-1 (Dionex) LOGBOOK

Work continued to page:____

Sequence: ______ Batch: ______ Method: ______ Date: ______

Seq. No.	Sample ID	Inj. Dilution	Analyst	QC	Standard	Comments

Reagents:

Add'l. Comments:

Revision 4/07

Page #: _____

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SOP Change History Sheet

Section No.	Section	Reason for Change
Revision 3: 10/31/05		
10.3.4 – 10.3.5	Procedure	USACE audit response
Appendix A	Logbook Sample Page	USACE audit response
5.2	Apparatus and Materials	Removed reference to column guard; updated manufacturer information
6.5	Reagents	Added Isopropyl Alcohol to list
10.3.3	Procedure	Removed reference to guard column and expanded HPLC table
10.3.6	Procedure	Added gradient parameters table
Figure 1		Removed
Revision 4: 03/13/2006		
Revisions to spelling, grammar and format made throughout document.		
1.1	Scope and Application	Deleted MDL reference to HPLC; replaced MDL folder with MDL book
1.2	Scope and Application	Added Octogen and Hexogen references
1.6	Scope and Application	Added verbiage concerning project requirements
4.1	Safety	Added MSDS availability verbiage
5.1.1	Apparatus and Materials	Deleted “consistent flow” from HPLC1 description
6.3	Reagents	Revised reagent water to reflect definition and ALSI in-house production

SOP Change History Sheet (continued)

Section No.	Section	Reason for Change

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6.6	Reagents	Added vendor information
6.7.2, 6.8.1, 6.8.2	Reagents	Revised holding parameters
6.7.3, 6.8.3.1, 6.8.3.2	Reagents	Revised preparation volumes and holding parameters
6.10, 6.11	Reagents	Added sections for sand and spike solution
6.12	Reagents	Revised reference to table, deleted spike solution
7.2	Instrument Calibration	Added references to linear regression
7.2.3.2	Instrument Calibration	Added minimum calibration requirements
7.3	Instrument Calibration	Revised verification of second source calibration
7.4	Instrument Calibration	Added 7.4.1-7.4.3 for clarification
8.2	Quality Control	Added verbiage about ongoing proficiency; added sections describing 4 QC check samples preparation and precision control limits
8.3	Quality Control	Removed verbiage about method blank/MDL association
8.3.3, 8.3.4	Quality Control	Revised analyte detection percentage in the method blank
8.4	Quality Control	Deleted surrogate recovery assessment concentration basis
8.4.2.2, 8.5.2	Quality Control	Revised wording about spike solution degradation
8.5	Quality Control	Added/revised laboratory performance assessment

SOP Change History Sheet (continued)

Section No.	Section	Reason for Change
8.6	Quality Control	Added MS/MSD rotation

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8.6.1.1	Quality Control	Added reference to "spiked" field sample and identification of specific matrix interference
8.6.1.2	Quality Control	Added verbiage for corrective action
8.6.2	Quality Control	Revised RPD from "50" to "30" %
8.7	Quality Control	Added section on Retention Time Windows
8.8	Quality Control	Added section referencing MDL; Removed section concerning accessing an internal standard
9.1.1	Sample Collection…	Added "amber" to further describe container
10.3.2	Procedure	Revised section removing calibration verification reference and adding table references
10.3.5	Procedure	Deleted previous verbiage about unnecessary confirmation; added verbiage about positive detection confirmations
10.3.6.1	Procedure	Added Table 4 references
10.3.11	Procedure	Added section addressing automatic and manual integration
11.1	Reporting Results	Added section for reporting raw data results
16	Troubleshooting	Added section
A	Appendix	Deleted Table 1 and added Tables 5 & 6
B	Appendix	Added Sample Logbook page

SOP Change History Sheet (continued)

Section No.	**Section**	**Reason for Change**

Revision 5: 11/16/07

Spelling, grammar, and formatting changes may have been made throughout SOP for clarity, correctness, and site conformity.

1.2	Scope and Application	Added Nitroglycerin

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4.2, 4.3	Safety	Added section
5	Apparatus and Material	Update numerous manufacturer and part numbers
6	Reagents and	Added NOTE, numerous updates to manufacturer part numbers, and reagent formulations
7.1	Instrument Calibration	Added Table
8.3.5	Quality Control	Added section
8.4	Quality Control	Added soil criterion
8.6.1, 8.6.2	Quality Control	Removed equations
10	Procedure	Numerous revisions made throughout section
11	Calculations	Added calculations from Section 8
Appendix B		Update to current logbook page

Revision 6: 01/29/08

8.6.1	Quality Control	Added DoD verbiage per DoD audit
8.7.1	Quality Control	Updated procedure per DoD audit
10.3.6	Procedure	Updated procedure per DoD audit
16.2	Troubleshooting	Added section per DoD audit

SOP Change History Sheet (continued)

Section No.	**Section**	**Reason for Change**
Revision 7: 4/14/08		
1.1	Scope and Application	Updated reference method
1.2	Scope and Application	Updated analyte list
6.9	Reagents	Added new analytes

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6.10	Reagents	Removed section
7.3, 7.4	Instrument Calibration	Updated criteria from 15% to 20%
10.3.2.3	Procedure	Added analytes to Table 3

SOP Concurrence Form

for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

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Document Title: Waste Disposal

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICE, INC. (ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

_____ _____
Helen MacMinn, Date
Quality Assurance Manager

_____ _____
Anna Milliken, Date
Operations Manager

Annual Review:

_____ _____
Reviewed By Date Reviewed

_____ _____
Approved By Date Approved

_____ _____
Reviewed By Date Reviewed

_____ _____
Approved By Date Approved

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Annual Review (continued):

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
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1 Scope and Application

1.1 This standard operating procedure is to be used as a guide for the control and disposal of hazardous and general laboratory waste. Waste streams are divided and segregated into different streams to facilitate disposal in an environmental friendly manner in accordance with all federal, state, and local applicable and relevant regulations.

1.2 Waste generated by ALSI can be classified into two specific categories, hazardous and non hazardous waste. It is important to determine the type of waste that needs to be handled so that it can be properly characterized and disposed of safely. When there is any doubt regarding the disposal of a material, the laboratory manager shall be contacted for guidance. Under no circumstances shall an unidentified waste be disposed of in the municipal trash unless it has been properly characterized.

1.3 It is ALSI policy to properly handle waste generated by our facility in an environmentally responsible manner. To assist ALSI, Tier, Inc of Downingtown, PA, serves as ALSI's consultant and disposal company for waste generated at our facility. Tier provides guidance and assistance on the storage, packaging, and transportation of waste from the laboratory facility.

1.4 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

1.5 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

2.1 Generally, wastes generated in each of the analytical sections are stored in satellite containers or in temporary storage areas. Each container shall be labeled with its contents. When satellite containers are full, they are emptied into the proper drums in the waste storage area. All solvent wastes are stored in the outside chemical cabinet. Once a sufficient amount of waste material has accumulated, Tier is contacted to remove the waste. Tier is responsible for proper drum labeling and packaging, generating the waste manifest, and the removal of the waste from the facility.

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3 Interferences

3.1 Not Applicable.

4 Safety

4.1 The toxicity or carcinogenicity of each sample, reagent, or material that needs to be disposed of may not be precisely known. As such, each material shall be handled cautiously and with care.

4.2 Chemical reagents that require disposal that are in labeled jars or containers most likely can be traced back to a Material Safety Data Sheet (MSDS). The MSDS shall be consulted for guidance when handling and disposing of all materials. ALSI maintains MSDS on all chemicals used. MSDS are available to all staff and located in the QA office.

4.3 For sample containers that have the laboratory identification number present, the Laboratory Information Management System (LIMS) can be consulted for information regarding the specific contents of the sample. Be aware that the sample may not have been tested for all of the parameters that could be present. Reasonable caution must be used when handling an unknown.

4.4 Appropriate PPE must be used when disposing of waste. At a minimum, this shall include a fully-buttoned lab coat, safety glasses, and PVC gloves. A respirator shall also be used when dealing with the disposal of solvents or solid waste material containing high solvent concentrations. Personnel using respirators shall be trained in and follow all applicable OSHA guidelines.

5 Apparatus and Materials

5.1 Gloves- usually latex gloves are sufficient when handling general laboratory waste. It is recommended that the gloves be doubled in the event that the outer glove develops a puncture or rip. Since several of the materials being handled may be unknown, doubling the gloves will provide an extra layer of protection if the material begins to degrade the glove. If degradation is noted, immediately remove both gloves and flush hands under water for 15 minutes. Upgrade the type of glove to a nitrile or silver shield as necessary. A variety of gloves is available from VWR Scientific. The part number is dependent on the type and size of glove ordered.

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5.2 Safety Glasses- Standard grade safety glasses shall always be worn when handling laboratory waste. Safety glasses can be purchased from VWR Scientific catalog #47746-188, or equivalent.

5.3 Drums- 55 gallon DOT approved steel drum, open top sealed with a drum ring. Drums are obtained from SAIC.

5.4 Drums – 55 gallon DOT approved steel drum, closed top with two bunghole openings. Drums are obtained from SAIC.

5.5 Respirator - A half face respirator is required for disposing of solvent waste. The respirator shall be equipped with organic vapor cartridges. NOTE: Only laboratory personnel that have been trained in OSHA guidelines and that have been fit tested are to use a respirator. Contact the laboratory manager for fit test information and contact personnel that are fit tested when solvent disposal is required. Respirators and cartridges can be purchased from VWR Scientific catalog #56222-944 and #56222-955, or equivalent.

5.6 Labels - A label shall be affixed to each drum describing its contents. Labels can be obtained from Tier, Inc.

5.7 Spill control material – includes spill pads, kitty litter, and adsorbent socks. Spill control materials are purchased and maintained by the ALSI safety committee. They are available from VWR Scientific.

6 Reagents

6.1 Not Applicable.

7 Instrument Calibration

7.1 Not Applicable.

8 Quality Control

8.1 It is important to keep the waste separated as indicated in the following sections. Separation of waste is required so that the waste can be properly disposed. In the event that the waste streams are mixed, the laboratory manager shall be immediately notified

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of the occurrence. The mixed waste drum will be relabeled to identify the most hazardous constituents and the drum will be manifested and removed from the laboratory under the new profile.

8.2 Tier serves as ALSI's primary consultant for waste removal. All of the typical waste streams have been classified, sampled, and profiled by Tier. Be sure to follow the guidance outlined in the procedure below so that waste is properly stored and disposed.

8.3 Any questionable classification of waste shall be brought to the attention of the laboratory manager. The laboratory manager will make the decision regarding how to classify the waste. When additional consideration is required, Tier will be consulted.

8.4 In some instances, it may be necessary to collect a sample from waste drums being removed from the site. Samples cannot be collected from the solid drums because the waste cannot be mixed sufficiently to obtain a representative sample. Therefore, samples can only be collected from the solvent waste drums. It is advisable to collect approximately 50 mL of liquid from each drum. The samples shall be archived for approximately one month and shall be analyzed as required in the event of a discrepancy between the manifest information and the actual drum contents.

8.5 All drums and waste storage containers shall be inspected for integrity before placing any waste in said container. Containers with questionable integrity (excessive rust, poor/warped lid, etc) shall not be used.

8.6 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

9 Sample Collection

9.1 When a sample from a liquid drum is required, the following procedure shall be followed

9.1.1 Carefully loosen the bunghole cap using the drum wrench. Allow the pressure and vapors to slowly escape before removing the cap completely. Avoid inhaling the vapors escaping from the drum. Carefully insert the pipette below the liquid surface and pipette approximately 40 to 50 mL of sample into the sample container. Secure the lid tightly on the container.

9.1.2 Label the container with the sample identification, drum identification, date, time and your initials. If necessary, mark an identification number on the drum with a grease pencil, such as "ALSICL01". Be sure the drums are uniquely identified and match the sample identification on the chain of custody. The grease pencil markings will be on the drum until

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ultimate, final disposal and will assist in identifying the drum at the treatment/disposal facility.

10 Procedure

Waste at ALSI have been categorized and characterized by Tier through on-site inspections and random sample collections. Generally, waste can be broken down into hazardous and non-hazardous solids and liquids. The following sections describe disposal procedure for waste encountered at ALSI.

10.1 Non-Hazardous General Waste

10.1.1 General waste accumulated throughout the laboratory is classified as non-hazardous waste and can be disposed of in the municipal trash containers. These wastes include, wrappers, packing, lunchroom waste, paper, used sample containers, used glassware, used personal protective equipment (PPE), and like items. Be sure that all sample containers disposed do not contain sample remains. PPE that may contain high levels of a hazardous material shall be segregated and the laboratory manager consulted for guidance.

10.2 Non-Hazardous Solid Waste

10.2.1 This category of waste has been profiled by Tier as "Waste filter paper and media (non-regulated), waste Approval #Q200000322A10. This waste stream consists of organic prep laboratory generated waste such as sodium sulfate cakes, filter paper and miscellaneous prep waste. This waste profile also includes the majority of used soil sample remains. The following steps shall be followed when disposing of these wastes.

10.2.2 Organic prep laboratory wastes are accumulated in satellite containers in the prep laboratory. When the container is full, transfer its contents to the waste disposal room. Identify the drum labeled "solid waste" and transfer the waste into that drum.

10.2.3 Soil samples are transferred to the waste disposal room by Sample Receiving. Soil samples are disposed of by removing the lid from the container, scraping the sample remains from the container, and transferring them to the "solid waste" drum. The sample container lid and jar can be disposed of in the municipal trash. Do not dispose of sludges in this drum. The waste classified under this profile must not contain freestanding liquids. Do not dump unopened containers or full containers

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into these drums. This creates a "container in a container" situation and hence is considered a mixed waste stream.

10.2.4 If there is no drum present or drums are full, obtain an empty, open-top drum from the drum storage area. Write on the drum lid with a grease pencil "solid waste"

10.2.5 Replace the lid on the drum and ensure that the waste storage room is locked when you leave.

10.3 Non-Hazardous Liquid Waste

10.3.1 Aqueous wastes generated in the laboratory are broken down into two waste streams - sanitary wastewater and laboratory wastewater. Since the ALSI facility is constructed to be a laboratory facility, the drains in the building have been constructed to separate sanitary waste from laboratory wastewater. All of the sanitary waste liquids (toilets and kitchen/ break area) are plumbed directly into the sanitary sewer connection. This waste liquid is not treated or controlled by ALSI. All of the laboratory sinks in the work areas in the laboratory are directed to an on-site waste treatment room. ALSI has a letter of agreement with the local wastewater treatment plant to discharge this waste stream into the sanitary sewer after pH adjustment to no lower than 6.0 and no higher than 9.0 pH units. If the treatment plant experiences any difficulties with the treatment process, ALSI and the treatment plant will work together to implement the necessary controls in our laboratory wastewater discharge. The laboratory manager and the maintenance staff at ALSI are responsible for the operation and maintenance of our treatment system.

10.3.2 The vast majority of aqueous samples received in the laboratory are from drinking water sources, ground water wells, and wastewater discharges from industries. Therefore, once these samples have been analyzed and it is time to dispose of them, they can be discharged to the treatment room without impacting our in-house treatment unit and the local treatment plant's process. The following procedure shall be used for disposing of waste aqueous samples.

10.3.3 The sample receiving department is responsible for identifying aqueous sample remains for disposal. Any aqueous sample that is a neat product, oil, sludge, or sample that has been analyzed and contains high levels of hazardous constituents shall be separated for disposal by one of the other methods outlined below. If you are not sure how to handle a sample, contact the laboratory manager for guidance.

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10.3.4 Once these samples have been identified, they shall be discharged directly into the tank in the treatment room.

10.3.5 Begin by opening the sample bottle and slowly pour the contents into the tank. Some samples have been preserved with acid and will exhibit low pH. Use caution when handling these liquids.

10.3.6 Continue until all aqueous samples have been discharged.

10.3.7 Discard the sample bottle and lid in the municipal trash.

10.4 PCB Waste

10.4.1 This waste stream has been identified as "PCB Contaminated Oils", Approval #WIP440581, and shall be handled with great care. The waste storage area will contain open-top drums identified as PCB oils.

10.4.2 PCB oils are those wastes that are oily and suspected of containing PCBs, samples sent to the laboratory specifically for PCB analysis that are known to contain high levels of PCBs from laboratory data, and GC laboratory generated PCB waste.

10.4.3 The waste material is transferred directly to the drum without being opened. This drum has been specifically profiled for this type of waste.

10.4.4 Place a layer of kitty litter in the drum to adsorb any liquids in the event of container breakage.

10.4.5 Secure the lid in place once disposal is completed.

10.4.6 In the event that a new drum is needed, follow the guidance provided above to start a new drum. Be sure to lock the waste storage room after you leave.

10.5 Autosampler Vials

10.5.1 ALSI and Tier have identified a waste stream commonly called "autosampler vials". This waste stream is comprised of unused sample extracts that remain from gas chromatography analysis and expired standard solutions.

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10.5.2 Used autosampler vials are accumulated in satellite containers located throughout the chromatography laboratories. After the sample is injected, the unused portion of the extract is transferred to the satellite container.

10.5.3 When the satellite container is full, its contents are transferred to the open-top drum located in the drum storage area labeled as “autosampler vials”. Carefully remove the container lid and avoid breathing vapors that escape from the container.

10.5.4 Transfer the contents of the satellite container directly into the autosampler vial drum.

10.5.5 Place a layer of kitty litter over the vials to adsorb any potential leakage.

10.5.6 Secure the lid.

10.5.7 If a new drum is required, follow the guidance above to start a new drum.

10.6 Solvent Waste

10.6.1 There are two type of solvent waste streams identified, chlorinated solvents, Methylene chloride waste, Approval #Q20000322A11, and non-chlorinated solvents, Approval #Q20000322A08. All liquid wastes are stored outside in the chemical cabinets. It is not permissible to store liquid waste in the area designated for solid waste. Inside the chemical cabinet, two drums will be present and marked as chlorinated and non-chlorinated waste. Liquid wastes are stored in closed top drums with two bungholes. Do not use the open top style of drum.

10.6.2 Solvent wastes are generated in several areas of the laboratory and are stored in safe areas throughout the laboratory in DOT approved containers. Once sufficient quantities of waste solvents are generated, they are transferred to the outside storage area.

10.6.3 When satellite containers require empting, contact the laboratory manager.

10.6.4 The laboratory manager shall designate a staff member who is permitted to wear a respirator to transfer the solvents to the solvent waste storage area.

10.6.5 While wearing a respirator, the waste solvent disposal designee shall carefully loosen the bunghole cap using the drum wrench. Once the vapor pressure is reduced, the cap can be removed.

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10.6.6 A grounding clip is attached to the container to be emptied, the receiving drum, and a ground. This procedure reduces the chance of a static spark, which may result in the ignition of the solvent.

10.6.7 Replace the drum cap tightly once all of the satellite containers have been emptied.

10.6.8 The following general guidance is used when disposing of solvent waste.

10.6.9 All chlorinated solvents, such as methylene chloride and freon, are transferred to the chlorinated solvent drum. All non-chlorinated solvents, such as hexane, acetone, toluene, etc are transferred to the non-chlorinated drum. The non-chlorinated drum can also be used to dispose of aqueous sample remains that are not suitable for conventional disposal. These items include, sludge samples, oil samples, paint, inks, etc. As always, if there is any question regarding the fate of a material, contact the laboratory manager.

10.7 Miscellaneous Items and Neat Chemicals and Reagents

10.7.1 From time to time there will be items that require special handling. These items include but are not limited to mercury waste, such as broken thermometers, expired pure chemicals, neat reagents, and material specifically received as hazardous materials. When these items are identified for discard, the laboratory manger shall be contacted. The laboratory manager shall contact Tier for advice and disposal information. These materials will be lab packed and disposed of by Tier in a responsible manner.

10.8 Waste Removal

10.8.1 Once a sufficient amount of waste has been accumulated, Tier is contacted by the laboratory manager to schedule a waste pick-up. Waste is removed from ALSI's laboratory facility approximately every 90 days. The numbers and types of drums to be removed are relayed to Tier.

10.8.2 When Tier arrives to pick-up waste, the laboratory manager shall be contacted.

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10.8.3 Tier and the laboratory manger will conduct an inspection of all containers that will be removed. The containers are inspected for leakage and premature ware.

10.8.4 All drum lids are secured in place and each drum is labeled by Tier. Each drum label contains the generator information, address, phone number, EPA generator ID number, the waste manifest number, approval number, and a description of the waste. Tier is responsible for generating the labels and supplying them to ALSI.

10.8.5 If necessary, the drums are placarded with hazard information. Tier is responsible for applying the necessary labels to each drum.

10.8.6 If solvent wastes are being removed, it is recommended that a sample be collected. Follow the guidance provided in the sample collection section of this SOP.

10.8.7 Tier is responsible for marking the drum lid with relevant manifest information using a grease pencil. Be sure each drum has been properly marked with the necessary information.

10.8.8 As the drums are loaded onto the truck, Tier and the ALSI will begin working on the hazardous waste manifest. Tier is responsible for preparing the manifest. All waste removed from ALSI must be documented on a manifest. The following shall be completed and reviewed on the manifest:

10.8.9 List the number and type of containers being removed.

10.8.10 Estimate the total weight of the containers and record this weight on the waste manifest.

10.8.11 Check that the identification of the containers being removed matches that of the manifest.

10.8.12 Double check the number of drums being removed against the quantity listed on the manifest.

10.8.13 Be sure that the truck driver and Tier are in agreement with all items listed on the manifest.

10.8.14 Once the manifest is completed, the laboratory manger will sign the manifest on behalf of ALSI. The transporter also needs to sign the waste

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manifest. No other person is designated to sign the manifest unless specifically designated by the laboratory manager.

10.8.15 The laboratory manager will receive a copy of the manifest.

10.8.16 Tier shall take the remaining copies to be forwarded to the state, and the disposal facilities.

10.8.17 Secure the waste storage areas and label new drums to begin the next accumulation cycle. Secure and lock the outside storage cabinet.

11 Documentation and Record Keeping

11.1 Tier will be responsible for forwarding a copy of the manifest to the state for their records.

11.2 Copies of the manifest and land ban documents (if required) are maintained by the laboratory manager.

11.3 Shortly after a disposal event occurs, the disposal facility will send a copy of the manifest indicating the acceptance of the material. This information is filed and maintained by the laboratory manager.

11.4 After the material has been accepted, the sample collected from the solvent drums can be disposed. If a discrepancy exists, the laboratory manager at his discretion will have the samples analyzed as required.

11.5 Any conflicts or rejection of waste due to the material being off the profile specifications will be worked out with the assistance of Tier.

12 Calculations

12.1 Not applicable.

13 Reporting Results

13.1 Not applicable.

14 Pollution Prevention

14.1 Not applicable.

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15 Definitions

15.1 Not applicable.

16 Troubleshooting

16.1 Not applicable.

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SOP Change History Summary

Section #	Section	Reason for Change
Revision 1: 11/02/2006		
1.5	Scope and Application	Added project requirements criteria statement
2.1	Summary of Method	Revised "liquid" to read "solvent"
4.2	Safety	Added MSDS availability
4.4	Safety	Added appropriate PPE
5	Apparatus and Materials	Added additional vendor information throughout; removed specifics on grease pencils and sampling equipment
5.3, 5.4	Apparatus and Materials	Replaced "manager" with "SAIC" for obtaining additional drums
5.5	Apparatus and Materials	Added requirement for OSHA guideline training
8.6	Quality Control	Added reference to QA Plan
9.1	Sample Collection	Removed requirement for Tier personnel to be present at sampling
10.2.2	Procedure	Added detailing for transferring solid waste
10.2.3	Procedure	Deleted reference to sample receiving performing transfer of samples
10.3.2, 10.3.4, 10.3.5, 10.3.6	Procedure	Revised discharge location from "any laboratory sink" to "the treatment room"
10.4	Procedure	Revisions made throughout section on disposal of PCB waste
12-16	Calculations, Reporting Results, Pollution Prevention, Definitions, Troubleshooting, SOP Change History Summary	Added all sections for site-wide SOP conformance

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SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

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Document Title: Standard Operating Procedure for Sample Receipt/Sample Handling

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICE, INC. (ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

_____ _____
Helen MacMinn, Date
Quality Assurance Manager

_____ _____
Anna Milliken, Date
Operations Manager

_____ _____
Steve Smith, Date
Validator

Annual Review:

_____ _____
Reviewed By Date Reviewed

_____ _____
Approved By Date Approved

_____ _____
Reviewed By Date Reviewed

_____ _____
Approved By Date Approved

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Annual Review (continued):

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

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1 Scope and Application

1.1 The purpose of this Standard Operating Procedure (SOP) is to demonstrate the procedures for receiving and handling of all samples coming into the laboratory.

1.2 This document states the laboratory's policies and procedures established in order to meet the requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

1.3 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

2.1 Samples are received from a variety of sources including ALSI field services personnel, walk-in clients, client's field personnel, and shipping carriers. Upon receipt of samples, sample receiving personnel complete the appropriate documentation. Sample receiving personnel also check for any non-conformances regarding the condition of the samples upon receipt.

3 Interferences

3.1 Not applicable.

4 Safety

4.1 ALSI maintains material safety data sheets (MSDS) on all chemicals used in this procedure. ALSI recommends that all individuals performing this SOP familiarize themselves with the MSDSs associated with the procedure prior to SOP performance. MSDS are available to all staff and are located in hard copy in the QA reference library and electronically on the ALSI server in the MSDS folder.

4.2 Incoming samples may contain a variety of unknown and potentially serious safety hazards associated with both the samples themselves and the preservatives added to them and as such, shall be treated with the utmost caution.

4.3 The minimum personal protective equipment requirements are safety glasses, a fully buttoned lab coat, and chemical resistant gloves.

4.4 Incoming coolers will be opened in or next to a ventilation hood when sample conditions or accompanying information dictate.

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5 Apparatus and Materials

5.1 pH paper (1-6 range), purchased from VWR catalog #66777-027, or equivalent

5.2 pH paper (9-14 range), purchased from VWR catalog #34175-499, or equivalent.

5.3 5-3/4" Pasteur pipettes, purchased from VWR catalog #14672-200, or equivalent.

5.4 Potassium iodide starch paper purchased from VWR catalog #60799-008, or equivalent.

5.5 Total chlorine paper, purchased from Fisher Scientific catalog #3108T34, or equivalent.

5.6 Thermometers, capable of measuring to 1°C in the range which includes -10°C to 40°C, purchased from VWR catalog #61222-504, or equivalent.

5.7 Calibrated infrared temperature gun, capable of measuring to 1°C; HB Infrared Thermometer 900LS, purchased from VWR, or equivalent.

5.8 Refrigerators. Each refrigerator used for storage of samples is maintained above the freezing point of water up to 6ºC. Temperatures are monitored 7 days a week and are recorded in the temperature logbook after the correction factor is applied. The refrigerators include:

5.8.1 Walk-in Refrigerator by AmeriKooler, Inc. with a Winland Electronics, Inc. temperature alarm located on upper level.

5.8.2 Walk-in Refrigerator by Bally, Serial #DX9101996-01 located on lower level.

5.8.3 Walk-in Refrigerator by Harford, Model DL3676W487-V located on upper level

5.8.4 Refrigerator #7 - Gibson Market Master located in the GC lab.

5.8.5 Refrigerator #10 - Gibson Market Master located in the GCMS lab.

5.8.6 Refrigerator #18 – Admiral. (Used for microbiology samples only.)

5.8.7 Refrigerator #26 – Kenmore located in the GC/MS lab.

5.8.8 Refrigerator #27 – General Electric. (Used for holding volatile samples in

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Sample Receiving before distribution to departments.)

6 Reagents

6.1 Not Applicable.

7 Instrument Calibration

7.1 Not Applicable.

8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 It is the responsibility of Sample Receiving personnel to check for and document any non-conformances regarding the condition of the samples upon receipt. These non-conformances may include but not be limited to, incorrect containers, preservatives, or packaging, unacceptable sample temperatures, expired holding times, and incomplete or inaccurate documentation. These non-conformances shall be recorded on the COC and communicated to the appropriate customer Service Representative so that the client can be notified, or the client may be contacted directly by receiving personnel. Inform Customer Services of any such communications. Any non-conformances associated with sample preservation shall also be recorded in the sample preservation logbook (Appendix A).

8.3 ALSI reserves the right to reject a sample upon receipt in the laboratory if any of the following conditions occur:

8.3.1 The sample is not properly identified on the sample label and/or the Chain-of-custody form.

8.3.2 The sample has exceeded the holding time for the requested analysis.

8.3.3 The incorrect preservative was used during sample collection.

8.3.4 Incorrect sampling protocols were used during sampling.

8.3.5 Improper sample container was used.

8.3.6 Insufficient sample is present to perform the requested analysis.

8.3.7 Improper storage or transport of sample has occurred prior to receipt.

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8.3.8 Excessive amount of sample has been collected or other conditions exist which would make disposal difficult.

8.3.9 Excessive air bubbles are present in samples requiring zero headspace.

8.4 An Initial Demonstration of Capability shall be documented as follows:

8.4.1 Completion of the "New Employee Orientation: Sample Receiving Training Checklist: Checking Sample Preservatives." See Appendix F.

8.4.2 Completion of the "New Employee Orientation: Sample Receiving Training Checklist: Measuring Cooler Temperatures." See Appendix F.

8.4.3 Completion of the "New Employee Orientation: Sample Receiving Training Checklist: Receiving Samples." See Appendix F.

8.4.4 Reading, understanding and acceptance of the contents of this SOP shall be documented through the signing of the 19-Rec/Han Concurrence form.

8.4.5 Corrective action for DOC failure:

8.4.5.1 Repeat training shall be performed until all forms are successfully completed.

8.4.5.2 It shall be at the discretion of supervisory staff and management to determine at what point repeat training is no longer applicable and reassignment or company termination is appropriate.

8.5 Ongoing proficiency on an annual basis, as specified in the QA Plan, Technical Training, does not apply to this procedure.

9 Sample Collection, Preservation and Handling

9.1 Not applicable.

10 Procedure

10.1 The Sample Receiving area is staffed Monday through Friday from 08:00 to 22:00. Samples are received from a variety of sources including ALSI field services personnel, walk-in clients, client's field personnel, and shipping carriers.

10.1.1 Samples received from a carrier (Fed-Ex, UPS, etc.) shall be accepted

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through a signature by a laboratory employee upon receipt at the facility and taken to the Sample Receiving area.

10.1.2 ALSI field personnel shall deliver all samples to the Sample Receiving area.

10.1.3 Walk-in clients and other client's field personnel shall be directed to the Sample Receiving area to deliver their samples.

10.2 All samples shall be accompanied by a Chain of Custody (Appendix C). All information required in the gray shaded area of the ALSI Chain shall be filled in by the client/sampler. If the sample is accompanied by a Chain of Custody from another source, the following information shall be included: client information (name, address, contact person, and phone number), sample(s) description, date/time sampled, analyses requested, date/time results required, PWSID information (if applicable), and any special deliverable or handling instructions (if applicable). The Chain of Custody shall also be signed by each individual having possession of the samples since collection (both when they receive the sample and when they relinquish it). A representative of the sample receiving department shall sign the Chain of Custody when the samples are received at the laboratory. If a walk-in customer does not have a Chain of Custody, one must be completed before samples will be accepted.

10.2.1 If a Chain of Custody references microbiological samples that require immediate attention due to holding time restrictions, the Chain of Custody may also be signed by a representative of the microbiology department. In signing the Chain of Custody, the representative from the microbiology department shall follow the same procedures as sample receiving personnel.

10.3 The condition of the samples at the time of receipt must be documented.

10.3.1 If the sample is from an EPA project, a DC-1 form (Appendix D) must be completed to document the condition of the samples at the time of receipt.

10.3.2 If the sample is from the EPA Cincinnati UCMR2 project, an ALSI EPA UCMR2 Sample Receipt Checklist (Appendix E) must be completed.

10.3.3 Any other checklist or cooler receipt form provided by a client must be completed in its entirety.

10.3.4 The condition of all other samples at the time of receipt shall be documented in the receipt information section of the ALSI Chain of

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Custody. If the Chain of Custody is from another source other than ALSI, a receipt information label shall be affixed to the Chain to record this information.

10.3.5 If the project requires an internal Chain of Custody, this procedure must be initiated by the Sample Receiving Department at login. Refer to the Legal Chain of Custody SOP (99-LCOC) for details.

10.3.6 The appropriate customer service representative shall be notified of non-conformances so that the client can be contacted and a decision made whether to run the sample as received or to resample. If required, sample receiving personnel can also make attempts to contact the client with any question or non-conformance. All information provided by the client shall be documented on the COC. Notify the appropriate customer service representative of all communication with the client.

10.4 The temperature of the cooler/container must be taken at the time of receipt.

10.4.1 If a temperature blank is provided with the cooler, a calibrated thermometer shall be used to determine the cooler temperature. The correction factor shall be applied to the measurement and the corrected temperature along with the thermometer ID shall be recorded on the Chain of Custody in the receipt information section.

10.4.2 If a temperature blank is not provided, the temperature of the sample is taken with a calibrated IR gun held no more than one inch away from the sample bottle or temperature blank. The reading must be taken directly through the side of the bottle. It shall not be taken through the lid or through any label, etc., adhered to the bottle. The correction factor shall be applied to the measurement and the corrected temperature along with the thermometer ID shall be recorded on the Chain of Custody in the receipt information section.

10.4.3 If this protocol is not feasible, a small amount of sample shall be decanted from a non-volatile bottle and a calibrated thermometer shall be used to read the temperature of the sample as discussed in Section 10.4.1.

10.4.4 If the temperature reading does not fall within the acceptable range (above the freezing point of water up to 6ºC), multiple readings shall be taken using at least two other bottles contained in the same cooler to insure that a representative reading has been obtained. All readings shall then be recorded along with the identification of the bottles from which they were taken.

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10.4.5 Samples that are hand delivered to the laboratory on the same day that they are collected may not meet these criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice.

10.4.6 All samples shall be observed for receipt on ice. The observation shall be documented by checking the appropriate Y/N box in the receipt information section of the Chain of Custody. The individual noting this fact shall also initial the corresponding line.

10.4.7 Samples shall remain on ice or be transferred to an appropriate refrigerator until processing. Microbiological samples shall be stored in Refrigerator 18 and volatile samples shall be stored in Refrigerator 27 to prevent contamination until transport to the appropriate departments.

10.5 All samples shall be checked for custody seals. The presence of custody seals shall be noted on the Chain of Custody by checking the appropriate Y/N box in the receipt information section of the Chain and initialing the corresponding line. If custody seals are present, the presence or absence of tampering shall be documented by checking the appropriate Y/N box in the receipt information section of the Chain. Details involving any tampering with custody seals shall be noted in the sample/COC comments section of the Chain.

10.6 All samples shall be checked for breakage/leakage. The presence or absence of damage shall be noted by checking the appropriate Y/N box under “Cont. in Good Cond.” in the receipt information section of the Chain. Details involving any breakage shall be noted in the sample/COC comments section of the Chain.

10.7 All samples shall be checked to insure that the information on the Chain of Custody agrees with the information on the container labels. This information would include sample descriptions, date/time sampled, sampler ID, and analyses requested. Whether or not the Chain of Custody and labels agree shall be documented by checking the appropriate Y/N box in the receipt information section of the Chain. Any discrepancies between the Chain and labels shall be specifically mentioned in the sample/COC comments section of the Chain.

10.8 All samples shall be checked to insure that the containers received are appropriate for the analyses requested and that a sufficient sample volume has been provided. This information shall be documented by checking the appropriate Y/N boxes under “Correct Containers” and “Correct Sample Volumes” in the receipt

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information section of the Chain. Any non-conformances shall be specifically mentioned in the sample/COC comments section of the Chain.

10.9 Samples contained in a septa-sealed container to be analyzed for volatile analytes, shall be checked for the appearance of headspace. Headspace is the appearance of air in a septum-lined container. A pea sized amount of air or smaller is allowable. Whether or not headspace is present shall be noted by checking the appropriate Y/N box in the receipt information section of the Chain. Any non-conformances shall be specifically mentioned in the sample/COC comments section of the Chain.

All volatile samples shall be held in the Sample Receiving refrigerator designated for VOC only (Refrigerator 27) and protected from contamination until the COC is entered into the LIMS system and the identifying labels are attached to each vial. The samples will then be transported to the VOC refrigerators in the organics lab for analysis.

10.10 The pH of all acid or caustic preserved bottles (with the exception of volatile containers) shall be taken using the pH paper listed in Section 5. Bottles shall be preserved according to the SOP 19-BOTTLE. See the most recent revision of the QA Manual, Appendix B, for a list of analyses that require pH testing.

10.10.1 The pH of the samples is measured by inserting a 5-3/4" Pasteur pipette into the sample. After the pipette is drawn out, a drop of the sample is placed on the pH paper. The pH is read using the color charts on each package of the pH strips. If the pH falls within the correct range, it is recorded on the lid of the sample container using a permanent waterproof marker and in the sample preservation log (see Appendix A).

10.10.2 If the pH is not within the acceptable range, the pH is adjusted by adding the appropriate preservative (refer to SOP 19-BOTTLE). The initial pH, the amount of preservative added, and the final pH are recorded on the lid of the sample container using a permanent waterproof marker and in the sample preservation log.

10.10.3 For samples requiring **DoD** certification, a small amount of sample shall be poured out of each bottle (excluding volatile samples) to check for the appropriate pH.

10.10.4 The following information must be recorded in the sample preservation log: the sample number, date received, initial pH, and the initials of

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the person taking the pH. In cases when the pH was adjusted, the amount of preservative added and the final pH are also recorded along with the lot of the preservative added. The log is generated in a Microsoft Excel spreadsheet that is protected against making any changes to the posted results. Each bottle is scanned using a Symbol Pocket PC with Pocket Excel installed.

10.10.5 Whether or not samples are preserved properly shall be documented by checking the appropriate Y/N box in the receipt information section of the Chain. Any non-conformances shall be specifically mentioned in the sample/COC comments section of the Chain.

10.10.6 If a metals bottle was acidified upon receipt at the laboratory, an orange sticker shall be placed on the bottle lid with the date and time of preservation, and shall be noted in the preservation log. The Metals Dept. shall verify the pH prior to use. NOTE: If the sample is to be analyzed for mercury, a corrective action report must be filled out.

10.11 All samples received must be checked for holding times.

10.11.1 Particular attention shall be given to any microbiological samples received, especially fecal coliforms which have a holding time of 6 hours for wastewater samples and frequently arrive at the laboratory with little time remaining prior to expiration. A representative of the microbiology department shall be notified immediately upon the arrival of these samples.

10.11.2 The presence of any other short holding time tests (less than 48 hours) that require immediate attention shall also be noted. If any short holding time tests require attention, the appropriate analytical department shall be notified. A list of holding times is maintained in the most recent revision of the QA Manual, Appendix B.

10.11.3 Particular attention shall also be given to soil samples that require any type of volatile analysis (8260, 8021, GRO, etc.). If these samples are received as encores or in soil jars they must be extracted within 48 hours of collection. Pre-weigh vials do not need any special treatment. The procedure for processing encores and soil jars is detailed in Appendix G.

10.11.4 Any samples received more than five days after the time of collection shall be evaluated for all holding times. Some extractions and wet chemistry procedure have 7 day holding times and while these would

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not normally be considered "short holds", they may be in jeopardy due to the excessive time elapsed between sampling and receipt. Analytical departments shall be notified of any such samples. Customer service personnel or a laboratory manager shall be consulted if a holding time is in question.

10.11.5 For any samples received at the laboratory with analyses that are already past hold at the time of receipt, the Chain of Custody shall be stamped with the red "EXPIRED" stamp and the expired tests noted in the stamp's box area.

10.12 The chlorine content shall be checked for samples requiring sodium thiosulfate or sodium sulfite as a preservative or samples requesting cyanide. See the most recent revision of the ALSI QA Manual, Appendix B, for a list of analyses that require sodium thiosulfate or sodium sulfite as a preservative.

10.12.1 In order to verify that the preservative has been added, the samples are tested for the absence of chlorine by inserting a 5-3/4" Pasteur pipette into the sample and placing a drop on KI paper for non drinking water samples. For drinking water samples, the residual chlorine is verified by using the total chlorine paper. If a color change to blue or violet is indicated, the chlorine in the sample shall be neutralized (see SOP 19-BOTTLE).

10.12.2 The presence/absence of chlorine must be documented in the sample preservation logbook file. Whether or not samples are preserved properly shall be documented on the Chain of Custody by checking the appropriate Y/N box in the receipt information section of the Chain and initialing the corresponding line. Any non-conformances shall be specifically mentioned in the sample/COC comments section of the Chain.

10.13 The turbidity of drinking water samples for metals analysis shall be determined by the Metals or Wet Chemistry department prior to analysis.

10.14 At this time, the samples are entered into the Horizon LIMS. See SOP for COC Entry (19-COC).

10.15 Labels generated by the Horizon LIMS are then affixed to the bottles and the bottles are stored as follows:

10.15.1 Volatile aqueous samples for GC are stored in Refrigerator #26 and #29

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located in the GC lab. Samples known to contain a high concentration of volatiles shall not be stored in this area. (Depending upon the matrix, high-concentration volatiles are often stored in the oil cabinet in Sample Receiving, or if requiring refrigeration, may be stored in the GCMS Soil Refrigerator #10.)

10.15.2 Volatile aqueous samples for GC/MS are stored in Refrigerators #26 and 29 located in the GC/MS lab. Samples known to contain a high concentration of volatiles shall not be stored in this area. (Depending upon the matrix, high-concentration volatiles are often stored in the oil cabinet in Sample Receiving, or if requiring refrigeration, may be stored in the GCMS Soil Refrigerator #10.)

10.15.3 Metals samples are stored in numerical order on shelves in the Inorganic Prep Lab excluding drinking water metals samples. Drinking water metals samples that do not require a digestion are stored in the Metals lab. Dissolved metals are stored in the Metals Department.

10.15.4 Total Organic Carbon and Total Organic Halogen samples are stored in the walk-in refrigerator on the lower level.

10.15.5 Samples requiring any sort of organic extractions are stored on the right side of the Harford walk-in refrigerator located on the upper level.

10.15.6 All other liquid samples not mentioned are stored in the sample receiving and lower level walk-in refrigerators in numerical order.

10.15.7 Solid samples are stored on the left side of the Harford walk-in refrigerator on the upper level.

10.15.8 Solid volatile samples and petroleum samples are stored in either GC or GCMS, wherever the testing is done.

10.15.9 A very limited number of samples do not require refrigeration. These are often associated with atypical analysis. Approval from a department supervisor is required to store samples without refrigeration.

10.15.10 Samples with special storage considerations like solvents or free product are stored in the oil cabinet.

10.16 All samples are stored at least two (2) weeks following the completion of the last analysis on the lab report with the exception of VOA vials and microbiology samples. Samples requiring special storage considerations are handled project by

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project by the appropriate customer service representative. Samples requiring **DoD** certification must be stored for 60 days.

10.17 Samples received over the weekend and during off-peak hours shall be handled in the manner outlined in Appendix B.

11 Calculations

11.1 Not applicable

12 Reporting Results

12.1 Not applicable

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

16 Troubleshooting

16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the instrumentation used in this method.

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Appendix A

Preservation Log available on the ALSI Network

Preservation Log

Container Number	pH Received	Adjustment Amount Needed	Final pH	Preservation Lot Number	Residual Chlorine	Date	Time	Initials	pH Recheck Date/Time/Recheck

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Appendix B

The following steps can be followed for samples received after hours and on weekends:

1. Sign the Chain of Custody (COC) in the "received by" section on the bottom of the COC form only after inspecting samples to ensure that all information on the COC matches the samples being submitted for client submission.

2. Tear off the gold copy of the COC (last page), and give it to the client submitting the sample.

3. Check the analyses required for the samples received on the COC for any analyses on the following list which require short holding times (HT). If any of these tests are required and there are no trained personnel available to complete the analysis within the holding time, contact the supervisor of the department that performs the analysis. If the supervisor cannot be reached, contact the Laboratory Manager or appropriate Customer Service Representative.

Analyze Immediately	24 Hour HT	48 Hour HT
Chlorine Demand	Hexavalent Chromium	BOD/CBOD
Turbidity	Total Coliform (30 hr)	Nitrate
Residual Chlorine		Nitrite
Total Chlorine		Orthophosphate
Odor		MBAS
Dissolved Oxygen		Osmotic Pressure
pH		Color
Sulfite		Settleable Solids
Fecal Coliform (6hr)		UV254
		Encore Extraction

4. Check the temperature of the samples as specified in Section 10.4

5. Place samples and COC form together in the lower level walk-in.

6. Leave an E-mail message for the appropriate Customer Service Representative letting them know what was received, and what actions were taken.

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Appendix C

Analytical Laboratory Services, Inc.
Environmental • Industrial Hygiene • Field Services
34 Dogwood Lane • Middletown, PA 17057 • 717.944.5541 • Fax: 717.944.1430

CHAIN OF CUSTODY/ REQUEST FOR ANALYSIS

ALL SHADED AREAS MUST BE COMPLETED BY THE CLIENT / SAMPLER. INSTRUCTIONS ON THE BACK.

Page ___ of ___
Courier:
Tracking #:
COC#

Co. Name:
Contact (Report to): Phone:
Address:

Bill to (if different than Report to): PO#:

Project Name/#: ALSI Quote #:

TAT: ☐ Normal-Standard TAT is 10-12 business days. Date Required:
☐ Rush-Subject to ALSI approval and surcharges. Approved By:

Email? ☐ -Y
Fax? ☐ -Y No.:

ANALYSES/METHOD REQUESTED

Sample Description/Location (as it will appear on the lab report)	COC Comments	Sample Date	Military Time	*G or C	**Matrix	Enter Number of Containers Per Analysis											
***Container Type																	
***Container Size																	
*** Preservative																	
1																	
2																	
3																	
4																	
5																	
6																	
7																	
8																	

Receipt Information (completed by Sample Receiving)
Performed by: INITIAL HERE
Cooler Temp:
Therm. ID:
No. of Coolers:
Notes:

Circle appropriate Y or N.				
Correct containers?	N	N	N	N
Correct sample volume?	Y	Y	Y	Y
Correct preservation?				
Headspace/Volatiles?				
Custody seals Present?	N	N	N	N	N
(If present) Seals intact?	Y	Y	Y	Y	Y
Received on Ice?				
COC/Labels complete/accurate?				
Container in good condition?				

SAMPLED BY (Please Print):
LOGGED BY(signature): DATE: TIME:
REVIEWED BY(signature): DATE: TIME:

Relinquished By / Company Name	Date	Time	Received By / Company Name	Date	Time
1			2		
3			4		
5			6		
7			8		
9			10		

Data Deliverables: ☐ Standard ☐ CLP-like ☐ NJ-Reduced ☐ NJ-Full ☐ (other)
EDDs Required? ☐ If yes, format type:
SDWA Forms?⇨ yes ☐ yes ☐ yes ☐ yes ☐
State Samples Collected In? MD ☐ NJ ☐ NY ☐ PA ☐ Other
⇨Enter PWSID No.
DOD Criteria Required?

ALSI FIELD SERVICES
☐ Pickup
☐ Labor
☐ Composite Sampling
☐ Rental Equipment
☐ Other:

* G=Grab; C=Composite
**Matrix: AI=Air; DW=Drinking Water; GW=Groundwater; OI=Oil; OL=Other Liquid; SL=Sludge; SO=Soil; WP=Wipe; WW=Wastewater
Copies: WHITE - ORIGINAL CANARY - CUSTOMER COPY
***Container Type: AG-Amber Glass; CG-Clear Glass, PL-Plastic. Container Size: 250ml, 500ml, 1L, 8oz., etc. Preservative: HCl, HNO3, NaOH, etc.
Rev 6/07

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Appendix D
DC-1 Form

Region III
SAMPLE LOG-IN SHEET

PAGE 1 OF 2

LOG-IN DATE:
LAB NAME:
RECEIVED BY:

DAS N0.:
SDG NO.:

SIGNATURE:

CHECK THE APPROPRIATE RESPONSE:

	PRESENT	ABSENT	INTACT	BROKEN
CUSTODY SEAL(S)				
CHAIN OF CUSTODY (COC) RECORD				
TRAFFIC REPORT OR PACKING LIST				
AIRBILL / STICKER				
SAMPLE TAGS				
SAMPLE TAG NUMBERS ON CHAIN OF CUSTODY				
DATE RECEIVED BY LAB:				
TIME RECEIVED:				
DOES INFORMATION AGREE ON COC AND TAGS				
10. AIRBILL NUMBER				

SAMPLE TRANSFER		
FRACTION	DATE	BY

REVIEWED BY:
DATE:

LOGBOOK NO.:
LOGBOOK PAGE NO.:

DC-1

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Region III
DC- 1
Data Package Inventory Checklist

PAGE 2 OF2

SAMPLE LOG-IN SHEET

EPA SAMPLE #	SAMPLE TAG #	LAB ASSIGNED #	CUSTODY SEAL #	SAMPLE CONDITION

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Appendix E

ALSI EPA UCMR2 Sample Receipt Checklist

Received by name:_____ Signature: _____.
Date received: _____.

Kit ID Number								
Time received								
Temperature 527 Bottles	(1)							
Temperature 529 Bottles	(1)							
Thermometer ID								
Were samples in amber glass 1 liter bottles?	(2)	Y N	Y N	Y N	Y N	Y N	Y N	Y N
Were bottles filled to at least 80 % capacity?	(2)	Y N	Y N	Y N	Y N	Y N	Y N	Y N
Are bottles and caps unbroken and not leaking?	(2)	Y N	Y N	Y N	Y N	Y N	Y N	Y N
Is a sample tracking form included and complete?	(3)	Y N	Y N	Y N	Y N	Y N	Y N	Y N
Do the bottles in the kit match the tracking form?	(3)	Y N	Y N	Y N	Y N	Y N	Y N	Y N
Do the collection dates on the bottles match the tracking form?	(3)	Y N	Y N	Y N	Y N	Y N	Y N	Y N
Are there no manual edits on the tracking form for "sample point information" or "PWS Name"?	(3)	Y N	Y N	Y N	Y N	Y N	Y N	Y N
Were the correct preservatives used for 527 (L-Ascorbic acid, Trisodium EDTA, and Potassium dihydrogen citrate)?	(4)	Y N	Y N	Y N	Y N	Y N	Y N	Y N
Were the correct preservatives used for 529 (Tris (hydroxymethylaminomethane), Copper sulfate, and Tris (hydroxymethylaminomethane-HCl)?	(4)	Y N	Y N	Y N	Y N	Y N	Y N	Y N
For 527 is the pH ≤ 4.0?	(4)	Y N	Y N	Y N	Y N	Y N	Y N	Y N
For 527 is the DPD test negative?	(4)	Y N	Y N	Y N	Y N	Y N	Y N	Y N
For 529 does a visual comparison indicate a properly preserved sample? The sample should be bright blue and clear of precipitate.	(4)	Y N	Y N	Y N	Y N	Y N	Y N	Y N
Was the EPA notified of any nonconformance?	(5)	Y N	Y N	Y N	Y N	Y N	Y N	Y N

Date/Time EPA Notified: _____. Notified by: _____
Date/Time Samples Logged In:_____. Logged by: _____
Date/Time Reviewed:_____. Reviewed by: _____

Revision 01/29/08

(1) If samples are received above 10 ° C they must be invalidated and GLEC / EPA notified
(2) Broken bottles, bottles with missing or loosed caps, or bottles filled to less than 80 % are invalid. If a back-up is available, recollection may not be necessary, but EPA / GLEC still needs to be notified.
(3) If sample tracking forms are missing, or information is missing, conflicting, or if there are manual edits, GLEC and EPA should be notified to determine if the samples are valid.
GLEC/ EPA do not need notified if:
1) Only one date is recorded and it makes logical sense. Assume the recorded date is valid. Fill the date in the missing location and initial it ALS-XYZ where XYZ are the sample receiving technician's initials.
2) There is an obvious transcription error in year recorded. Use the logical year and make the correction by drawing a single line through the incorrect year and initializing as in "1" above.
3) One date is within the past two days and the second is some time in the future. Use the logical date and make the correction by drawing a single line through the incorrect year and initializing as in "1" above.
(4) Samples that don't meet presrvation criteria should be invalidated and GLEC / EPA notified.
(5) Notification of the problem should be e-mailed to the EPA Task Order project officer (fair.pat@epamail.epa.gov) with "Sampling Error" in the subject line. A copy should also be e-mailed to the EPA Project Officer (hautman.dan@epa.gov), the EPA UCMR2 Sampling Coordinator (Newport.bonnie@epa.gov) and the EPA's Implementation Contractor (UCMRsmall@glec.com). Include a copy

Revision 01/29/08

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Appendix F

New Employee Orientation

Sample Receiving Training Checklist

Procedure: Checking Sample Preservatives

SOP:_____

Revision:_____

	Analyst	Trainer
1) Analyst has read and understands SOP 19-Rec/Han and signed the concurrence form.	_____	_____
2) Analyst knows where to find the list of sample types that require preservation.	_____	_____
3) Analyst knows which pH paper to use for checking the pH of acidified samples and where to find it.	_____	_____
4) Analyst knows how to apply the sample to the pH paper and how to interpret the reading for acidified samples.	_____	_____
5) Analyst knows which pH paper to use for checking the pH of basic samples and where to find it.	_____	_____
6) Analyst knows how to apply the sample to the pH paper and how to interpret the reading for basic samples.	_____	_____
7) Analyst knows how to adjust the pH of any sample if the measured value is not acceptable.	_____	_____
8) Analyst knows which test papers to use for checking chlorine content and where to find them.	_____	_____
9) Analyst knows how to apply the sample to the test papers and how to interpret the reading for chlorine content.	_____	_____

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Rev. 01/08

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	Analyst	Trainer
10) Analyst knows how to properly complete the preservation logbook.	_____	_____
11) Analyst knows that DoD samples require a small amount of Sample to be poured out for sampling pH.	_____	_____
12) Analyst knows how to complete all information on the chain of custody regarding preservation including where to record non-conformances.	_____	_____
13) Analyst is aware that when metals samples are acidified upon Receipt a copy of the preservation log must accompany the sample to the metals prep department.	_____	_____
14) Analyst has demonstrated his/her ability on at least five separate projects/workorders.	_____	_____

The workorders used for demonstration are:

_____
_____
_____
_____
_____

_____	_____	_____
Analyst Name	Analyst Signature	Date

_____	_____	_____
Supervisor Name	Supervisor Signature	Date

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New Employee Orientation

Sample Receiving Training Checklist

Procedure: Measuring Cooler Temperatures

SOP:_____

Revision:_____

	Analyst	Trainer
1) Analyst read and understands SOP 19-Rec/Han and signed the concurrence form.	____	____
2) Analyst knows the location of the mercury thermometer.	____	____
3) Analyst knows where to find and how to apply the correction factor for the mercury thermometer.	____	____
4) Analyst can properly take and record a cooler temperature using a provided temperature blank.	____	____
5) Analyst knows the acceptable limits for cooler temperatures are above the freezing point of water to 6 degrees C.	____	____
6) Analyst knows the location of the IR temperature gun.	____	____
7) Analyst knows how to properly use the IR gun to take a sample temperature (i.e. where to shoot the gun through the bottle).	____	____
8) Analyst knows where to find and how to apply the correction factors for the IR temperature gun.	____	____
9) Analyst knows to take multiple temperatures if the initial bottle used is outside the acceptable limits.	____	____
10) Analyst is aware that samples received same day as sampling are acceptable if evidence exists that the chilling process was begun.	____	____

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Rev. 5/05

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	Analyst	Trainer
11) Analyst is aware that designated refrigerators are available for "holding" micro and volatile samples during processing.	_____	_____
12) Analyst has demonstrated this ability on at least five separate projects/workorders.	_____	_____

The workorders used for demonstration are:

_____
_____
_____
_____
_____

Analyst Name	Analyst Signature	Date
Supervisor Name	Supervisor Signature	Date

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UNCONTROLLED DOCUMENT

New Employee Orientation

Sample Receiving Training Checklist

Procedure: Receiving Samples

SOP:_____

Revision:_____

	Analyst	Trainer
1) Analyst has read and understands SOP 19-Rec/Han and signed the concurrence form.	_____	_____
2) Analyst will ensure that all samples are accompanied by a chain of custody.	_____	_____
3) If a walk-in client attempts to drop off samples not accompanied by a chain of custody, the analyst provides a chain for them to complete on-site.	_____	_____
4) Analyst receives custody of the sample(s) by signing the chain of custody upon receipt of samples, including the date and time received.	_____	_____
5) When samples are received with a chain of custody from a source other than ALSI, the analyst will confirm client and sample information is present.	_____	_____
6) Analyst checks for the presence of custody seals and whether they are intact and records all findings including any non-conformances appropriately on the chain of custody.	_____	_____
7) Analyst checks the sample temperature and for the presence of ice and records all findings including any non-conformances appropriately on the chain of custody.	_____	_____
8) Analyst checks for breakage and leakage and records all findings including any non-conformances appropriately on the chain of custody.	_____	_____

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UNCONTROLLED DOCUMENT

	Analyst	Trainer
9) Analyst compares the information on the chain of custody to the information on the bottle labels and records all findings including any non-conformances on the chain of custody.	_____	_____
10) Analyst checks to see that the sample containers are appropriate for the analyses requested and records all findings including any non-conformances on the chain of custody.	_____	_____
11) Analyst checks all volatile containers for headspace and records all findings including any non-conformances on the chain of custody.	_____	_____
12) Analyst properly checks for preservatives and records all findings including any non-conformances on the chain of custody.	_____	_____
13) Analyst knows what analytes have short holding times and notifies the appropriate departments upon receipt.	_____	_____
14) Analyst has demonstrated this ability on at least five separate projects/workorders.	_____	_____

The workorders used for demonstration are:

_____
_____
_____
_____
_____

_____	_____	_____
Analyst Name	Analyst Signature	Date

_____	_____	_____
Supervisor Name	Supervisor Signature	Date

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Appendix G

Receipt of Soil Volatile Samples in Encores or Soil Jars

Volatile soil samples received in soil jars or encores must be extracted into sodium bisulfate and/or methanol vials within 48 hours from the time of collection. Samples received with little time remaining prior to expiration require immediate attention. Proceed as follows:

1) When these types of samples are received, check the sample containers against the COC. Following procedure, note any discrepancies on the COC.

2) Log the samples into Horizon.

3) Initiate the ALSI Encore Internal COC Logbook. Even if the samples cannot be logged into Horizon for any reason, continue with this step. In such a case, use the sample description in lieu of a Horizon sample number.

4) Transfer the samples to the GC/MS laboratory along with a copy of the COC.

   NOTE: Samples must be delivered to an actual person who acknowledges receipt of the samples by initialing the Encore Internal COC Logbook. The samples cannot be left on a counter or placed in the refrigerator without documented receipt. If an individual qualified to extract encores is not available, refer to the GC/MS phone list located in sample receiving and contact an individual to come in and perform the extractions.

5) Complete the Encore Internal COC Logbook. (See Appendix H)

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Appendix H

ALSI ENCORE INTERNAL COC LOGBOOK

Sample #	Date Sampled	Time Sampled	Analysis Method	Date Received in Lab	Time Received in Lab	Received within 48 hours (Y/N)	Relinquished By	Received By	Date Delivered to Analyst	Time Delivered to Analyst	Comments

Rev. 4/03

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SOP Change Summary

Section No.	Section	Reason for Change
Revision 7:		
5.9	Apparatus and Materials	Correction to current SOP
6.0	Reagents	Correction to current SOP
10.2.8	Procedure	A2LA Audit Response
10.6-10.7	Procedure	Horizon LIMS Implementation
Revision 8:		
10.2.4	Procedure	New Jersey DEP audit response
Revision 9:		
2	Summary of Method	DoD audit response 5/13/05
4	Safety	DoD audit response 5/13/05
5	Apparatus and Materials	DoD audit response 5/13/05
6	Reagents	DoD audit response 5/13/05
7	Instrument Calibrations	DoD audit response 5/13/05
8	Quality Control	DoD audit response 5/13/05
10	Procedure	DoD audit response 5/13/05
Appendix C	Sample Collection, Containers & Preservation For Organic Contaminants	DoD audit response 5/13/05
Appendix D	ALSI Chain of Custody	DoD audit response 5/13/05
Appendix E	DC-2 Data Package Inventory Checklist	DoD audit response 5/13/05

SOP Change Summary (continued)

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Section No.	Section	Reason for Change

Revision 10: 10/06/2006

Revised refrigeration temperatures to read "…above the freezing point of water up to 6ºC…" to reflect NELAC verbiage.

1.3	Scope and Application	Added use of project specific criteria
4.1	Safety	Added statement about maintaining MSDS
6.4.1, 6.4.2	Reagents	Revised volume of dispenser tops from 1 mL to 2 mL; revised number of squirts to accommodate new dispenser volume
6.4.3.3	Reagents	Revised volume added to TOC bottle
8.1	Quality Control	Added verbiage indicating that the SOP falls under the QA Plan umbrella
8.4, 8.5	Quality Control	Added initial DOC and statement about ongoing proficiency
10.10.4	Procedure	Removed recording of tests required in sample preservation logbook; added generation of logbook in a Microsoft Excel spreadsheet
11	Calculations	Added section
12	Reporting Results	Added section
16	Troubleshooting	Added section

Removed Appendix duplicating QA Plan Appendix B, Container, Preservation, Storage and Holding Times

C	Appendix	Updated COC to current form
E	Appendix	Added examples of training forms

SOP Change Summary (continued)

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Section No.	Section	Reason for Change
Revision 11: 02/05/07		
5.8.3	Apparatus and Materials	Added new walk-in refrigerator
6.1	Reagents	Added ALSI generated reagent water
6.3	Reagents	Replaced "well/tap" water with "reagent" water
6.4.10	Reagents	Added Ethylenediamine (EDA) Solution
10.2.1	Procedure	Added specific verbiage for microbial samples
10.11	Procedure	Added verbiage about sample holding times, microbial sample test times, soil samples and corrective actions for samples past holding time
10.13	Procedure	Added Wet Chem as lab for turbidity analysis
10.15.5, 10.15.7	Procedure	Added specific sample storage locations; deleted defunct refrigerators
B	Appendix	Added Fecal Coliform to "Analyze Immediately" column; Added Total Coliform to "24 Hour HT" column
F	Appendix	Added directions for the receipt of soil volatile samples in Encores or Soil Jars
G	Appendix	Added example of Encore Internal COC logbook page
Revision 12: 01/29/08		
2	Summary	Reformatted section
4.1	Safety	Updated MSDS availability
5.6, 5.7	Apparatus and Materials	Updated thermometer information
6	Reagents	Removed inapplicable reagents

SOP Change Summary (continued)

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Section No.	Section	Reason for Change
7.1, 7.2	Instrument Calibration	Removed sections
10.3.1	Procedure	Replaced DC-2 with DC-1
10.3.2	Procedure	Added section
10.3.5	Procedure	Updated procedure per DoD audit
10.4.1	Procedure	Removed “mercury”
10.10.2	Procedure	Removed recording lot# per DoD audit
10.10.6	Procedure	Updated procedure per DoD audit
Appendixes		Updated numbering
Appendix D		Replace DC-2 form with DC-1 form
Appendix E		Added section
Appendix F		Replace USACE with DoD per DoD audit

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SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

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Document Title: Standard Operating Procedure for Chain of Custody Entry

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICE, INC. (ALSI)
Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

_____ _____
Helen MacMinn, Date
Quality Assurance Manager

_____ _____
Steve Smith, Date
Sample Receiving

_____ _____
Deborah J. Wood, Date
Validator

Annual Review:

_____ _____
Reviewed By Date Reviewed

_____ _____
Approved By Date Approved

_____ _____
Reviewed By Date Reviewed

_____ _____
Approved By Date Approved

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Annual Review (continued):

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

Reviewed By	Date Reviewed
Approved By	Date Approved

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1 Scope and Application

1.1 This standard operating procedure addresses the entry of chain-of-custodies for all customers of ALSI. This document shall serve as a guideline to entering the chain-of-custody when the sample(s) are received in the Sample Receiving Department.

1.2 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.

1.3 Individual projects may override criteria listed in this SOP.

2 Summary of Method

2.1 Enter your user name and password to access the Horizon LIMS.

2.2 From the Samples Menu select Login, then select By Container.

2.3 Access the "sample login" section and enter all data pertaining to the individual sample such as date/time sampled and analyses required.

3 Interferences

3.1 Not Applicable.

4 Safety

4.1 Samples may contain compounds and preservatives that are significant health hazards. All possible steps should be taken to limit analyst contact with these samples. The minimum personal protective equipment requirements when handling samples are PVC gloves, safety glasses, and a fully-buttoned lab coat. This PPE should reduce the possibility of contact to a safe level, but the analyst should not limit themselves to these PPE requirements.

4.2 ALSI maintains material safety data sheets (MSDS) on all chemicals used in this procedure. MSDS are available in hard copy in the QA office and electronically on the ALSI network.

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5 Apparatus and Materials

5.1 Laboratory computer station having access to the current version of Horizon; Horizon 9.0 is in use at the time of SOP 19-COC, revision 4.

6 Reagents

6.1 Not Applicable

7 Instrument Calibration

7.1 Not Applicable

8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 An Initial Demonstration of Capability shall be documented as follows:

8.2.1 Completion of the "New Employee Orientation: Sample Receiving Training Checklist: Sample Login." See Appendix C.

8.2.2 Corrective action for DOC failure:

8.2.2.1 Repeat training shall be performed until all forms are successfully completed.

8.2.2.2 It shall be at the discretion of supervisory staff and management to determine at what point repeat training is no longer applicable and reassignment or company termination is appropriate.

8.3 Ongoing proficiency on an annual basis, as specified in the QA Plan, Technical Training, does not apply to this procedure.

9 Sample Collection, Preservation, and Handling

9.1 Not Applicable

10 Procedure

10.1 Accessing Horizon

10.1.1 Double-click on the Horizon icon on the desktop menu. The Horizon login screen will appear.

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10.1.2 Enter your user name (first initial and last name) and password (password provided to the IT department.) The Horizon main menu will appear.

10.2 Creating Workorders

10.2.1 From the main menu select "Samples".

10.2.2 Select "Login".

10.2.2.1 Select "By Container"

10.2.3 From the Client field, click on the LOV (List Of Values) and select the client from the list.

10.2.4 Select the "Profile" field and click on the LOV (List of Values) icon.

10.2.5 Select the "Profile" field and click on the LOV (List of Values) icon.

10.2.6 The list shown will be only the profiles available for the client you have selected. Scroll down the list and pick the profile that best matches the COC you are logging in.

10.2.6.1 Profiles for DEP reportable drinking water samples will be identified by the 7 digit DEP identification number.

10.2.7 Selecting the profile will fill in all client information on the "Customer Login" screen. This information includes the client number and name, the type of deliverable, the report format, the sequence, the status, charges, the earliest due date, and the date created.

10.2.7.1 The type of deliverable can be changed if the COC requests a type different from the listed in the profile. Options under the LOV menu include: CM (commercial w/o deliverable), CT (commercial timeframe invoice), IL (invoicing at large), CQ (commercial QA/QC package), and SD (EPA sample deliverable group).

10.2.7.2 The report format can be changed if the COC requests a type different from that listed in the profile. Options under the LOV menu include: 40CFR (40 CFR report deliverable), SDWA (SDWA deliverable), Standard (standard

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deliverable), and UCMR (UCMR EDD deliverable).

10.2.8 Under "Work ID" field, type in a keyword(s) that will identify that project. These keywords can be identifiers such as "monthly DEP", weekly wastewater, etc. or they can be project or job numbers provided by a specific client. You can also press enter from this field and the computer will fill in the default workorder ID associated with the profile you have chosen. The workorder ID field must be blank for the default ID to work.

10.2.9 The PO field will be empty at this point. If a purchase order number is available enter it her. Unless the client has a PO that is used consistently, the PO in the "case" can be saved in the "Client Info". If the PO is stored in the client profile, the PO field will be filled in by the LIMS.

10.2.10 From the Collector field click on the LOV (List Of Values) Icon. A list will appear. Begin typing the last name of the collector, this will begin reducing the list of names making it easier to find the collector you are looking for.

10.3 Sample Login

10.3.1 From the Sample ID field, click on the LOV (List Of Values) Icon. If any valid ID's are stored in the client profile, they will appear in a list. If no valid ID's are available, the user can manually type in a Sample ID. Refrain from using abbreviations, all small cases or all upper case.

10.3.2 The "Phone", "Report to", "PO", "Location", "Description", "Rec Codes", "Chain", "Original", "Keywords", and "Paired" fields are empty fields.

10.3.3 Enter the date and time collected.

10.3.4 The "Matrix" field is filled in according to the profile, but can be changed if necessary. A list of matrix options can be seen by accessing the LOV menu for the "Matrix" field.

10.3.5 Enter the date and time received. The date and time received is the date and time the last signature on the COC was completed upon receipt by the sample receiving department.

10.3.6 The "Type" field and "Mgr" fields are filled in according to the profile.

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10.3.7 The "Turn" field is filled in according to the profile, but can be changed if necessary. A list of options can be seen by accessing the LOV menu for the "Turn" field. When selecting an option, always pick one expressed as "Workdays from Receipt".

10.3.8 The "Priority" field is filled in according to the profile, but can be changed if necessary. A list of options can be seen by accessing the LOV menu for the "Priority" field.

10.3.9 Click on the "Load Line" button from the top of the Horizon Screen. A list of available testing will be displayed associated with the client and profile chosen.

10.3.9.1 Line Item 1 for the profile will initially appear on the first line of the "Line Item" section. Click on the "Line Item"

10.3.9.2 All the line items available under that profile will appear. Line items can be a single test or a group of several different tests.

10.3.9.3 Select a line item that includes tests codes necessary for this sample. Click on "Set Line Item" Unchecking any tests will cause that test or tests not to load in the workorder.

10.3.9.4 You can continue to add testing from the list by "appending line items. Appending will add to what has already been loaded into the workorder. Setting A Line will cause everything loaded in the workorder to be replaced. Select the Line Item you wish to append then, click on "Append Line Item" to add these codes to the test codes already selected. Again unchecking any test will cause that test not to load.

10.3.9.5 Continue in this manner until all test codes needed are selected.

10.3.9.6 Once all test codes are loaded that are needed, click on OK on the Line Items window.

10.3.9.7 If a needed test code is not included as a line item, it can be entered manually. In the next unused Type field key in the type of container, or click on the LOV Icon to select from a list.

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10.3.9.8 Next key in the preservative or click on the LOV Icon and select the preservative from the list.

10.3.9.9 In the CC field key in "OK"

10.3.9.10 In the Count field key in the number of containers received.

10.3.9.11 Click in the ACODE 1 Field and then click on the LOV Icon and select the test code you are trying to add. You can continue to add additional testing to that bottle by going to the next ACODE to the Right. There are 16 available ACODES on a single line. You more is needed for a single bottle continue on the next line making sure The "Type" field remains blank. When the LIMS sees a blank type field, it automatically associates the testing for that line with the bottle listed above that line.

10.3.9.12 Once all test codes are entered, and all bottles are correct, click on "Save".

10.3.9.13 If auxiliary data is required for the sample, such as for DEP reportable samples, the auxiliary data field will appear.

10.3.9.14 Auxiliary data information will include: 1) Raw or Finished Water; Level of Compositing (Usually 0); Source (Surface (1), GUDI (2), or Groundwater (3); Start of Sampling Period; and End of Sampling Period.

10.3.9.15 Once the Auxiliary Data is entered, click "Save".

10.3.9.16 A "Lab ID" number will be generated, and unique bottle IDs will also be generated for each container, and the sample will be saved, and labels will print.

NOTE: If a second sample is included on the workorder, the LIMS will advance to the next sample. Proceed starting with Section 10.3.1. Information from the first sample will be carried over to the second and can be saved as is or changed. DO NOT changed the Work ID Field. If this field is altered in any way, the LIMS will begin a new workorder.

10.3.9.17 To begin a new workorder simply start from Section 10.2.3 and remember that you must changed the Work ID field to clear the

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WO number that the LIMS last assigned. You can also hold down the shift key while pressing F7 to clear the enter Container Login Screen the begin again from section 10.2.3. This is probably the "safest" way until you are comfortable with changing workorders.

10.4 Creating DEP Sample Identifier

10.4.1 From the Horizon main menu select the "Clients" icon.

10.4.2 Select "Setup/Edit"

10.4.3 Select the profile the DEP identified is to be added to.

10.4.4 Select "Valid ID" (Double check)

10.4.5 Add the new DEP identifier.

11 Calculations

11.1 Not Applicable

12 Reporting Results

12.1 Not Applicable

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

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15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

16 Troubleshooting

16.1 Refer to the maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the apparatus used in this method.

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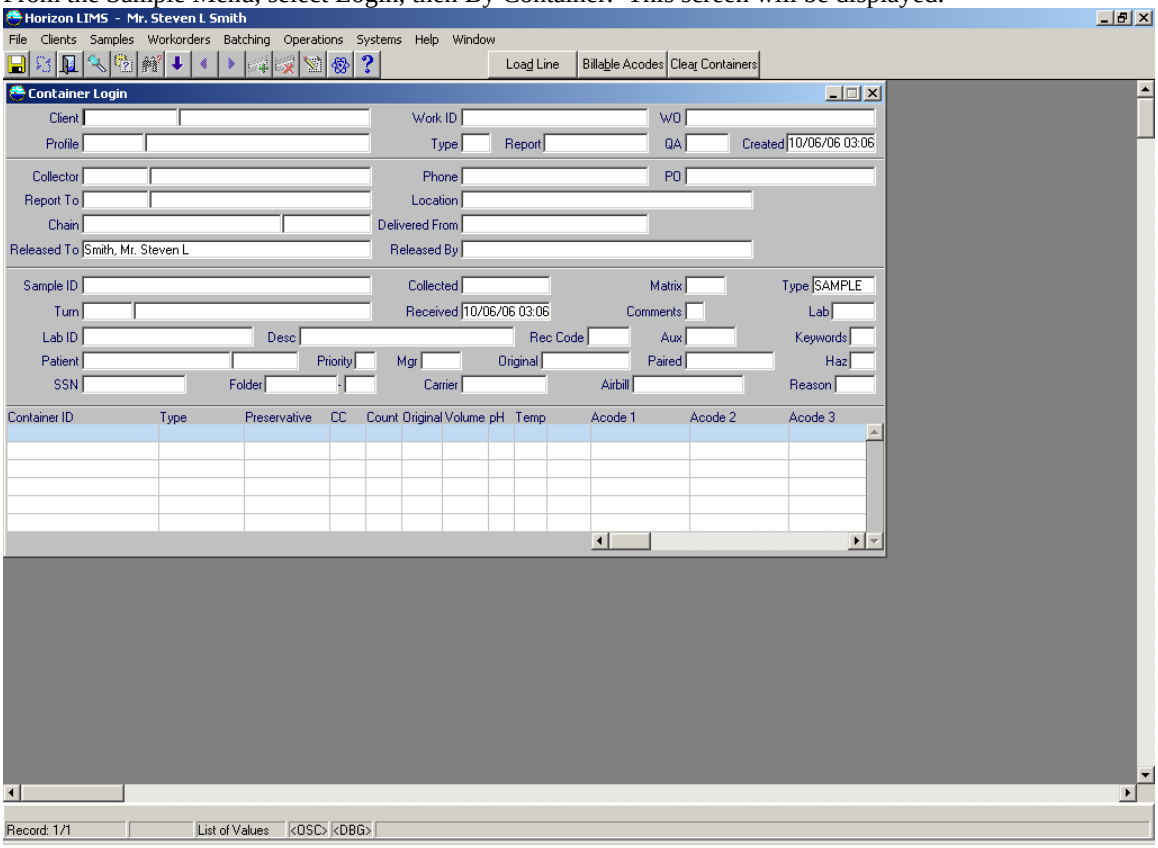
APPENDIX A

From the Sample Menu, select Login, then By Container. This screen will be displayed:



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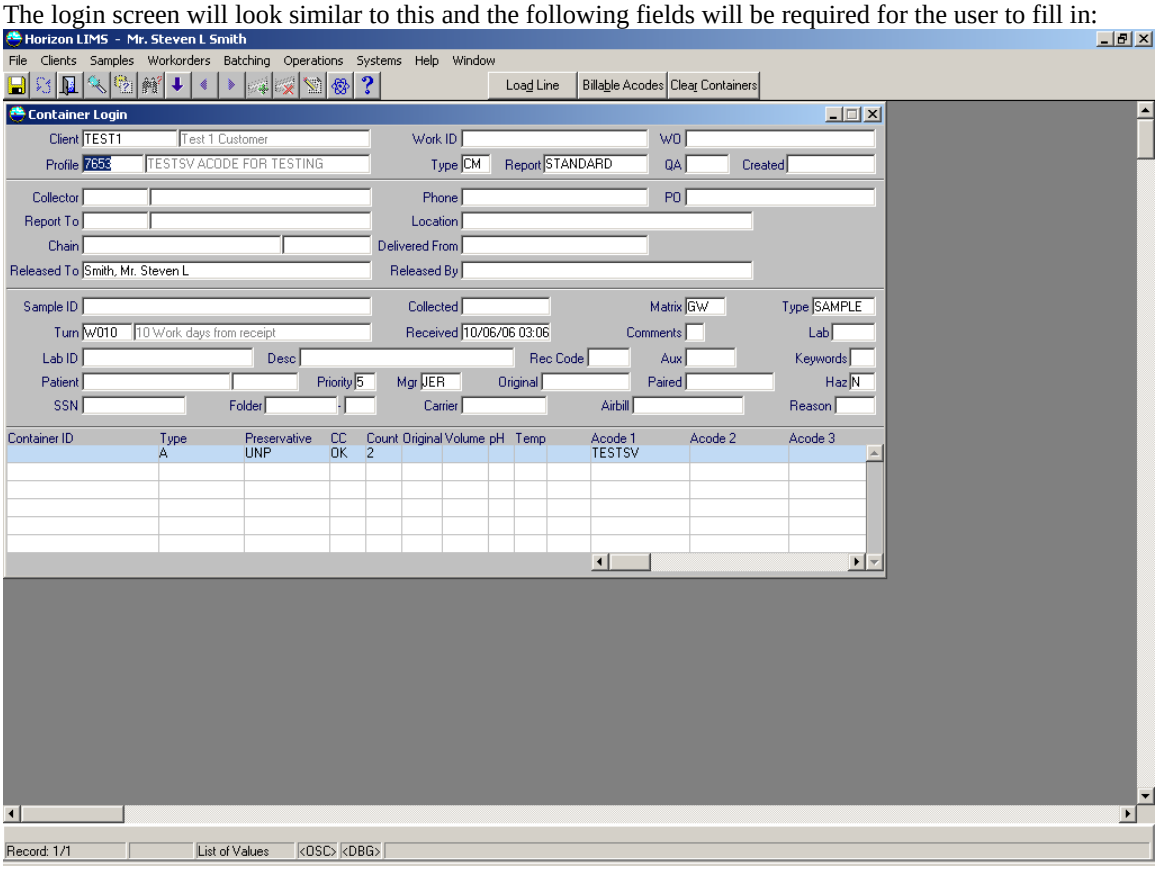
In the Client Field, Click on the List Of Values button. The client for the workorder will be displayed here. The login screen will look similar to this and the following fields will be required for the user to fill in:



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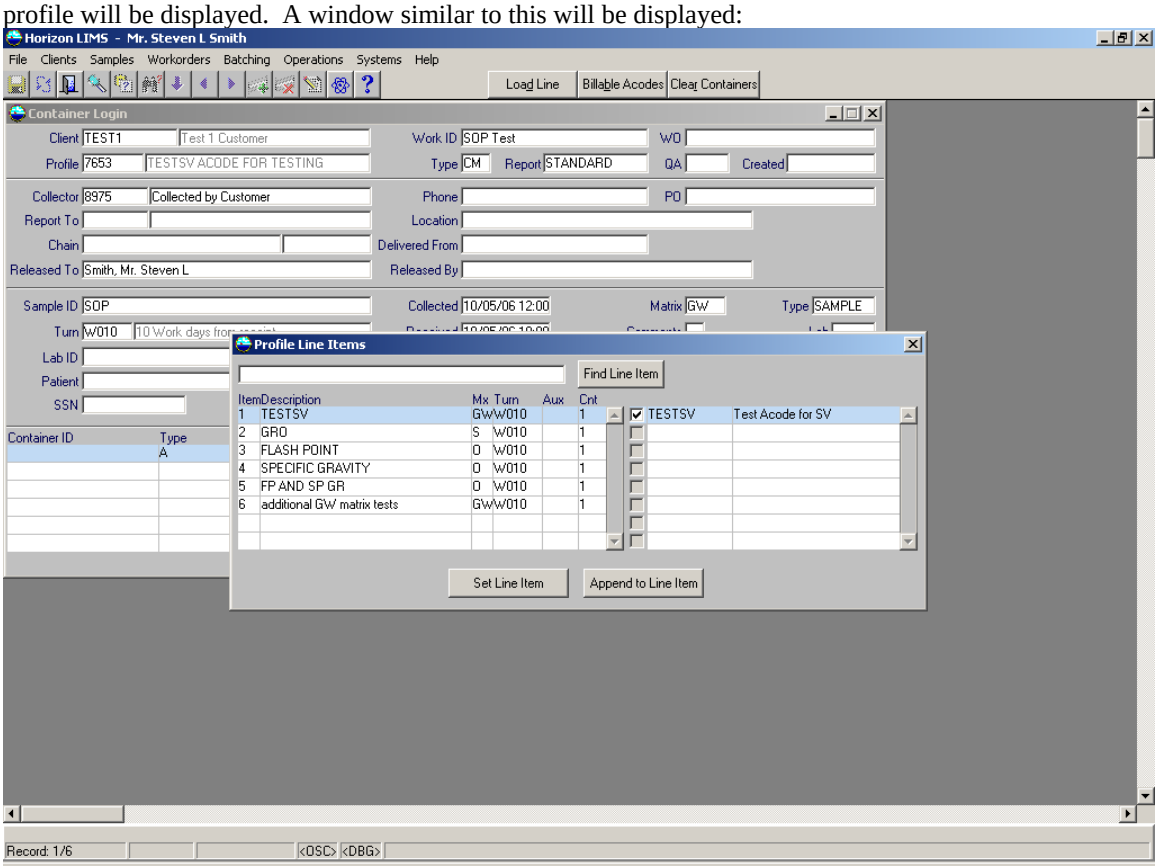
When the user is ready to add testing, simply click on the Load Line button. All available testing for the client and profile will be displayed. A window similar to this will be displayed:



From the Profile Line Items window, select the line item that best matches the COC being worked on. If a line item contains more testing then what is requested, simply uncheck what isn't to be added to the workorder. Click on the Set Line Item button. If additional line items are to be appended, simply click on the line item to be appended, uncheck any testing not needed to be added and click Append to Line Item.

NOTE: Clicking on the Set Line Item Button replaces everything.

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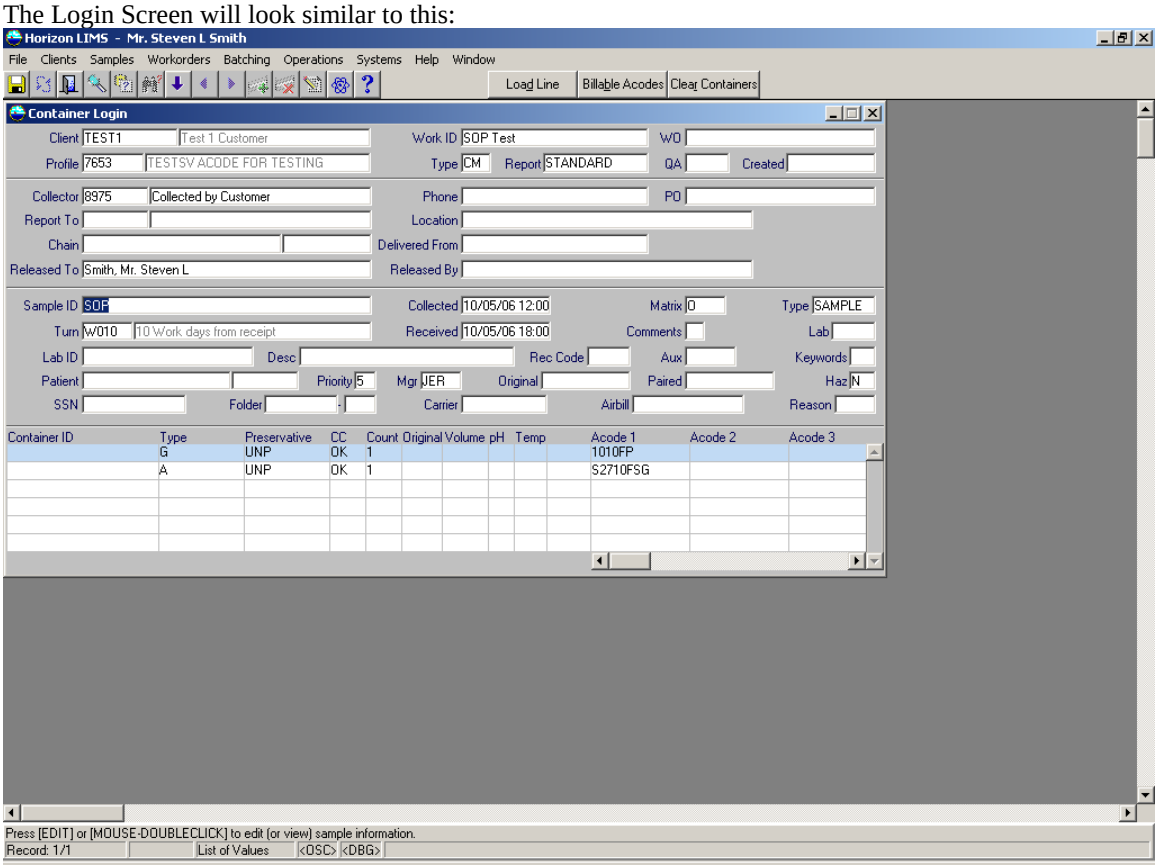
The Login Screen will look similar to this:



At this point the user can manually change a bottle or preservation for any bottle listed.
Once the user is satisfied to save and print labels, Click on the save button in the upper left corner of the login screen.

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APPENDIX B

New Employee Orientation

Sample Receiving Training Checklist

Procedure: Sample Log-in

SOP:_____

Revision:_____

	Analyst	Trainer
1) Analyst has read and understands SOP 19-COC and signed the concurrence form.	_____	_____
2) Analyst can access the Horizon LIMS.	_____	_____
3) Analyst can create a new workorder.	_____	_____
3) Analyst knows how to select a client profile.	_____	_____
4) Analyst knows how to change deliverable type and report format if necessary.	_____	_____
5) Analyst knows how to access the sample log-in screen.	_____	_____
6) Analyst knows how to select a sample collector.	_____	_____
7) Analyst knows how to select a sample ID, including an ID for a DEP reportable sample.	_____	_____
8) Analyst knows how to enter the date/time collected and received.	_____	_____
9) Analyst knows how to change the sample matrix if necessary.	_____	_____
10) Analyst knows how to modify the priority and turn-around fields if necessary.	_____	_____
12) Analyst knows how to add test codes using the "set line item" function.	_____	_____
13) Analyst knows how to append line items.	_____	_____

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Rev. 5/05

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	Analyst	Trainer
14) Analyst knows how to add test codes manually that are not included in line items.	_____	_____
15) Analyst knows how to enter auxiliary data.	_____	_____
16) Analyst knows how to create DEP sample identifiers.	_____	_____
17) Analyst demonstrated this ability on at least five separate projects/workorders, one of which is a DEP sample.	_____	_____

The workorders used for demonstration are:

_____
_____
_____
_____
_____

_____	_____	_____
Analyst Name	Analyst Signature	Date
_____	_____	_____
Supervisor Name	Supervisor Signature	Date

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SOP Change Summary

Section No.	Section	Reason for Change
Revision 4: 11/22/2006:		
1.2	Scope and Application	Added references to the most current NELAC standards
1.3	Scope and Application	Added use of project specific criteria
4.1	Safety	Added statement about maintaining MSDS
5.1	Apparatus and Materials	Revised reference to a specific Horizon version to read "current"
8.1	Quality Control	Added standard verbiage concerning ALSI QA Plan
8.2	Quality Control	Added DOC requirements
8.2.2	Quality Control	Added DOC failure corrective action
8.3	Quality Control	Added comment concerning ongoing proficiency
16	Troubleshooting	Added section
A	Appendix	Updated appendix
B	Appendix	Deleted screenshots; added training documentation

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SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date

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APPENDIX C

FIELD FORMS

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 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.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : _____ INSTRUMENT NAME/MODEL: _____

SITE NAME: _____ MANUFACTURER: _____

PROJECT No.: _____ SERIAL NUMBER: _____

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

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.

Remarks: ______________

Drilling Area Background (ppm): ____

Converted to Well: Yes ____ No ____ Well I.D. #: ______________

TETRA TECH NUS, INC.

CHAIN OF CUSTODY

NUMBER 0584

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 GRAB (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

DISTRIB WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY)

4/02R

FORM NO. TINUS 001

Tetra Tech NUS, Inc.

FIELD PORTABLE XRF SAMPLE ANALYSIS LOG SHEET

Project Site Name: _____ Measurement Units: _____

Project No: _____ Run Length/Duration _____

Analyzer: _____ Analysis Date: _____

Sample ID	Analysis Time	Element Analysis	Result Run 1	Result Run 2	Result Run 3	Results Avg.	Comments

Notes:

Tetra Tech NUS, Inc.

QA SAMPLE LOG SHEET

Project Site Name: _____
Project Number: _____
Sample Location: _____
QA Sample Type:

[] Trip Blank
[] Source Water Blank

Sample ID Number: _____
Sampled By: _____
C.O.C. Number: _____

[] Rinsate Blank
[] Other Blank _____

SAMPLING DATA:

Date: _____
Time: _____
Method: _____

WATER SOURCE:

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

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):

EQUIPMENT INSPECTION

COMPANY:_____ **UNIT NO.**_____

REQUENCY: Inspect at the initiation of the project, after repairs, once every 10-day shift.

Inspection Date: ____/____/____ Time: _____ Equipment Type:_____
(e.g., bulldozer, generator)

	Good	Need Repair	N/A
Tires or tracks	☐	☐	☐
Hoses and belts	☐	☐	☐
Cab, mirrors, safety glass	☐	☐	☐
- Turn signals, lights, brake lights, etc. (front/rear) for equipment approved for highway use?	☐	☐	☐
- Is the equipment equipped with audible back-up alarms and back-up lights?	☐	☐	☐
Horn and gauges	☐	☐	☐
Brake condition (dynamic, park, etc.)	☐	☐	☐
Fire extinguisher (Type/Rating -_____)	☐	☐	☐
Fluid Levels:			
- Engine oil	☐	☐	☐
- Transmission fluid	☐	☐	☐
- Brake fluid	☐	☐	☐
- Cooling system fluid	☐	☐	☐
- Windshield wipers	☐	☐	☐
Hydraulic oil	☐	☐	☐
Oil leak/lube	☐	☐	☐
Coupling devices and connectors	☐	☐	☐
Exhaust system	☐	☐	☐
Blade/boom/ripper condition	☐	☐	☐
Access-ways: Frame, hand holds, ladders, walkways (non-slip surfaces), guardrails?	☐	☐	☐
Power cable and/or hoist cable	☐	☐	☐
Steering (standard and emergency)	☐	☐	☐

Safety Guards:

	Yes	No
– Around rotating apparatus (belts, pulleys, sprockets, spindles, drums, flywheels, chains) all points of operations protected from accidental contact?_____	☐	☐
– Hot pipes and surfaces exposed to accidental contact?_____	☐	☐
– All emergency shut offs have been identified and communicated to the field crew?_____	☐	☐
– Have emergency shutoffs been field tested? _____	☐	☐
– Results?_____	☐	☐
Are any structural members bent, rusted, or otherwise show signs of damage?_____	☐	☐
– Are fueling cans used with this equipment approved type safety cans?_____	☐	☐

EQUIPMENT INSPECTION (Continued)

- Have the attachments designed for use (as per manufacturer's recommendation) with this equipment been inspected and are considered suitable for use?_____ ☐ ☐

Portable Power Tools:

- Tools and Equipment in Safe Condition?_____ ☐ ☐
- Saw blades, grinding wheels free from recognizable defects (grinding wheels have been sounded)? ☐ ☐
- Portable electric tools properly grounded?_____ ☐ ☐
- Damage to electrical power cords?_____ ☐ ☐
- Blade guards in place?_____ ☐ ☐
- Components adjusted as per manufacturers recommendation?_____ ☐ ☐

Cleanliness:

- Overall condition (was the decontamination performed prior to arrival on-site considered acceptable)?_____
- Where was this equipment used prior to its arrival on site?_____
- Site Contaminants of concern at the previous site?_____
- Inside debris (coffee cups, soda cans, tools and equipment) blocking free access to foot controls?_____

Operator Qualifications (as applicable for all heavy equipment):

- Does the operator have proper licensing where applicable, (e.g., CDL)?_____
- Does the operator, understand the equipment's operating instructions?_____
- Is the operator experienced with this equipment? _____
- Does the operator have emotional and/or physical limitations which would prevent him/her from performing this task in a safe manner?_____
- Is the operator 21 years of age or more?_____

Identification:

- Is a tagging system available, for positive identification, for tools removed from service?___

Additional Inspection Required Prior to Use On-Site

	Yes	No
- Does equipment emit noise levels above 90 decibels?	☐	☐
- If so, has an 8-hour noise dosimetry test been performed?	☐	☐

- Results of noise dosimetry:_____
- Defects and repairs needed:_____
- General Safety Condition: _____
- Operator or mechanic signature:_____

Site Safety Officer Signature:_____

Approved for Use: ☐ Yes ☐ No

SITE-SPECIFIC TRAINING DOCUMENTATION

My signature below indicates that I am aware of the potential hazardous nature of performing remedial investigation activities at _____ and that I have received site-specific training which included the elements presented below:

- Names of designated personnel and alternates responsible for site safety and health
- Safety, health, and other hazards present at the sites
- Use of personal protective equipment
- Safe use of engineering controls and equipment
- Medical surveillance requirements
- Signs and symptoms of overexposure
- Contents of the Health and Safety Plan
- Emergency response procedures (evacuation and assembly points)
- Initial response procedures
- Review of the contents of relevant Material Safety Data Sheets
- Review of the use of Safe Work Permits

I have been given the opportunity to ask questions and all of my questions have been answered to my satisfaction. I further state, that the dates of my training (introductory, refresher, and supervisory, as applicable) and my medical surveillance requirements are accurate and correct to the best of my knowledge.

Name (Printed and Signature)	Site-Specific Training Date	40-Hour Training (Date)	8-Hour Refresher Training (Date)	8-Hour Supervisory Training (Date)	Medical Exam

MEDICAL DATA SHEET

This Medical Data Sheet must be completed by all on-site personnel and kept in the command post during the conduct of site operations. This data sheet will accompany any personnel when medical assistance is needed or if transport to hospital facilities is required.

Project _____

Name _____ Home Telephone _____

Address _____

Age _____ Height _____ Weight _____

Name of Next Kin _____

Drug or other Allergies _____

Particular Sensitivities _____

Do You Wear Contacts? _____

Provide a Checklist of Previous Illnesses or Exposure to Hazardous Chemicals _____

_____

_____

What medications are you presently using? _____

_____

_____

Do you have any medical restrictions? _____

_____

_____

Name, Address, and Phone Number of personal physician: _____

_____

_____

_____

I am the individual described above. I have read and understand this HASP.

_____ _____

Signature Date

APPENDIX D

SAMPLE SIZE CALCULATIONS

DISCUSSION OF SAMPLING SIZE CALCULATION

Taking advantage of the expectation that there will be at each site an area of contaminated soil within an area of uncontaminated soil, a stratified sampling design was proposed. This required each area of significantly different concentration level to be treated as a single population. By doing this, the mean and standard deviation expected for the samples to be collected within each stratum would be different and this difference typically allows a lesser total number of samples to be collected while still meeting the specified decision performance. Do to the uses of the ranges present there are two stratified sampling plans that will be used. In the first situation the range will be split into two strata a clean, and a dirty. In the second situation the range will be split into three strata a clean, a dirty, and a transition stratum. For both situations the following inputs and assumptions were used:

- Null hypothesis (baseline condition) = Remedial action is needed.
- Chance of not taking action when it should be taken (alpha) = 15 percent.
- Chance of taking action when it should not be taken (alpha) = 25 percent.
- The contaminated area would be remediated so it is no more contaminated than the uncontaminated area.
- A typical estimate for standard deviation of data is equal to the range of expected concentrations divided by six. (The conservative approach of taking the maximum value divided by six was used so this would give us a larger standard deviation and thus larger sample size estimates).

By separating the site into two strata, there is a set of performance criteria for each stratum:

<u>Stratum #1 (Clean area):</u>

AL=400
Range of Values: 0-600
Mean = 250;
Alpha = 15 percent;
Beta = 25 percent;
Std. Dev. = 600/6 =100
Delta = difference from mean to AL = 150
Number of samples = 5

Stratum #2 (Contaminated area)

AL = 400
Range of Values = 2,000-50,000
Mean = 15000;
Alpha = 15 percent;
Beta = 25 percent;
Std. Dev. = 50000/6 = 8333
Delta = difference from mean to AL= 14600
Number of samples = 5

For the second situation where there are 3 stratums present

By separating the site into three strata, there is a set of performance criteria for each stratum:

Stratum #1 (Clean area)

AL = 400
Range of Values: 0-600
Mean = 250;
Alpha = 15 percent;
Beta = 25 percent;
Std. Dev. = 600/6 =100
Delta = difference from mean to AL= 150
Number of samples = 5

Stratum #2 (Transition Zone)

AL = 400
Range of Values: 400-5000
Mean = 2000;
Alpha = 15 percent;
Beta = 25 percent;
St. Dev. = 5000/6=833
Delta = difference from mean to AL= 1600
Number of samples = 5

Stratum #3 (Dirty)

AL = 400
Range of Values: 2000-50000;
Mean = 15000;
Alpha = 15 percent;
Beta = 25 percent;
St. Dev. = 50000/6=8333
Delta = difference from mean to AL= 14600
Number of samples = 5