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TIER I SAMPLING AND ANALYSIS PLAN (FIELD SAMPLING PLAN AND QUALITY
ASSURANCE PROJECT PLAN) FOR MUNITIONS RESPONSE PROGRAM EXTENDED SITE
INSPECTION OF MAGAZINE POINT BOMBING TARGET NAS PENSACOLA FL
3/1/2012
TETRA TECH

Comprehensive Long-term Environmental Action Navy

CONTRACT NUMBER N62467-04-D-0055



Rev. 0
3/12

Tier I Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan) for Munitions Response Program Extended Site Inspection of Magazine Point Bombing Target

**Naval Air Station Pensacola
Pensacola, Florida**

Contract Task Order 0148

March 2012



NAS Jacksonville
Jacksonville, Florida 32212-0030

SAP Worksheet #1 – Title and Approval Page

(UFP-QAPP Manual Section 2.1)

**TIER I SAMPLING AND ANALYSIS PLAN
(FIELD SAMPLING PLAN AND QUALITY ASSURANCE PROJECT PLAN)
FOR
MUNITIONS RESPONSE PROGRAM
EXTENDED SITE INSPECTION OF MAGAZINE POINT BOMBING TARGET
NAVAL AIR STATION PENSACOLA
PENSACOLA, FLORIDA**

March 2012

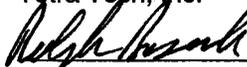
**Prepared for:
Department of the Navy
Naval Facilities Engineering Command
Southeast
NAS Jacksonville
Jacksonville, Florida 32212-0030**

**Prepared by:
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661 Andersen Drive
Pittsburgh, Pennsylvania 15220**

**Prepared under:
CONTRACT N62467-04-D-0055
CONTRACT TASK ORDER 0148**

Review Signatures:

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Task Order Manager
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 Date: 09/10/04/12

Kelly Carper
Quality Assurance Manager
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 Date: 10/04/12

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John Schoolfield
Navy Remedial Project Manager
NAVAC SE

_____ Date: _____

Kenneth Bowers
NAVFAC QAO/Chemist
NAVFAC SE

_____ Date: _____

Tim Woolheater
Remedial Project Manager
US EPA Region 4

_____ Date: _____

Dave Grabka
Remedial Project Manager
FDEP

_____ Date: _____

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EXECUTIVE SUMMARY

Tetra Tech, Inc. (Tetra Tech) has prepared this Sampling and Analysis Plan (SAP) under the Comprehensive Long-Term Environmental Action Navy (CLEAN) Contract No. N62467-04-D-0055, Contract Task Order (CTO) 0148. This SAP has been prepared for the extended Site Inspection (SI) for Munitions Constituents (MC) under the Munitions Response Program (MRP) at the Magazine Point Bombing Target located at Naval Air Station (NAS) Pensacola, Pensacola, Florida.

A Preliminary Range Assessment was finalized for NAS Pensacola in 2009, and the Magazine Point Bombing Target site was identified for further investigation. An SI for munitions and explosives of concern (MEC) was conducted at the site under an approved December 2009 Uniform Federal Policy (UFP)-SAP. No sampling has been conducted for munitions constituents (MC).

The Magazine Point Bombing Target is located at Naval Air Station (NAS) Pensacola in Pensacola, Florida ([Figure 1](#)). The entire Magazine Point Bombing Target site occupies a 72-acre area located on Magazine Point Peninsula. The Magazine Point Bombing Target was first denoted on a 1933 historical map as a chevron-shaped target located adjacent to Pensacola Bay along with one powder magazine and a radio spotting system. Based on the location of the bombing target in relation to Chevalier Field ([Figures 2](#) and [3](#)), it is likely that the Magazine Point Bombing Target site was used as a practice bombing range where practice bombs were dropped from aircraft with the intent of hitting a target on the ground.

The Magazine Point Bombing Target is located in a relatively flat area. It gently slopes to the north towards a bayou that flows into Bayou Grande and to the east to Pensacola Bay ([Figure 2](#)).

Whereas proximity to Chevalier Field probably limited munitions used at the site to practice bombs with inert fillers, those bombs may have included spotting charges, which would be defined as munitions and explosives of concern (MEC). Therefore, as recently as 2009, the area within the 500-foot scoring arc was thought to be an area potentially containing MEC.

A visual survey of the area around the Magazine Point Bombing Target was conducted on November 30, 2007 as part of the PA. The area from the shoreline of Pensacola Bay to the heavy vegetation line adjacent to the WWTP fence was visually surveyed. Much of the area was covered with various storm debris washed onto the shore. Concrete and asphalt pieces were also present in this area. These materials have been deliberately placed there for shoreline stabilization. A large mound of dirt was observed just south of the Bombing Target; the history and use of the mound is unknown. The heavily vegetated area included thick growth of slash pines, vines, and various shrubs such as saltbrush.

Because of inaccessibility, the heavily vegetated area and the area inside the WWTP were not surveyed. Tetra Tech personnel conducted a site walk on March 27, 2009 as part of a Site Inspection (SI) and confirmed the observations from the PA. A detector-aided survey was conducted by UXO Technicians February 16-21, 2010. No MEC, munitions potentially presenting an explosive hazard (MPPEH), or related debris was observed during the PA, SI visual, or the detector-aided surveys.

The primary objective of the SI was to determine whether further response actions or a Remedial Investigation (RI) was appropriate to restore the site to an acceptable environmental condition. During the SI, the background information provided in the PA Report was evaluated. In addition, supplemental site-specific environmental data were collected to determine types and rough orders of magnitude of MEC quantities at the site, and to refine site boundaries (footprint reduction).

Although no detections of MEC, MPPEH, or munitions debris (MD) were noted at the Magazine Point Bombing Target during the geophysical investigation, limited sampling at the site was recommended in the SI Report (Tetra Tech, 2010) to confirm that no MC (i.e., metals) associated with the practice bombs used at the site are present in the soil, and to verify that explosives (i.e., TNT, RDX, HMX, etc.) were not present in bombs dropped at the Magazine Point Bombing Target site.

Empirical Laboratory will be used for fixed-base laboratory (FBL) analysis for the incremental samples collected under this SAP. Data validation will be performed by Tetra Tech.

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ACRONYMS

bgs	below ground surface
CAS	Chemical Abstract Service
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CLEAN	Comprehensive Long-Term Environmental Action Navy
CRREL	Cold Regions Research Engineering Laboratory
CSM	Conceptual site model
CTO	Contract task order
DAF	Dilution attenuation factor
DL	Detection Limit
DMM	Discarded military munitions
DoD	Department of Defense
DQI	Data Quality Indicator
DQO	Data Quality Objective
DVM	Data Validation Manager
EDD	Electronic Data Deliverable
ELAP	Environmental Laboratory Accreditation Program
ERSM	Environmental Remediation Site Manager
FBL	Fixed Base Laboratory
FDEP	Florida Department of Environmental Protection
FOL	Field operations leader
FTMR	Field Task Modification Request
GPS	Global Positioning System
HASP	Health and Safety Plan
HPLC	High Performance Liquid Chromatography
HSM	Health and Safety Manager
ICP-AES	Inductively coupled plasma atomic emission spectroscopy
IDW	Investigation-derived waste
kg	kilogram
LCS	Laboratory control sample
LOD	Limit of Detection
LOQ	Limit of Quantitation
MC	Munitions constituents
MCL	Maximum contaminant level
MCS	Media Cleanup Standards
MD	Munitions debris

mm	millimeter
MEC	Munitions and explosives of concern
MMRP	Military Munitions Response Program
MPPEH	Munitions potentially presenting explosive hazard
MRP	Munitions Response Program
NAS	Naval Air Station
NAVFAC	Naval Facilities Engineering Command
NEDD	NIRIS Electronic Data Deliverable
NFA	No further action
NIRIS	Naval Installation Restoration Information Solution
OSHA	Occupational Safety and Health Administration
PA	Preliminary Assessment
PAL	Project Action limit
PM	Project Manager
PPE	Personal protective equipment
PQLG	Practical Quantitation Limit Goal
PT	Proficiency test
QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan
QC	Quality control
QSM	Quality Systems Manual
RCRA	Resource Conservation and Recovery Act
RI	Remedial investigation
RPD	Relative percent difference
RPM	Remedial Project Manager
RSD	Relative standard deviation
RSL	Regional screening level
SAP	Sampling and Analysis Plan
SCTL	Soil cleanup target level
SI	Site inspection
SOP	Standard operating procedure
SPLP	Synthetic precipitation leaching procedure
SSO	Site Safety Officer
TCLP	Toxicity characteristic leaching procedure
Tetra Tech, Inc.	Tetra Tech
TOM	Task Order Manager
UFP-SAP	Uniform Federal Policy-Sampling and Analysis Plan

UXO	Unexploded ordnance
USEPA	United States Environmental Protection Agency
WWTP	Wastewater treatment plant

SAP Worksheet #2 -- SAP Identifying Information

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number: Naval Air Station (NAS) Pensacola – Magazine Point Bombing Target
Site Name: NAS Pensacola, Pensacola, Florida
Operable Unit: Munitions Response Program Site
Contractor Name: Tetra Tech, Inc. (Tetra Tech)
Contract Number: N62467-04-D-0055
Contract Title: Comprehensive Long-Term Environmental Action Navy (CLEAN)
Work Assignment Number (optional): Contract Task Order (CTO) 0148

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)* (USEPA, 2005) and *EPA Guidance for Quality Assurance Project Plans, USEPA QA/G-5, QAMS (USEPA, 2002)*.
2. Identify regulatory program: Department of Defense (DoD) Military Munitions Response Program (MRP) using the general Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) process.
3. This SAP is a project-specific SAP.
4. List dates of scoping sessions that were held:

Scoping Session	Date
<u>DQO Scoping Meeting (Project Team)</u>	<u>July 29, 2011</u>

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

Title	Date
<u>Not Applicable – This is the initial MRP Site Investigation (SI) SAP for Munitions Constituents (MC)</u>	

6. List organizational partners (stakeholders) and connection with lead organization:
Florida Department of Environmental Protection (FDEP) (regulatory stakeholder)
United States Environmental Protection Agency (USEPA) Region IV (regulatory stakeholder)
U.S. Navy, Naval Air Station Pensacola (property owner)

7. Lead organization
Naval Facilities Engineering Command Southeast (NAVFAC SE)

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:

All SAP elements are included.

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
A. Project Management		
<i>Documentation</i>		
1	Title and Approval Page	NA
2	Table of Contents SAP Identifying Information	NA
3	Distribution List	NA
4	Project Personnel Sign-Off Sheet	NA
<i>Project Organization</i>		
5	Project Organizational Chart	NA
6	Communication Pathways	NA
7	Personnel Responsibilities and Qualifications Table	NA
8	Special Personnel Training Requirements Table	NA
<i>Project Planning/Problem Definition</i>		
9	Project Planning Session Documentation (including Data Needs tables) Project Scoping Session Participants Sheet	NA
10	Conceptual Site Model, 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		
<i>Sampling Tasks</i>		
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
<i>Analytical Tasks</i>		
23	Analytical SOPs, Analytical SOP References Table	NA
24	Analytical Instrument Calibration Table	NA
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	NA
<i>Sample Collection</i>		
26	Sample Handling System, Documentation Collection, Tracking, Archiving, and Disposal Sample Handling Flow Diagram	NA
27	Sample Custody Requirements, Procedures/SOPs Sample Container Identification Example Chain-of-Custody Form and Seal	NA

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
<i>Quality Control (QC) Samples</i>		
28	Laboratory QC Samples Table, Screening/Confirmatory Analysis Decision Tree	NA
<i>Data Management Tasks</i>		
29	Project Documents and Records Table	NA
30	Analytical Services Table Analytical and Data Management SOPs	NA
C. Assessment Oversight		
31	Planned Project Assessments Table, Audit Checklists	NA
32	Assessment Findings and Corrective Action Responses Table	NA
33	QA Management Reports Table	NA
D. Data Review		
34	Verification (Step I) Process Table	NA
35	Validation (Steps IIa and IIb) Process Table	NA
36	Analytical Data 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)
John Schoolfield	MRP Lead / Remedial Project Manager (RPM)/ Manages Project Activities for Navy	NAVFAC SE	(904) 542-6828	john.schoolfield1@navy.mil	Not Applicable (NA)
Greg Campbell	Site Manager / Point of Contact (POC) / Manages Site Activities	NAVY (Pensacola)	(850) 452-3131 ext. 3007	gregory.campbell@navy.mil	NA
Dave Grabka	RPM / Provides State Regulator Input	FDEP	(850) 245-8997	david.grabka@dep.state.fl.us	NA
Tim Woolheater	RPM / Provides USEPA Regulator Input	USEPA Region IV	(404) 562-8510	woolheater.tim@epa.gov	NA
Debra Humbert (cover letter only)	Program Manager / Manages Program Activities	Tetra Tech	(412) 921-8968	debra.humbert@tetrattech.com	NA
Ralph Basinski	Task Order Manager (TOM) / Manages Project Activities	Tetra Tech	(412) 921-8308	ralph.basinski@tetrattech.com	NA
Kelly Carper	Tetra Tech Quality Assurance Manager (QAM)/ Manages project QC	Tetra Tech	412.921.7273	kelly.carper@tetrattech.com	NA

Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-Mail or Mailing Address	Document Control Number (Optional)
Edward Sedlmyer (shared copy with Joseph Samchuck)	Project Chemist / Provides Technical Coordination with Laboratories	Tetra Tech	(412) 921-8704	edward.sedlmyer@tetrattech.com	NA
Joseph Samchuck (shared copy with Edward Sedlmyer)	Data Validation Manager (DVM) / Manages Data Validation	Tetra Tech	(412) 921-8510	joseph.samchuck@tetrattech.com	NA
Jim Goerdt	Field Operations Leader (FOL) / Manages Field Operations	Tetra Tech	(412) 921-8425	james.goerdt@tetrattech.com	NA
Brian Richard	Lab (Empirical) Project Manager (PM) / Representative for Laboratory and Analytical Issues	Empirical Laboratories	(615) 345-1115 X249	brichard@empirlabs.com	NA

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

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

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

1. In the case of regulatory agency personnel with oversight authority approval letters or e-mails will constitute verification that applicable Worksheets of the SAP have been reviewed. Copies of regulatory agency approval letters / e-mails will be retained in the project files and are listed in [Worksheet #29](#) as project records.

2. E-mails will be sent to Navy, Tetra Tech, and subcontractor project personnel whom will be requested to verify by e-mail that they have read the applicable SAP / Worksheets and the date on which they were reviewed. Copies of the verification e-mail will be included in the project files and identified in [Worksheet #29](#).

3. Signature and date will appear on [Worksheet #4](#).

A copy of the signed [Worksheet #4](#) will be retained in the project files and identified as a project document in [Worksheet #29](#).

Name	Title/Role	Telephone Number (Optional)	Signature/E-Mail Receipt	SAP Worksheets Reviewed	Date SAP Read
Navy and Regulator Project Team Personnel					
John Schoolfield	NAVFAC SE MRP Lead/ RPM / Manages Project Activities for Navy	(904) 542-6828		All	
Greg Campbell	NAS Pensacola Point of Contact (POC) / Manages Site Activities	(850) 452-3131 ext. 3007		All	

Name	Title/Role	Telephone Number (Optional)	Signature/E-Mail Receipt	SAP Worksheets Reviewed	Date SAP Read
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Tetra Tech Project Team Personnel

Ralph Basinski	Tetra Tech TOM / Manages Project Activities	(412) 921-8308		All	
Kelly Carper	Tetra Tech Quality Assurance Manager (QAM) / Manages Corporate QA Program and Implementation	(412) 921-7273		All	
Edward Sedlmyer	Tetra Tech Project Chemist / Provides Technical Coordination with Laboratories	(412) 921-8704		Worksheets #12, #14, #15, #19, #20, #23-28, #30, #34-37	
Jim Goerd	Tetra Tech FOL / Manages Field Operations	(412) 921-8425		All	
TBD	Tetra Tech Field Staff	(xxx) xxx-xxxx		Worksheets #2, #5, #6, #7, #8, #10, #11, #14, #17-22, #26, #27, #29	
Matt Soltis	Tetra Tech Health and Safety Manager (HSM) / Manages Corporate Health and Safety Program	(412) 921-8912		Health and Safety Plan (HASP) ⁽¹⁾	
Joseph Samchuck	Tetra Tech Data Validation Manager (DVM) / Manages Data Validation	(412) 921-8510		Worksheets #12, #14, #15, #19, #20, #23-28, #30, #34-37	

Subcontractor Personnel

Brian Richard	Laboratory Project Manager Empirical Laboratories / Representative for Laboratory and Analytical Issues	(615) 345-1115 X249		Worksheets #12, #14, #15, #19, #20, #23-28, #30, #34-37	
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1. The HASP is a stand-alone document, which is provided to the Navy under separate cover.

SAP Worksheet #6 -- Communication Pathways

[\(UFP-QAPP Manual Section 2.4.2\)](#)

The communication pathways for the Sampling and Analysis Plan (SAP) are shown below.

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
Regulatory Agency Interface	USEPA RPM FDEP RPM Navy RPM	Tim Woolheater Dave Grabka John Schoolfield	404-562-8510 850-245-8997 904-542-6418	The Navy RPM will contact the regulatory agency via phone and/or e-mail within 24 hours of recognizing the issue whenever issues arise.
Field Progress Reports	Tetra Tech FOL Tetra Tech TOM	Jim Goerdts Ralph Basinski	412-921-8425 412-921-8308	The Tetra Tech FOL will contact the Tetra Tech TOM on a daily basis via phone, and submit progress summaries daily via e-mail.
Gaining site access	Tetra Tech FOL NAS Pensacola ERSM	Jim Goerdts Greg Campbell	412-921-8425 850-452-3131 x3007	The Tetra Tech FOL shall contact the NAS Pensacola ERSM verbally or via e-mail at least 3 days prior to commencement of field work to arrange for access to the site for all field personnel.
Obtaining utility clearances	Tetra Tech FOL	Jim Goerdts	412-921-8425	The Tetra Tech FOL shall submit a NAS Pensacola Excavation Permit to the NAS Pensacola ERSM at least 10 days prior to commencement of field work and also contact Florida One-Call at least 3 days prior to commencement of field work to complete a utility clearance ticket for the area(s) under investigation.

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
Stop Work due to Safety Issues	Tetra Tech FOL/Site Safety Officer (SSO) Tetra Tech TOM Tetra Tech HSM Navy RPM NAS Pensacola ERSM	Jim Goerd Ralph Basinski Matt Soltis John Schoolfield Greg Campbell	412-921-8425 412-921-8308 412-921-8912 904-542-6418 850-452-3131 x3007	If Tetra Tech is the responsible party for a stop work command, the Tetra Tech FOL will inform onsite personnel, subcontractor(s), the NAS Pensacola Site Manager, and the identified Project Team members within 1 hour (verbally or by e-mail). If a subcontractor is the responsible party, the subcontractor PM must inform the Tetra Tech FOL within 15 minutes, and the Tetra Tech FOL will then follow the procedure listed above.
Sampling and Analyses Plan (SAP) Changes prior to Field/Laboratory Work	Tetra Tech FOL/SSO Tetra Tech TOM Navy RPM USEPA RPM FDEP RPM	Jim Goerd Ralph Basinski John Schoolfield Tim Woolheater Dave Grabka	412-921-8425 412-921-8308 904-542-6418 404-562-8510 850-245-8997	The Tetra Tech TOM will document the proposed changes via a Field Task Modification Request (FTMR) form (see Appendix C) within 5 days and send the Navy RPM a concurrence letter within 7 days of identifying the need for change if necessary. SAP amendments will be submitted by the Tetra Tech TOM to the Project Team (including the USEPA and FDEP RPMs) for review and approval via e-mail within 1 business day.

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
SAP Changes in the Field	Tetra Tech FOL/SSO Tetra Tech TOM Navy RPM NAS Pensacola ERSM USEPA RPM FDEP RPM	Jim Goerd Ralph Basinski John Schoolfield Greg Campbell Tim Woolheater Dave Grabka	412-921-8425 412-921-8308 904-542-6418 850-452-3131 x3007 404-562-8510 850-245-8997	<p>The Tetra Tech FOL will verbally inform the Tetra Tech TOM on the day that the issue is discovered. The Tetra Tech TOM will inform the Navy RPM and the NAS Pensacola ERSM (verbally or via e-mail) within 1 business day of discovery. Significant SAP changes will be communicated to regulators prior to implementation.</p> <p>The Navy RPM will issue a scope change (verbally or via e-mail), if warranted. The scope change is to be implemented before further work is executed.</p> <p>The Tetra Tech TOM will document the change via an FTMR form within 2 days of identifying the need for change and will obtain required approvals within 5 days of initiating the form.</p>
Field Corrective Actions	Tetra Tech TOM Tetra Tech QAM Navy RPM USEPA RPM FDEP RPM	Ralph Basinski Kelly Carper John Schoolfield Tim Woolheater Dave Grabka	412-921-8308 412-921-7273 904-542-6418 404-562-8510 850-245-8997	<p>The Tetra Tech QAM will notify the Tetra Tech TOM verbally or by e-mail within one business day that the corrective action has been completed. The Tetra Tech TOM will then notify the Navy RPM (verbally or by e-mail) within 1 business day. Significant corrective actions will be communicated to regulators prior to implementation.</p>

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathway To/From, etc.)
Analytical Corrective Actions	Laboratory PM Tetra Tech Project Chemist Tetra Tech DVM Tetra Tech TOM Navy RPM	Brian Richard Ed Sedlmyer Joseph Samchuck Ralph Basinski John Schoolfield	615-345-1115 Ext. 249 412-921-8704 412-921-8510 412-921-8308 904-542-6418	<p>The Laboratory PM will notify (verbally or via e-mail) the Tetra Tech Project Chemist within 1 business day of when an issue related to laboratory data is discovered.</p> <p>The Tetra Tech Project Chemist will notify (verbally or via e-mail) the DVM and the Tetra Tech TOM within 1 business day.</p> <p>Tetra Tech DVM or Project Chemist notifies Tetra Tech TOM verbally or via e-mail within 48 hours of validation completion that a non-routine and significant laboratory quality deficiency has been detected that could affect this project and/or other projects. The Tetra Tech TOM verbally advises the – Navy RPM within 24 hours of notification from the Tetra Tech Project Chemist or DVM. The Navy RPM takes corrective action appropriate for the identified deficiency. Examples of significant laboratory deficiencies include data reported that have a corresponding failed tune or initial calibration verification. Corrective actions may include a consult with the Navy Chemist.</p>

SAP Worksheet #7 -- Personnel Responsibilities and Qualifications Table

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

Name	Title/Role	Organizational Affiliation	Responsibilities
John Schoolfield	RPM	NAVFAC SE	Oversees project, scoping, data review, and evaluation.
Greg Campbell	NAS Pensacola Site Manager	NAVFAC SE	Serves as the on-site point of contact and oversees project, scoping, data review, and evaluation.
Dave Grabka	FDEP RPM	FDEP	Participate in scoping, data review, and evaluation and serves as FDEP PM.
Tim Woolheater	USEPA RPM	USEPA Region IV	Participate in scoping, data review, and evaluation and serves as USEPA Region IV PM.
Ralph Basinski	TOM	Tetra Tech	<p>Oversees project, financial, schedule, and technical day to day management of the project.</p> <ul style="list-style-type: none"> • Ensures timely resolution of project-related technical, quality, and safety questions associated with Tetra Tech operations. • Functions as the primary Tetra Tech interface with the Navy RPM, Tetra Tech field and office personnel, and laboratory points of contact. • Ensures that Tetra Tech health and safety issues related to this project are communicated effectively to all personnel and off-site laboratories. • Monitors and evaluates all Tetra Tech subcontractor performance. • Coordinates and oversees work performed by Tetra Tech field and office technical staff (including data validation, data interpretation, and report preparation). • Coordinates and oversees maintenance of all Tetra Tech project records. • Coordinates and oversees review of Tetra Tech project deliverables. • Prepares and issues final Tetra Tech deliverables to the Navy.

Name	Title/Role	Organizational Affiliation	Responsibilities
Jim Goerdts	FOL, Site Safety Officer (SSO)	Tetra Tech	<p>Supervises, coordinates, and performs field sampling activities</p> <ul style="list-style-type: none"> • Ensures that all health and safety requirements unique to the SI are implemented. • Functions as the on-site communications link between field staff members, NAVFAC SE, and the Tetra Tech TOM. • Alerts off-site analytical laboratories of any special health and safety hazards associated with environmental samples. • Oversees the mobilization and demobilization of all field equipment and subcontractors. • Coordinates and manages the field technical staff. • Adheres to the work schedules provided by the Tetra Tech TOM. • Ensures the proper maintenance of site logbooks, field logbooks, and field recordkeeping. • Initiates FTMRs (field change orders) when necessary. • Identifies and resolves problems in the field, resolving difficulties via consultation with the OLF Saufley POC and the NAVFAC SE RPM, implementing and documenting corrective action procedures, and providing communication between the field team and project management.
Kelly Carper	QAM	Tetra Tech	<p>Reviews SAP, oversees preparation of laboratory scope, coordinates with laboratory, and data quality review. Ensures quality aspects of the CLEAN program.</p> <ul style="list-style-type: none"> • Develops, maintains, and monitors quality assurance (QA) policies and procedures. • Provides training to Tetra Tech staff in QA/Quality Control (QC) policies and procedures. • Conducts systems and performance audits to monitor compliance with environmental regulations, contractual requirements, QAPP requirements, and corporate policies and procedures. • Audits project records. • Monitors subcontractor quality controls and records. • Assists in the development of corrective action plans and ensuring correction of non-conformances reported in internal or external audits. • Ensures that this SAP meets Tetra Tech, Navy, USEPA, and FDEP requirements. • Oversees the responsibilities of the Tetra Tech Project QA/QC Advisor. • Prepares QA reports for management.

Name	Title/Role	Organizational Affiliation	Responsibilities
Matt Soltis	HSM	Tetra Tech	Oversees CLEAN Program Health and Safety Program <ul style="list-style-type: none">• Provides technical advice to the Tetra Tech TOM on matters of health and safety.• Oversees the development and review of the Health and Safety Plan (HASP).• Conducts health and safety audits.• Prepares health and safety reports for management.

Name	Title/Role	Organizational Affiliation	Responsibilities
Edward Sedlmyer	Project Chemist	Tetra Tech	<p>Coordinates analyses with laboratory chemists, ensures the scope is followed, reviews data packages, and communicates with Tetra Tech staff.</p> <ul style="list-style-type: none"> • Ensures that the project meets objectives from the standpoint of laboratory performance • Provides technical advice to the Tetra Tech team on matters of project chemistry. • Monitors and evaluates subcontractor laboratory performance. • Ensures timely resolution of laboratory-related technical, quality, or other issues affecting project goals. • Functions as the primary interface with the subcontracted laboratory and the Tetra Tech TOM. • Coordinates and oversees work performed by the subcontracted laboratory. • Oversees the completion of Tetra Tech data validation. • Coordinates and oversees review of laboratory deliverables. • Recommends appropriate laboratory corrective actions.
Joseph Samchuck	DVM	Tetra Tech	<ul style="list-style-type: none"> • Oversees data validation activities • Serves as communication link between Tetra Tech and laboratories on data validation and electronic data positing activities. • Establishes Tetra Tech data validation protocols in support of projects
Brian Richard	Laboratory PM	Empirical Laboratories	<p>Coordinates analyses with laboratory chemists, ensures that scope of work is followed, reviews data packages, and communicates with Tetra Tech staff.</p>

SAP Worksheet #8 -- Special Personnel Training Requirements

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

Project Function	Specialized Training by Title or Description of Course	Training Provider	Training Date	Personnel / Groups Receiving Training	Personnel Titles / Organizational Affiliation	Location of Training Records / Certificates
Field Technicians	40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) 8-hour HAZWOPER Refresher	Various	Current	Field sampling personnel	All field staff / 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
Health and Safety Officer	First Aid / Cardiopulmonary Resuscitations Training	Red Cross	Current	Field Personnel	Tetra Tech	Tetra Tech project office and field office
UXO Avoidance	Various training elements, as required in DoD Explosive Safety Board (DDESB) Technical Paper (TP)-18 ⁽¹⁾	DoD or other approved formal course	Current	UXO Technicians supporting UXO avoidance	UXO Technician/ 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 as described under Occupational Safety and Health Administration (OSHA) 29 Code of Federal Regulations (CFR) 1910.120(b)(4).

SAP Worksheet #9 -- Project Scoping Session Participants Sheet

(UFP-QAPP Manual Section 2.5.1)

Project Name: NAS Pensacola Munitions Constituents (MC) Site Projected Date(s): September 2011 Task Order Manager: Ralph Basinski		Site Name: Magazine Point Bombing Target - Naval Air Station (NAS) Pensacola Site Location: NAS Pensacola, Florida			
Date of Session: July 29, 2011 Scoping Session Purpose: Pre-DQO scoping internal meeting					
Name	Title	Affiliation	Phone #	E-mail Address	Project Role
John Schoolfield	Navy RPM	NAVFAC SE	904-543-3991	john.schoolfield1@navy.mil	Navy oversight
David Grabka	RPM	FDEP	850-245-8997	david.grabka@dep.state.fl.us	FDEP oversight
Julie Corkran	RPM	USEPA	404-562-8547	corkran.julie@epa.gov	EPA oversight
Terry Zdon	EPA Consultant	Tech Law	404-562-8547	tzdon@techlawinc.com	EPA consultant
Ralph Basinski	TOM	Tetra Tech	412-621-8308	ralph.basinski@tetrattech.com	Task Order Manager
Amber Igoe	Environmental Scientist	Tetra Tech	850-385-9866	amber.igoe@tetrattech.com	Scribe

Background:

According to the SI, the Magazine Point Bombing Target site was first denoted on a 1933 historical map, but was not shown on a 1939 historical map, indicating the period of use was approximately the early 1930s. It is believed that only practice bombs were used at the site. During the initial SI, a detector-aided surface survey was conducted on 100 percent of the accessible site. No MEC or other munitions debris was observed

during the investigation. Limited sampling at the site was recommended in the SI Report (Tetra Tech, 2010) to ensure no munitions constituents (MC) (i.e., metals and explosives) associated with the practice bombs used at the site are present in the soil.

Action Items:

- 1) Determine appropriate sampling methodology
- 2) Determine constituents to be analyzed
- 3) Decide on location of sample area

Comments/Decisions:

- 1) Consensus was reached by the Project Team that the best soil sampling approach would be the utilization of an Incremental Sampling Methodology.
- 2) Because of the relatively small size of the target area to be sampled (approximately 1 acre), a single Decision Unit was identified.
- 3) Prior to sample collection, a trained UXO technician will clear the area. A portion of the area has been covered with concrete construction material to prevent beach erosion and will limit the use of the hand-held metal detector (i.e.: Schonstedt). The UXO Technician will perform a visual inspection in this area.
- 4) The soil samples will be analyzed for RCRA 8 metals and explosives to confirm that explosives were not used in the practice bombs. A separate metals sample will be collected prior to the grinding step.
- 5) The sample collection depth will be 0 to 6 inches below ground surface (bgs) unless instructed otherwise, and the proposed number of increment samples is a minimum of 30 and a maximum of 100 (approximately 60).

Note: Subsequent to the meeting, a review of the munitions ordnance data sheets for potential metallic constituents resulted in a revised list of metals as presented in [Worksheet #15](#).

SAP Worksheet #10 – Conceptual Site Models

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

10.1 SITE DESCRIPTION

The Magazine Point Bombing Target is located at Naval Air Station (NAS) Pensacola in Pensacola, Florida (**Figure 1**). The entire Magazine Point Bombing Target site occupies a 72-acre area located on Magazine Point Peninsula. The bombing target center was located within the boundaries of the main NAS Pensacola installation (**Figure 2**). The Magazine Point Bombing Target was first denoted on a 1933 historical map as a chevron-shaped target located adjacent to Pensacola Bay along with one powder magazine and a radio spotting system (**Figure 3**). The Magazine Point Bombing Target was not shown on a 1939 historical map, indicating the period of use for the Magazine Point Bombing Target was approximately the early 1930s. No additional archival records or references to the range were located during the Preliminary Assessment (PA) that indicated munitions used or construction details. The location of the former Magazine Point Bombing Target site currently encompasses the waste water treatment plant (WWTP) located northwest of the chevron-shaped bombing target (indicated by circular structures on **Figure 3**), undeveloped areas of Magazine Point, and a portion of Pensacola Bay. Based on the location of the bombing target in relation to Chevalier Field (**Figures 2 and 3**), it is likely that the Magazine Point Bombing Target site was used as a practice bombing range where practice bombs were dropped from aircraft with the intent of hitting a target on the ground.

Site 13 (Rubble Disposal Area) is located along the eastern shoreline of Magazine Point, and a portion of it bisects the Bombing Target Area. The Site consists of scattered dumped rubble, concrete, asphalt, and shore-washed debris.

A remedial investigation has been conducted at Site 13. The Final Remedial Investigation Report (RIR), dated September 1995, states that the Site 13 soil conditions were characterized by generally non-detect organic concentrations with isolated detections of PAHs and phenols, and non-detect to trace concentrations of heavy metals. The RIR concluded that additional assessment work at Site 13 was not required, given the low detected concentrations in soil and no risk related pathways. Moreover, contaminants were absent in groundwater.

A letter of concurrence regarding No Further Action was received from the U.S. EPA Region IV on June 21, 1996, and a letter of concurrence regarding approval of the Final RIR was received from the Florida Department of Environmental Protection on August 14, 1996.

The location of Site 13 within the Bombing Target Area does not affect the current sampling plan.

The Magazine Point Bombing Target is located in a relatively flat area. It gently slopes to the north towards a bayou that flows into Bayou Grande and to the east to Pensacola Bay ([Figure 2](#)). Magazine Point is a peninsula which has little natural relief. No natural surface water features were noted in the Magazine Point area, although various drainage ditches and storm water detention ponds are present. The drainage ditches are located primarily around buildings and developed areas. A portion of Pensacola Bay is included within the site boundary. Surface water runoff from the site drains directly into Pensacola Bay. A portion of the site is located within a 100-year floodplain that occurs along Pensacola Bay. Depth to groundwater at Building 3644 was measured at approximately 5 to 7 feet below ground surface (bgs); however, Building 3644 is located 1,300 feet west of the Bombing Target and groundwater may be closer to the surface at the site given its proximity to Pensacola Bay.

Whereas proximity to Chevalier Field probably limited munitions used at the site to practice bombs with inert fillers, those bombs may have included spotting charges, which would be defined as munitions and explosives of concern (MEC). Therefore, as recently as 2009, the area within the 500-foot scoring arc was thought to be an area potentially containing MEC.

10.2 SITE INSPECTION RESULTS

A visual survey of the area around the Magazine Point Bombing Target was conducted on November 30, 2007 as part of the PA. The area from the shoreline of Pensacola Bay to the heavy vegetation line adjacent to the WWTP fence was visually surveyed. Much of the area was covered with various storm debris washed onto the shore. Concrete and asphalt pieces were also present in this area. These materials have been deliberately placed there for shoreline stabilization. A large mound of dirt was observed just south of the Bombing Target; the history and use of the mound is unknown. The heavily vegetated area included thick growth of slash pines, vines, and various shrubs such as saltbrush. Because of inaccessibility, the heavily vegetated area and the area inside the WWTP were not surveyed. Tetra Tech personnel conducted a site walk on March 27, 2009 as part of a Site Inspection (SI) and confirmed the observations from the PA. No MEC, munitions potentially presenting an explosive hazard (MPPEH), or related debris was observed during either the PA or SI visual surveys.

An SI was conducted at the Magazine Point Bombing Target in February 2010. Survey transect lines were established using hand-held global positioning system (GPS) units. Survey areas were divided into approximately 100-foot by 100-foot survey grids, and temporary markings (e.g., plastic flagging, non-metallic pin flags, etc.) were used to mark locations of transects for vegetation management and surveying. The grids were further divided into 5-foot wide survey lanes ensuring maximum coverage with the survey instrument. A Schonstedt GA-52Cx was used as the primary survey instrument to conduct the surveys. Given the nature of the site and its known use of mostly ferrous munitions, this was the most appropriate technology for this operation based on industry standards. In addition to the Schonstedt

instrument, a White's Spectrum XLT all-metals detector was used in surface survey areas to assist in the location of metal targets with little or no ferrous metal content.

The metal detectors used by the Unexploded Ordnance (UXO) Team during the detector-aided surface survey had a detection depth that was limited by the size and orientation of the target and soil characteristics of the work area. The UXO Team completed the detector-aided surface survey over a 5-day period. Approximately 20 percent of the total accessible area at the Magazine Point Bombing Target site was surveyed every day, and 100 percent coverage of the targeted area was achieved.

The primary objective of the SI was to determine whether further response actions or a Remedial Investigation (RI) was appropriate to restore the site to an acceptable environmental condition. During the SI, the background information provided in the PA Report was evaluated. In addition, supplemental site-specific environmental data were collected to determine types and rough orders of magnitude of MEC quantities at the site, and to refine site boundaries (footprint reduction).

Although no detections of MEC, MPPEH, or munitions debris (MD) were noted at the Magazine Point Bombing Target during the geophysical investigation, limited sampling at the site was recommended in the SI Report (Tetra Tech, 2010) to confirm that no MC (i.e., metals) associated with the practice bombs used at the site are present in the soil, and to verify that explosives (i.e., TNT, RDX, HMX, etc.) were not present in bombs dropped at the Magazine Point Bombing Target site.

10.3 CONCEPTUAL SITE MODEL (CSM)

The results of the February 2010 SI indicate that MEC have not been detected and are not expected to be present at the site. Practice bombs do not contain explosive fillers; however, MC can originate from small quantities of black powder or pyrotechnics potentially contained in the bomb casings to serve as spotting charges. These spotting charges provide visual indication of a bomb impacting the ground.

Practice bombs were filled with inert fillers such as sand or concrete in place of explosives. However, practice bombs containing small spotting charges, which would have been discharged on impact to provide a visual means of identifying the impact location. These charges are a potential source of MC contamination. Black powder (a mixture of charcoal, potassium nitrate, and sulfur) and pyrotechnic formulations (some of which may have contained black powder), may have been used as signaling or spotting charges. The charcoal (i.e., wood and carbon) and sulfur components of black powder are relatively inert and pose no human health threat. Potassium nitrate is very soluble in water and is expected to have been washed away by storm surges or precipitation. Some bombs may have contained titanium tetrachloride. After release from a bomb, titanium tetrachloride reacts with water to form hydrochloric acid and titanium dioxide, a relatively inert, white mineral commonly used as paint pigment.

Other chemicals used in components of practice bombs include chlorates, red phosphorus, and nitrocellulose. It is expected that chlorates, which are generally very soluble in water, have been washed out of the soil and are not present at environmentally significant concentrations. Neither red phosphorus nor nitrocellulose represents a significant environmental hazard. The phosphorus is oxidized to phosphate over time and forms phosphate salts with metals in soil, many of which are insoluble in water except at very low concentrations. Nitrocellulose is relatively insoluble. Phosphates and nitrocellulose are essentially non-toxic, in part because of their low solubility, and do not represent a significant human health risk.

Metals commonly associated with practice bombs are components of spotting charges, and components and alloying elements of the metal bomb shells. These metals are copper and zinc in the shotgun cartridges of spotting charges; iron in the bomb casings, and trace metals which are present in iron including cadmium, chromium, manganese, molybdenum, nickel, and vanadium. Another metal commonly associated with munitions is lead, although lead is not necessarily a component of steel practice bombs.

The release of chemicals at the Magazine Point Bombing Target occurred primarily to the air with the discharge of the spotting charges, and to surface soil as metal practice bomb shells and shell fragment. Metals are the contaminants most likely to have been released; however, there is a chance that live bombs were used on occasion. The use of live bombs could have released nitramine or nitroaromatic explosives to the air and to surface and shallow subsurface soil.

MC released to the air would have settled onto the ground surface. Therefore, most MC contamination at the Magazine Point Bombing Target is expected to be located in shallow surface soil. The spatial distribution of MC concentrations is likely to be very heterogeneous. If MC are present, migration may occur via surface soil erosion or by human activities (including mowing, grading, or other site work). MC can also percolate into deeper soil under the influence of precipitation; organic explosive compounds and the more mobile metals would be the most likely to percolate. Future construction, excavation, and maintenance workers at the site could be exposed to MC, especially MC in surface soil where the MC concentrations are expected to be the greatest. At the Magazine Point Bombing Target site, MC are most likely to be transported into Pensacola Bay as the result of storm surges that cause the water level to rise up over normally dry land. Construction debris has been deposited over a large portion of the site to prevent shoreline erosion; this may have resulted in the release of contaminants. It is impractical to distinguish construction debris metal contamination from practice bomb contamination. Organic explosive compounds do not occur naturally, and are very unlikely to have been components of the debris. Metals, however, do occur naturally in soil and can leach from the soil into groundwater. A background soil investigation at NAS Pensacola determined that soil background concentrations of metals likely to be

associated with practice bombs do not exceed standards for acceptable exposure of humans to soil. The background concentrations are also less than FDEP leachability criteria; exceedances of these criteria indicate that leaching to groundwater presents a potential threat to groundwater quality.

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

[\(UFP-QAPP Manual Section 2.6.1\)](#)

11.1 PROBLEM STATEMENT

The Project Team must address the two problem statements described below.

Problem Statement #1:

The Project Team plans to collect and analyze milled incremental soil samples which should better represent site conditions. The Project Team must therefore also confirm that available NAS Pensacola background metals concentrations data are representative of site soil that is not contaminated, and that they can be accurately compared to metals concentrations data obtained from milled incremental soil samples. NAS Pensacola background concentrations of metals in soil are available; however, they were obtained from the analysis of discrete (unmilled) soil samples. For some metals, milling can increase the detected metals concentration because milling releases metals that occur naturally in the soil. Detected background concentrations of these metals in milled samples would appear to be greater than the NAS Pensacola background concentrations obtained from discrete samples, even when there is no real difference.

Therefore, the Project Team will compare the metals concentrations from milled incremental soil samples at the site to the metals concentrations obtained from unmilled incremental soil samples from the same sample locations. If this comparison indicates that milling increases the metal concentrations relative to unmilled samples, the team will conclude that the currently available NAS Pensacola metals background data, which are based on unmilled discrete samples, are not truly representative of background and will collect new background data from milled samples. If milling does not increase metal concentrations in the soil samples, the currently available NAS Pensacola background metal concentrations will be considered acceptable. The soil background data that are concluded to best represent background metal concentrations will be used as the project action limits (PALs) if the metals concentration exceeds a risk-based concentration (see Problem Statement #2).

Problem Statement #2:

Malleable metals, such as aluminum, copper, and lead could smear in the grinding chamber during the milling process if a significant amount of larger particle size metal particles are present in the ISM samples. This could result in low results in some samples and carryover issues in other samples. The Project Team will review field and laboratory observations regarding the presence of metallic particles and compare the concentrations of aluminum, copper and lead in the triplicate samples. If this evaluation

indicates that smearing effects may be occurring, additional sieving and fractional analysis will be considered or alternate sample preparation techniques investigated.

Problem Statement #3:

MC that are potentially present in surface soil from historical bombing practices could pose an unacceptable level of risk to human receptors. The human receptors that could potentially be exposed to contamination are: future construction, maintenance, and site occupational workers; and hypothetical future residents. Exposure pathways of significance for these receptors to MC in surface soil are:

- Future construction workers: incidental ingestion, inhalation, and dermal contact.
- Maintenance workers: ingestion, inhalation, and dermal contact.
- Site occupational workers: incidental ingestion and inhalation.
- Hypothetical future residents: incidental ingestion, inhalation, and dermal contact.

The hypothetical future residents would experience the most frequent exposure and their exposure durations would likely be the longest per exposure; therefore, the residents are considered to be the most sensitive of these receptors. The Project Team will determine whether concentrations of select MC as identified in the CSM could pose an unacceptable level of risk to any of the receptors by comparing site data to PALs (described in [Worksheet #11](#)). Responses will be initiated based on sample results, unacceptable risks may be evaluated in greater detail, or options for reducing the risk to a level that is protective of human health may be recommended.

11.2 DECISION INPUTS

To address Problem Statements #1 and #2 presented in [Worksheet #11](#), the following data are needed:

- Mean concentrations of potential site MC metals (see [Worksheet #15](#)) obtained without milling of soil samples. These data will be compared to milled site soil metals concentrations to address Problem Statement #1.
- Mean concentrations of potential MC (select metals and explosives) at the Magazine Point Bombing Target (from milled samples). The list of surface soil MC that are potentially site-related (based on the CSM of [Worksheet #10](#)) are presented in [Worksheet #15](#). Incremental sampling with milling (grinding) is required to obtain the mean site concentrations with minimal data variability (while minimizing analytical costs) to address Problem Statement #2.

- Project Action Levels (PALs). Comparison of site MC concentrations to PALs will determine whether MC concentrations in surface soil are high enough to pose a potentially unacceptable level of human health risk (Problem Statement #2). For all background metal concentrations that are greater than risk-based PALs, the risk-based PAL will be replaced with the background metal concentration as determined by resolution of Problem Statement #1.
- Location data for sampling points (Problem Statements #1 and #2). Vertical elevation measurements are not required. Because incremental sampling is planned, the vertices of the grid over which incremental samples are collected must be documented. The centroid of these vertices must also be documented. Use of a GPS with sub-meter accuracy (or better) is sufficient for this. The coordinates will be documented in the Florida North State Plane, NAD 83.

Note: Background MC concentrations are not needed for organic explosives because these chemicals do not occur naturally in soil. The metals targeted for this investigation are identified in [Worksheet #15](#).

11.3 STUDY BOUNDARIES

The area that represents the greatest potential human health risk is the center line and surrounding area of the bombing target because most bombs would have landed there, and most MC releases would have occurred there. An Sample Unit approximately 1 acre in size oriented along the bombing target center line represents the greatest exposure risk for the most sensitive receptor: the hypothetical future resident. Therefore, the soil population that must be represented by mean MC concentrations is the Magazine Point Bombing Target soil within the top 6 inches of the ground surface in this area.

For the comparison described in Problem Statement #2 to be valid, the soil MC data obtained from milled and unmilled samples must be as comparable as is reasonably possible. To achieve this, the unmilled soil must be composited from the same samples included in the milled incremental samples. Milled samples may exhibit higher metals concentrations than the unmilled samples as a result of milling; therefore, the comparison to screening criteria may be conservative in that it would tend to indicate an exceedance of criteria than would be indicated by data from unmilled samples.

Vegetation and rocks/sticks, etc. with a grain size of 2 millimeter (mm) or larger should be removed as much as practicable prior to subsampling in the laboratory because grain sizes less than 2 mm represent the greatest human health risk for ingestion and inhalation.

11.4 ANALYTIC APPROACH

The following decision rule will be used to address Problem Statement #1 presented in [Worksheet #11](#):

If any mean site metal concentration obtained from milled site incremental samples exceeds the mean site metal concentration from unmilled site incremental samples by more than 20 percent, conclude that the milled sample and unmilled sample data are not comparable and collect a milled sample background metal data set for the affected metals; otherwise, use the available NAS Pensacola unmilled background metal concentrations (obtained from discrete samples) to represent background metal concentrations

The following decision rule will be used to address Problem Statement #2 presented in [Worksheet #11](#):

If field and laboratory data indicate that aluminum, lead or copper metallic particles may be present, and comparisons of concentrations of these metals indicate that smearing effects may be occurring, conclude that smearing effects may be occurring. Therefore additional samples must be collected, and additional sieving and fractional analysis or alternate sample collection techniques will be considered.

The following decision rule will be used to address Problem Statement #3 presented in [Worksheet #11](#):

If the mean surface soil concentration (based on milled incremental site samples) of any of the select metal or explosive MC target analytes exceeds its PAL (see [Worksheet #15](#)), then convene the Project Team to evaluate whether to recommend conducting an immediate response action to reduce risks quickly, or further evaluation to more accurately estimate risks; otherwise, recommend no further action.

The Project Team will generally recommend more immediate or aggressive actions as the concentrations of target analytes increase relative to PALs and background concentrations.

SAP Worksheet #12 -- Measurement Performance Criteria Table Field Quality Control Sample – All fractions

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

Measurement Performance Criteria Table – Field QC Samples

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 Replicates ⁽¹⁾	All fractions	One per grinding batch	Representativeness	Percent Relative Standard Deviation (RSD) of $\leq 20\%$	S
Cooler Temperature Indicator	All analytical groups	One per cooler	Representativeness	Temperature must be between 0 and 6 degrees Celsius ($^{\circ}\text{C}$).	S&A

(1) One duplicate and one triplicate sample are collected for each type of activity.

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
Preliminary Assessment	Malcolm Pirnie/Preliminary Assessment Additional Areas of Concern Naval Air Station Pensacola, Florida , August 2009	Malcolm Pirnie	Basis for UFP-SAP, Site History, and CSM	The information is qualitative and no quantitative (site-specific nature and extent of contamination) information is available. The information was used to establish the field work program and identify the area most likely to be contaminated.
Site Inspection Report	Tetra Tech/Site Inspection Report for Munitions Response Program Site Inspections at 13 Sites Naval Air Station Pensacola, Pensacola and Outlying Landing Fields Bronson, Corry Station, and Saufley Field, September 2010	Tetra Tech/Detector-aided Surface Survey and Geophysical Survey/February and March 2010	Determine Decision Units	None

SAP Worksheet #14 -- Summary of Project Tasks

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

14.1 FIELD PROJECT TASKS

Site-specific SOPs have been developed for the proposed field activities at NAS Pensacola (Magazine Point Bombing Target) and are located in [Appendix A](#). Field tasks are summarized below with a short description for each task.

- Mobilization / Demobilization
- Excavation Permit / Utility Clearance
- Site-Specific Health and Safety Training
- Sample Collection Tasks
- Incremental Soil Sampling
- GPS Locating
- Investigation-Derived Waste Management
- Field Equipment Decontamination
- Field Documentation Procedures
- Sample Handling
- QC Tasks

Mobilization / Demobilization

Mobilization will consist of: the delivery of all equipment, materials, and supplies to the site; complete assembly in satisfactory working order of all such equipment at the site; and satisfactory storage at the site of all such materials and supplies. The Tetra Tech FOL or designee will coordinate with the NAS Pensacola Site Manager to identify appropriate locations for the storage of equipment and supplies. Site-specific health and safety training for all Tetra Tech field personnel and subcontractors will be provided as part of site mobilization.

Demobilization will consist of the prompt and timely removal of all equipment, materials, and supplies from the site following completion of the work. Demobilization includes the cleanup and removal of waste generated during the performance of the investigation.

Excavation Permit / Utility Clearance

At least 10 days prior to commencement of any intrusive activities, the Tetra Tech FOL or designee will submit a completed NAS Pensacola Excavation Permit to the ERSM for processing. Also, at least 3 days prior to commencement of any intrusive activities, the Tetra Tech FOL or designee will contact the Florida

Sunshine One-Call to complete a utility clearance ticket for the area under investigation. Work permits, if required by the facility, will be obtained prior to conducting field activities. The Tetra Tech FOL will be responsible for coordinating these activities.

Site-Specific Health and Safety Training

The UXO Technicians who will be responsible for UXO avoidance must meet the specialized training requirements specified in DDESB TP-16 (see [Worksheet #8](#)). The SSO must have first aid and cardiopulmonary resuscitation training. There are no other specialized/non-routine project-specific training requirements or certifications needed by personnel to successfully complete the project or tasks. All field personnel will have appropriate training to conduct the field activities to which they are assigned. Each site worker will be required to have completed the Occupational Safety and Health Administration (OSHA) 40-hour course (and 8-hour refresher, if applicable) in health and safety training. All field crews will be required to attend a short (less than 30 minutes) safety briefing to be conducted by the on-site UXO Technician. Safety requirements are addressed in greater detail in the site-specific Health and Safety Plan (HASP).

Sample Collection Tasks

The sampling and analysis program is outlined in [Worksheet #17](#) and [Worksheet #18](#). Sample collection will be in accordance with the site-specific SOPs listed in [Worksheet #21](#) and provided in [Appendix A](#). The sampling requirements for each type of analysis (i.e., bottleware, preservation, holding time) are listed in [Worksheet #19](#).

Incremental Soil Sampling

The incremental sampling methodology will be utilized during this field sampling event. A total of three incremental samples will be collected: the original sample, a duplicate sample, and a triplicate sample. Individual increments will be collected from the surface soil (0 to 6-inches bgs) in accordance with [SOP-05](#) (Incremental Sampling, [Appendix A](#)). The individual increments that make up a single sample will be collected from the sample area grid consisting of 60 evenly sized cells.

Global Positioning System Locating

A GPS unit will be used to locate the four corners of the proposed Sampling Unit in accordance with [SOP-07](#) (Global Positioning System, [Appendix A](#)). The GPS equipment will be checked on control monuments before and after each day's use; these checks will be documented in the field notebook. To ensure sub-meter accuracy, the GPS SOP requires a minimum of six satellites to capture a position.

Investigation-Derived Waste (IDW) Management

Since all sample material collected within the sample probe is to be utilized in the incremental sample, no solid or semi-solid IDW in the form of soil will be generated during the field activities.

IDW generated will include personal protective equipment (PPE) and decontamination fluids, which will be handled in accordance with [SOP-08](#) (Management of Investigation-Derived Waste, [Appendix A](#)).

Field Decontamination Procedures

Decontamination of sampling equipment will not be necessary for dedicated and disposable hand trowels. Decontamination of reusable sampling equipment (e.g., soil probes) will be conducted prior to sampling and when switching between collection of the original sample and the duplicate or triplicate sample. Decontamination of equipment will be conducted according to the sequence established in [SOP-06](#) (Decontamination of Field Sampling Equipment, [Appendix A](#)).

Field Documentation Procedures

Field documentation will be performed in accordance with [SOP-03](#) (Sample Custody and Documentation of Field Activities, [Appendix A](#)).

Sample Handling

Methods for sample handling will be in accordance with [SOP-03](#) (Sample Custody and Documentation of Field Activities). Sample labeling will be in accordance with [SOP-01](#) (Sample Labeling), and the sample numbering scheme will be in accordance with [Worksheet #18](#) and [SOP-02](#) (Sample Identification and Nomenclature). The selection of sample containers, sample preservation, packaging, and shipping will be in accordance with [Worksheet #19](#) and [SOP-04](#) (Sample Preservation, Packaging, and Shipping). All above referenced SOPs can be found in [Appendix A](#) of this UFP-SAP.

Quality Control Tasks

QC samples in the form of a duplicate and a triplicate sample will be collected for analytical comparison.

ADDITIONAL PROJECT-RELATED TASKS

Additional project-related tasks will include:

- Analytical Tasks
- Data Management
- Data Tracking

- Data Storage, Archiving and Retrieval
- Data Security
- Electronic Data
- Data Review
- Project Reports

Analytical Tasks

Chemical analyses will be performed by Empirical, which is a Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP)-accredited and Florida accredited laboratory. Copies of Empirical accreditations are provided in [Appendix B](#). Analyses will be performed in accordance with the analytical methods identified in [Worksheet #19](#). Empirical Labs will perform the chemical analyses following laboratory-specific SOPs (see [Worksheet #19](#) and [Worksheet #23](#)) developed based on the methods listed in [Worksheet #19](#).

All soil results will be reported by the laboratory on an adjusted dry-weight basis. Results of percent moisture will be reported in each analytical data package and associated electronic data files. This information will also be captured in the project database, which will eventually be uploaded to the Naval Installation Restoration Information Solution (NIRIS) database. Percent moisture information will also be captured in the project report.

The analytical data packages provided by Empirical Labs will be in a Contract Laboratory Program-like format and will be fully validatable and contain raw data, summary forms for all sample and laboratory method blank data, and summary forms containing all method-specific QC [results, recoveries, relative percent differences (RPDs), relative standard deviations (RSD), and/or percent differences, etc.]. A summary of the analytical data deliverable elements follows:

Analytical Data Deliverable Elements	Explosives	Metals
Cover Title Page	X	X
Chain of custody forms (field and internal)	X	X
Sample receipt forms	X	X
Case Narrative	X	X
Summary data package (containing just CLP-like summary forms 1-15 as applicable)	X	X
Sample data (Tabulated summarized results and raw data)	X	X
Analytical sequence tabulated summary	X	X
Initial calibration tabulated summary	X	X
Continuing calibration tabulated summary	X	X
Raw data for all calibration standards	X	X

Analytical Data Deliverable Elements	Explosives	Metals
ICP Interelemental Correction Factors		X
ICP linear ranges summary		X
Standard preparation logbook pages	X	X
Surrogate Recovery tabulated summary	X	
MS/MSD Recovery tabulated summary	X	X
Method blank tabulated summary	X	X
Organic analyses QC raw data	X	
ICP interference check sample tabulated summary		X
Spike sample recovery tabulated summary		X
Post digestion spike sample recovery tabulated summary		X
Duplicate sample tabulated summary		X
Laboratory control sample tabulated summary	X	X
Standard additions results tabulated summary		X
ICP Serial Dilution tabulated summary		X
Inorganic analyses QC raw data		X
QC sample preparation logbook pages	X	X
Sample preparation Logs	X	X
Percent solids determination log	X	X

Additionally, when manual integrations are performed, raw data records shall include a complete audit trail for those manipulations (i.e., the chromatograms obtained before and after the manual integration must be retained to permit reconstruction of the results). This requirement applies to all analytical runs including calibration standards and QC samples. The person performing the manual integration must sign and date each chromatogram and document the rationale for performing manual integration (electronic signature is acceptable). Records for manual integrations may be maintained electronically as long as all requirements, including signature requirements, are met and the results can be historically reconstructed.

Data Management

The principal data generated for this project will be from field data and laboratory analytical data. Field sampling log sheets will be organized by date and medium, 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 Empirical will meet the requirements of the technical specifications. Electronic data results will be automatically downloaded into the Tetra Tech database in accordance with the proprietary Tetra Tech processes.

The Tetra Tech TOM (or designee) is responsible for the overall tracking and control of data generated for the project.

Data Tracking

Data are tracked from generation to archiving in the Tetra Tech project-specific files. The Tetra Tech Project Chemist (or designee) is responsible for tracking the samples collected and shipped to the analytical laboratory. Upon receipt of the data packages from Empirical, the Tetra Tech Project Chemist will monitor the data validation effort, which includes verifying that the data packages are complete and results for all samples have been delivered by Empirical.

Data Storage, Archiving, and Retrieval

The data packages received from Empirical will be tracked in the data validation logbook. After the data are validated, the data packages will be entered into the Tetra Tech Navy 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 Tetra Tech FOL to be entered into the Navy CLEAN file system prior to archiving in secure project files. Project files will be audited for accuracy and completeness. At the completion of the Navy contract, the records will be stored by Tetra Tech. The secure project files will be stored by Tetra Tech [at Business Records Management (BRM)] as per [Worksheet #29](#).

Data Security

Access to Tetra Tech project files is 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, and access to the data files is restricted to qualified personnel only. File and data backup procedures are routinely performed.

Electronic Data

All electronic data is validated and qualifiers are manually added to the database. The manually entered qualifiers are verified by the DVM by direct QA, as well as an electronic verification step using proprietary Tetra Tech processes. Then data will be compiled into a NIRIS Electronic Data Deliverable (NEDD) and loaded into NIRIS in accordance with Navy procedures. This process includes a QA review of the data to ensure that the content and format of the data satisfy the requirements of NIRIS uploads. The NEDD is submitted through a data checker into NIRIS which also ensures the format is acceptable.

Data Review

This review will comprise data verification, validation, and usability assessment. The data verification and validation processes and requirements are described in [Worksheets #34, #35, #36, and #37](#). The data usability assessment will, at a minimum, constitute evaluation of the following characteristics to ensure that the amount, type, and quality of data are sufficient to achieve project objectives. The means of conducting these evaluations will vary depending on the nature of the data. For example, soil boring logs will generally be evaluated qualitatively or semiquantitatively whereas precision, accuracy, and sensitivity of analytical data will generally be evaluated quantitatively and may be based on, or may supplement, data validation findings. Examples include:

- Comparing actual to intended sampling locations, and verifying that the correct datum was used to delineate contamination.
- Evaluating trends across sample delivery groups or sampling events.
- Assessing quantitative relationships between parameters.
- Identifying potential errant or outlier data points.
- Assessing planning assumption validity.
- Evaluating the potential for contamination of samples by samplers.

Data quality indicators to be evaluated during this assessment include:

Precision

A semi quantitative estimate of the uncertainty in contaminant concentrations as a function of location will be made.

Accuracy

Accuracy data will be evaluated to ensure sampling and measurement accuracy is within or exceeds analytical method specifications, and may depend in part on the data validation findings.

Representativeness

This evaluation will assess whether the data are adequately representative of intended populations based on the sample collection and data generation requirements specified in this SAP.

Completeness

Completeness for this project will be determined based on the number of sample results for each target analyte and each sample type that are usable as determined through data validation and data assessment. Data values rejected during data validation (indicated by an "R" or "UR" flag) will be considered unusable unless additional review and documentation by one or more technical team members demonstrates that the rejection was erroneous. To monitor completeness, the number of usable, valid results for each soil type and analyte will be counted and compared to the completeness objective of 95%.

Percent completeness will be calculated using the following equation:

$$\% \text{ Completeness} = \frac{(\text{number of valid measurements})}{(\text{number of measurements planned})} \times 100\%$$

Comparability

This will be accomplished by comparing overall precision and bias among data sets for the matrix and analytical fraction for each sampled area. This will not require quantitative comparisons unless the Tetra Tech Project Chemist indicates that such quantitative analysis is beneficial to the project and the Tetra Tech TOM agrees.

Sensitivity

The Tetra Tech Project Chemist will determine whether project sensitivity goals were achieved by comparing non-detect values to PALs.

Other Quantitative Characteristics

These may include quantities such as verification of soil volume calculations, soil disposal cost estimates, etc., that are used to determine whether the contaminants are sufficiently well delineated to estimate remediation costs.

If significant data quality deficiencies are detected that prevent the attainment of project objectives, the limitations on the affected data will be described in the project report. The Tetra Tech TOM will bring these deficiencies to the attention of the Project Team for their evaluation, and the team will determine an appropriate corrective action depending on the circumstances.

Project Reports

Draft and final versions of the Project Report will be prepared. These reports will include the following sections:

- Executive Summary – will include a brief description of the work conducted and the findings.
- Introduction and Background – will include 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 – will include a summary of the work performed in accordance with the approved UFP-SAP and any field modifications as documented by the Tetra Tech FOL. This section will include maps showing the sampling locations and tables summarizing the data collected.
- Data Quality – will include a summary of quantitative analytical performance indicators such as completeness, precision, bias, and sensitivity; and qualitative indicators such as representativeness and comparability. This section will include a reconciliation of project data with the data quality objectives (DQOs) and an identification of deviations from this UFP-SAP.
- A data usability assessment will be used to identify significant deviations in analytical performance that could affect the ability to meet project objectives.
- Human Health Risk Screening Assessment – data will be compared to human health screening values.
- Project objective attainment.

SAP Worksheet #15 -- Reference Limits and Evaluation Table

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

Matrix: Soil

Analyte	Chemical Abstract Service (CAS) number	Project Action Limit (PAL) (mg/kg)	PAL Reference ⁽¹⁾	Project Quantitation Limit Goal (PQLG) (mg/kg)	Empirical Labs LOQ (mg/kg)	Empirical Labs LOD (mg/kg)	Empirical Labs DL (mg/kg)
Inorganics							
Aluminum	7229-90-5	7700	EPA RSL	2600	10	5	2.5
Antimony	7440-36-0	3.1	EPA RSL	1	0.50	0.4	0.25
Cadmium	7440-43-9	7	EPA RSL	2	0.25	0.1	0.05
Copper	7440-50-8	150	Residential SCTL	50	0.5	0.4	0.2
Chromium	7440-47-3	12000	EPA RSL	0.004	0.5	0.2	0.1
Iron	7439-89-6	5400	EPA Migration to GW	1800	5	3	1.5
Lead	7439-92-1	400	EPA RSL	130	0.25	0.15	0.075
Manganese	7439-96-5	180	EPA RSL	60	0.75	0.3	0.15
Molybdenum	7439-98-7	39	EPA RSL	1.2	1	0.5	0.25
Nickel	7440-02-0	130	Leachability SCTL	43	0.5	0.3	0.15
Vanadium	7440-62-2	39	EPA RSL	13	0.625	0.5	0.25
Zinc	7440-66-6	2300	EPA RSL	760	1	0.5	0.25
Explosives							
1,3,5-Trinitrobenzene	99-35-4	34	EPA RSL Migration	10	0.080	0.040	0.020
1,3-Dinitrobenzene	99-65-0	0.028	EPA RSL Migration	0.01	0.080	0.040	0.020
2,4,6-Trinitrotoluene	118-96-7	0.26	EPA RSL Migration	0.09	0.080	0.040	0.020
2,4-Dinitrotoluene	121-14-2	0.0056	EPA RSL Migration	0.002	0.080	0.040	0.020

Analyte	Chemical Abstract Service (CAS) number	Project Action Limit (PAL) (mg/kg)	PAL Reference ⁽¹⁾	Project Quantitation Limit Goal (PQLG) (mg/kg)	Empirical Labs LOQ (mg/kg)	Empirical Labs LOD (mg/kg)	Empirical Labs DL (mg/kg)
2,6-Dinitrotoluene	606-20-2	0.4	EPA RSL Migration	0.1	0.080	0.040	0.020
2-Amino-4,6-dinitrotoluene	35572-78-2	0.46	EPA RSL Migration	0.15	0.080	0.040	0.020
2-Nitrotoluene	88-72-2	0.005	SCTL Leach	0.002	0.080	0.040	0.020
4-Amino-2,6-dinitrotoluene	19406-51-0	0.46	EPA RSL Migration	0.15	0.080	0.040	0.020
4-Nitrotoluene	99-99-0	0.068	EPA RSL Migration	0.023	0.080	0.040	0.020
3-Nitrotoluene	99-08-1	0.024	EPA RSL Migration	0.008	0.080	0.040	0.020
HMX	2691-41-0	19.8	EPA RSL Migration	6.6	0.080	0.040	0.020
Nitrobenzene	98-95-3	0.00158	EPA RSL Migration	0.0005	0.080	0.040	0.020
RDX	121-82-4	0.0046	EPA RSL Migration	0.0015	0.080	0.040	0.020
Tetryl	479-45-8	11.8	EPA RSL Migration	4	0.080	0.040	0.020

- (1) The PAL references are: SCTL-Leach – FDEP Soil Cleanup Target Level, Leachability to Groundwater (FDEP, 2005); EPA-RSL – Regions 3, 6, and 9 Regional Screening Level, Direct Contact Residential, adjusted to 1/10 of value for noncarcinogens (USEPA, November 2011); EPA RSL Migration – Regions 3, 6, and 9 Soil Screening Level, Risk-Based Migration-to-Groundwater, Dilution Attenuation Factor (DAF) = 20 (USEPA, November 2011).

Notes: The most currently available RSLs will be used for the final report.

Bolded rows indicate that the PAL is between the laboratory LOQ and DL. The Partnering Team has agreed to accept this data for decision making as long as results below the LOQ are "J" qualified and discussed in the uncertainties section of the SI Report.

Shaded and Bold row indicate the PAL is less than the DL; therefore, the Partnering Team has agreed to replace the PALs with the laboratory LOQs for decision making purposes, as suggested in "Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits" (FDEP, October 2004).

SAP Worksheet #16 -- Project Schedule/Timeline Table

[\(UFP-QAPP Manual Section 2.8.2\)](#)

Activity	Organization	Anticipated Date of Completion
Prepare Draft SI Work Plan and Appendices	Tetra Tech	11/11
Submit Draft SI Work Plan and Appendices	Tetra Tech	11/11
Navy and Regulator Review	Navy, USEPA, and FDEP	03/12
Receive Comments/Comment Resolution	Tetra Tech	03/12
Prepare Final SI Work Plan and Appendices	Tetra Tech	03/12
Submit Final SI Work Plan and Appendices	Tetra Tech	03/12
Field Investigation	Tetra Tech	04/12
Laboratory Analysis	Empirical Laboratories	05/12
Data Validation	Tetra Tech	06/12
Database Entry	Tetra Tech	06/12
Prepare Rough Draft SI Report	Tetra Tech	08/12
Submit Rough Draft SI Report and Appendices	Tetra Tech	09/12
Navy Review	Navy	09/12
Receive Comments/Comment Resolution	Tetra Tech	09/12
Prepare Draft SI Report	Tetra Tech	10/12
Submit Draft SI Report	Tetra Tech	10/12
Navy and Regulator Review	Navy, USEPA, and FDEP	11/12

Activity	Organization	Anticipated Date of Completion
Receive Comments/Comment Resolution	Tetra Tech	12/12
Prepare Final SI Report	Tetra Tech	02/13
Submit Final SI Report	Tetra Tech	02/13

SAP Worksheet #17 – Sampling Design and Rationale

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

The sampling design for evaluating potential risks (Problem Statement #2 and Decision Rule #2) requires collection of multiple soil aliquots (increments) over a regular grid. These samples, each consisting of 60 separate increments, will be combined through milling and blending to obtain a representative average concentration of the area covered by the grid. This approach limits analytical costs yet provides assurance that the estimate of average concentration over the sampled area is a reasonable estimate of the mean concentration with relatively low variability compared to discrete sample collection. The sampling design also requires collection of two additional samples (duplicate and triplicate) in the same manner from the same gridded area to obtain a direct measurement of the variability in the estimate of mean concentration.

The proposed soil sampling program is focused on the area encompassing the former main target area at the Magazine Point Bombing Target site as shown on [Figure 3](#). The collection of the samples will provide information about any potential contamination in the surface soil at the site. All referenced field standard operating procedures (SOPs) are presented in [Appendix A](#). The soil samples from each individual grid cell will be collected from the 0 to 6-inch bgs interval in accordance with [SOP 05](#) presented in [Appendix A](#). As presented on [Figure 4](#), the Sampling Unit measures 375 feet by 120 feet and covers approximately 1 acre. Individual sample grid cells measure 25 feet by 30 feet. The information below provides details on the sampling effort for the incremental sample collection.

As a safety measure, prior to conducting the activities associated with the sampling event, a UXO Technician will perform a visual inspection of the sample areas to ensure the safety of the sampling team.

Sample Collection

The individual sample aliquots will be obtained by pushing the step probe sampler to a depth of 6 inches bgs. The aliquot samples will then be placed into a large sealable plastic bag labeled with the corresponding sample ID, date, and time. An attempt will be made in the field to remove all vegetation and rocks/sticks greater than 2 mm in size. All sample increments will be collected from 0 to 6 inches bgs. The complete incremental sample will consist of a total mass of approximately 1.5 to 2 kg of soil. The sample will be placed on ice and shipped to the laboratory in accordance with [SOP 03](#).

Prior to beginning sample collection, the Sampling Unit boundaries will be accurately determined by use of a hand-held GPS unit and marked by stakes, flagging, or some other means of clear visual reference in the field. The individual evenly-sized grid cells will then be located utilizing a measuring tape. The

proposed Sampling Unit will contain 60 cells with each individual cell measuring 30 feet by 25 feet (see [Figure 4](#)).

To begin actual sample collection, starting with the northwestern most cell, the sample team will begin collection of the original sample increments (denoted as “0” on [Figure 4](#)). The location of the sample increment will be consistent in each subsequent cell as the sample collection moves from west to east across the sample unit. When the sample crew reaches the eastern most cell in the row, the sample team will move to the sample cell directly to the south and then begin moving east to west while maintaining the consistent sample location within each cell. This movement of side to side increment sample collection will continue until the sample team has reached the last cell which will be the southeastern most cell. At this point, the collection of the original incremental sample (X1-IS-001A) will be complete. The sample bag will then be placed in a cooler on ice and the sample probe will undergo field decontamination as described in [SOP-06 \(Appendix A\)](#).

The next incremental sample to be collected will be the duplicate sample (denoted as “Δ” on [Figure 4](#)). Again, the sample team will begin in the northwestern most cell; however, the sample location within the cell will differ from the original sample (see [Figure 4](#)). Sample collection will move south down through the cells. When the sample team reaches the southernmost cell, the sample team will move to the sample cell directly to the east and then begin moving north up through the cells while maintaining the consistent sample location within each cell. This movement of up and down the sample unit will continue until the sample team has reached the last cell which will be the northeastern most cell. At this point, the collection of the duplicate incremental sample (X1-IS-002A) will be complete. The sample bag will be placed in a cooler on ice and the sample probe will undergo field decontamination as described in [SOP-06 \(Appendix A\)](#).

The final incremental sample collected will be the triplicate sample (denoted as “X” on [Figure 4](#)). The sample team will begin in the northeastern most cell with the sample location again different from the original and duplicate sample location (see [Figure 4](#)). Sample collection will move east to west across the grid. When the sample team reaches the western most cell in the row, the sample team will move to the sample cell directly to the south and then begin moving west to east while maintaining the consistent sample location within each cell. This movement of side to side will continue until the sample team has reached the last cell which will be the southwestern most cell. At this point, the collection of the triplicate incremental sample (X1-IS-003A) will be complete. The sample bag will be placed in a cooler on ice and the sample probe will undergo field decontamination as described in [SOP-06 \(Appendix A\)](#).

Placement of the grid is not critical as the deposition of contamination on the scale of sampling is a reasonably random process such that any regular grid of sampling locations effectively represents a random sample.

Sample Analysis

To allow for determining whether unmilled metals discrete samples are adequately representative of unmilled sample background metal concentrations, the three incremental samples that are collected will also be sampled in a way that removes a minor amount of mass from each sample to be milled, yet is representative of the incremental sample mass as a whole.

Approximately 1.5 to 2 kg of soil (possibly containing vegetation such as roots, small twigs, and grass plus small stones) will be delivered to the laboratory for each of three samples. The laboratory shall follow SW-846 Method 8330B for air drying samples and sieving to remove vegetation and particles greater than 2 mm in size. Each of the three samples will be processed further as described below.

After sieving, but prior to further processing, the laboratory technician will remove at least thirty, 1-gram subaliquots of sample from the total sample mass that has been air-dried and sieved. The subaliquots shall be obtained by spreading out the dried and sieved sample and removing approximately 1 gram of mass from each of 30 different randomly selected locations. These subaliquots will be mixed thoroughly without grinding, and then two subaliquots of sample equal to approximately 5 grams each shall be removed from this submass.

The remaining dried and sieved sample mass (nominally 1.5 to 2 kg mass) shall then be processed further by milling in a ring mill and blending the milled portions to yield a nominal 1.5 to 2 kg of milled and well-mixed sample mass. Upon completion of all milling and subsampling, the laboratory will have the following from each of the samples submitted for analysis:

- Two 5-gram submasses of soil that have not been milled but are representative of the entire 1.5 to 2 kg incremental sample mass.
- One nominal 1.5 to 2 kg mass of milled and blended soil.

The laboratory shall digest and analyze a 5-gram aliquot of milled and a 5-gram aliquot of unmilled sample for each of the three samples submitted for metals analysis. Metal target analytes are listed in [Worksheet #15](#). The extra unmilled 5-gram mass from each sample is a backup sample to be used in the event that the analytical sample preparation process fails for the original 5-gram unmilled sample mass.

The laboratory shall also extract and analyze a 10-gram aliquot of milled sample for each of the three samples submitted for explosives analysis. Explosives target analytes are listed on [Worksheet #15](#).

In summary, three incremental samples will be collected from a 1-acre sampling grid (original, duplicate, and triplicate). Each incremental sample will represent 60 separate soil increments. Analysis of each site incremental sample will yield one milled and one unmilled concentration measurement for each metal target analyte. Unmilled samples will not be analyzed for target analyte explosives.

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

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

Sample Location	Sample ID ⁽¹⁾	Analyses ⁽²⁾			Sampling SOP Reference
		Explosives	Metals (milled)	Metals (un-milled)	
X1-IS-001 (original)	X1-IS-001A-0006	1	1	---	SOP-05
	X1-IS-001B-0006	---	---	1	
X1-IS-002 (duplicate)	X1-IS-002A-0006	1	1	---	
	X1-IS-002B-0006	---	---	1	
X1-IS-003 (triplicate)	X1-IS-003A-0006	1	1	---	
	X1-IS-003B-0006	---	---	1	

- (1) "A" indicates sample will be milled during the analytical process. "B" indicates sample will not be milled during the analytical process.
- (2) The analytical laboratory will receive the three "A" samples from the field and will then be responsible for collecting the "B" subsamples for the non-milled metals analysis.

SAP Worksheet #19 -- Analytical SOP Requirements Table - Empirical Laboratories

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

Matrix	Analytical Group	Analytical and Preparation Method/SOP Reference	Containers (number, size, and type)	Sample Volume (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)
Incremental Soil Samples	Explosives	SW-846 8330B/ Empirical SOP-327	Plastic bag or other container	1 to 2 kg	Cool to ≤ 6 °C	14 days until extraction, 40 days to analysis
	Metals	SW-846 3050B/6010C Empirical SOP-100/105	Plastic bag or other container	1 to 2 kg	None	180 days to analysis

SAP Worksheet #20 -- Field Quality Control Sample Summary Table – Analytical Samples

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

Matrix	Analytical Group	No. of Samples⁽¹⁾	No. of MS/MSDs⁽²⁾	No. of Duplicate Samples	Total No. of Samples to Lab⁽²⁾
Soil	Explosives	3	---	---	3
	Metals	3	---	---	3

- 1 Three samples will be collected in the field. Each sample will be submitted to the FBL for both explosives and metals analysis.
- 2 The number of samples includes the original, duplicate, and triplicate samples collected in the field. No additional MS/MSD or duplicates will be collected.

SAP Worksheet #21 -- Project Sampling SOP References Table

(UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date, and/or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
SOP-01	Sample Labeling, 09/11, Revision 0	Tetra Tech	Not Applicable (NA)	N	Contained in Appendix A
SOP-02	Sample Identification Nomenclature, 09/11, Revision 0	Tetra Tech	NA	Y ⁽¹⁾ (project-specific SOP)	Contained in Appendix A
SOP-03	Sample Custody and Documentation, 09/11, Revision 0	Tetra Tech	Field log book, sample log sheets	N	Contained in Appendix A
SOP-04	Sample Preservation, Packaging, and Shipping 09/11, Revision 0	Tetra Tech	NA	N	Contained in Appendix A
SOP-05	Incremental Composite Sampling, 09/11, Revision 0	Tetra Tech	CRREL or equivalent sample tool	N	Contained in Appendix A
SOP-06	Decontamination, 09/11, Revision 0	Tetra Tech	NA	N	Contained in Appendix A
SOP-07	Global Positioning System, 09/11, Revision 0	Tetra Tech	GPS unit (sub-meter)	N	Contained in Appendix A
SOP-08	Investigation-Derived Waste, 09/11, Revision 0	Tetra Tech	NA	Y ⁽²⁾ (project-specific SOP)	Contained in Appendix A

- 1 Modified to include specific sample nomenclature requirements for Magazine Point Bombing Target incremental samples.
- 2 Modified to delete portion of SOP that does not apply to this project (i.e., soil disposal).

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 (CA)	Responsible Person	SOP Reference	Comments
GPS	Positioning	Beginning and end of each day used	Accuracy: sub-meter horizontal dilution of precision (HDOP)<3, number of satellites at least six	Wait for better signal, replace unit, or choose alternate location technique	FOL	SOP-07	None

SOPs are located in [Appendix A](#).

SAP Worksheet #23 -- Analytical SOP References Table - Empirical Laboratories

(UFP-QAPP Manual Section 3.2.1)

Lab SOP Number	Title, revision date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Variance to QSM? (Y or N)	Modified for Project Work? (Y/N)
Empirical SOP-100	Metals Digestion/ Preparation, Methods 3005A/ USEPA CLP ILMO 4.1 Aqueous, 3010A, 3030C, 3050B, USEPA CLP ILMO 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C (Revision 21, 09/01/10)	Definitive	Soil/ Metals Digestion	NA/Preparation	Empirical	N	N
Empirical SOP-105	Metals by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) Technique, SW-846 Methods 6010B, 6010C, USEPA Method 200.7, Standard Methods 19 th Edition 2340B, USEPA CLP ILMO 4.1 (Revision 16, 04/11/10)	Definitive	Soil/ Metals	ICP-AES	Empirical	Y (samples will be concentrated 2g/100 mL)	N
Empirical SOP-327	Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC) Method 8330, 8330A, 8330B, and 8332 (Revision 18, 09/07/10)	Definitive	Soil/ Explosives	HPLC	Empirical	N	N

SAP Worksheet #24 -- Analytical Instrument Calibration Table - Empirical Laboratories

[\(UFP-QAPP Manual Section 3.2.2\)](#)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference ⁽¹⁾
HPLC – Explosives (including NG)	Initial Calibration (ICAL) - minimum 5 points	Annually or more often as needed due to changes in response or retention times or following major instrument maintenance.	Average response factor (RF) ≤ 20 percent relative standard deviation (%RSD) ; if a linear fit is used, correlation coefficient (r) ≥ 0.995 ; or $r^2 \geq 0.99$ using 6 points.	Determine and correct reason for failure. Repeat calibration.	Analyst/ Supervisor	Empirical SOP-327
	Second-source Initial calibration verification (ICV)	Following initial calibration prior to the analysis of samples.	80-120 percent recovery (%R) of the true value.	Investigate reasons for failure, reanalyze once. If still unacceptable, repeat calibration.	Analyst/ Supervisor	
	Continuing calibration verification (CCV)	Daily prior to the analysis of samples, every 10 sample injections or 12 hours (whichever is more frequent), and at the end of the run.	Less than 20 percent difference (%D) for each target analyte.	If %D is high and sample result is ND (non-detect), qualify/narrate with project approval. If %D is low or project approval not received, reanalyze all samples since the last successful CCV.	Analyst/ Supervisor	
	Continuing calibration blank (CCB)	After the initial CCV, after every 10 samples, and at the end of the sequence.	No analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze calibration blank and previous 10 samples.	Analyst/ Supervisor	
ICP-AES Metals	ICAL - a 1-point calibration per manufacturer's guidelines is prepared for all target analytes	At the beginning of each day, or if the QC is out of criteria, prior to sample analysis.	None; only one high standard and a calibration blank must be analyzed. If more than one calibration standard is used, r must be ≥ 0.995 .	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	Empirical SOP-103, 104, 105
	ICV – Second Source	Following ICAL, prior to the analysis of samples.	The %R of all target analytes must be within 90-110% of true value.	Investigate reasons for failure, reanalyze once. If still unacceptable, correct problem and repeat ICAL.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference ⁽¹⁾
	CCV	At the beginning and end of the sequence and after every 10 samples.	The %R must be within 90-110% of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze all affected samples.	Analyst/ Supervisor	
	Initial Calibration Blank (ICB)	Before beginning a sample sequence.	No target analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze.	Analyst / Supervisor	
	CCB	After the initial CCV, after every 10 samples, and at the end of the sequence.	No target analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze calibration blank and all affected samples.	Analyst / Supervisor	
	Low-Level Check Standard (if using 1-point ICAL) – not for mercury	Daily after 1-point ICAL and before samples.	The %R must be within 80-120% of true value.	Investigate and perform necessary equipment maintenance. Recalibrate and reanalyze all affected samples.	Analyst / Supervisor	
	Interference Check Standards (ICS – ICS A and ICS B)	At the beginning of an analytical run.	ICS A recoveries must be within the absolute value of the LOD; and ICS B recoveries must be within 80-120 %R of true value.	Terminate analysis; locate and correct problem; reanalyze ICS.	Analyst / Supervisor	

1 Laboratory SOPs are subject to revision and updates during duration of the project; the laboratory will use the most current revision of the SOP at the time of analysis.

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

[\(UFP-QAPP Manual Section 3.2.3\)](#)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person	SOP Reference
ICP-AES	Clean the torch assembly and the spray chamber when they become discolored or when degradation in data quality is observed. Clean the nebulizer, and check the argon supply. Replace the peristaltic pump tubing as needed.	Metals	Inspect the torch, nebulizer chamber, pump, and tubing	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	Empirical SOP-105
HPLC	Check pressure and gas supply daily – change when <200 pounds per square inch (psi), change analytical column as needed, change mobile phase when insufficient for run or contamination, change inlet filters as needed for contamination.	Explosives	Check pump pressure, check for leaks, check for adequate mobile phase.	Prior to initial calibration or as necessary.	CCV < 20% difference.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst/ Supervisor	Empirical SOP-327

SAP Worksheet #26 -- Sample Handling System

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

SAMPLE HANDLING SYSTEM

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): FOL/Tetra Tech
Sample Packaging (Personnel/Organization): FOL/Tetra Tech
Coordination of Shipment (Personnel/Organization): FOL/Tetra Tech
Type of Shipment/Carrier: Express Mail – overnight courier
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Custodians/Empirical Laboratories
Sample Custody and Storage (Personnel/Organization): Sample Custodians/Empirical Laboratories
Sample Preparation (Personnel/Organization): Extraction Lab, Metals Preparation Lab/Empirical Laboratories
Sample Determinative Analysis (Personnel/Organization): Gas Chromatography/Mass Spectrometry Lab, Metals Lab/ Empirical Laboratories
SAMPLE ARCHIVING
Field Sample Storage: 60 days from receipt of collection.
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 60 days
Biological Sample Storage (No. of days from sample collection): NA
SAMPLE DISPOSAL
Personnel/Organization: Sample Custodians/ Empirical Laboratories

SAP Worksheet #27 – Sample Custody Requirements Table

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

SAMPLE CUSTODY REQUIREMENTS

Field Chain of Custody

To ensure the integrity of a sample from collection through analysis, an accurate, written record that traces the possession and handling of the sample is necessary. This documentation is referred to as the chain-of-custody (COC) form. COC 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 collection, preparation, handling, storage, and shipping of all samples. 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 a 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 SI 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). Field COC requirements are provided in [SOP-03](#). Upon arrival at the laboratory, internal sample custody procedures will be followed as defined in the laboratory SOPs included in [Appendix D](#).

Laboratory Chain of Custody - Empirical

Laboratory sample custody procedures (receipt of samples, archiving, and disposal) will be used according to Empirical [SOPs](#). Coolers will be received and checked for proper temperature. A sample cooler receipt form will be filled out to note conditions and any discrepancies. The chain-of-custody will

be checked against the sample containers for correctness. Samples will be logged into the laboratory information management system (LIMS) and given a unique log number which can be tracked through processing. The client will be notified of any problems.

SAP Worksheet #28 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Soil					
Analytical Group	Metals					
Analytical Method / SOP Reference	SW-946 6010C/ Empirical SOP-105					
QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One per preparatory batch of 20 or fewer samples of similar matrix.	All target analytes must be $\leq \frac{1}{2}$ LOQ.	Re-analysis to confirm the positive value. Re-prepare and reanalyze samples associated with the blank.	Analyst, Supervisor	Bias/ Contamination	Same as QC Acceptance Limits
LCS	One per preparatory batch of 20 or fewer samples of similar matrix.	%R must be within 80-120% of true value.	Correct problem, then reprepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Contact client if samples cannot be reanalyzed within hold time.	Analyst, Supervisor	Accuracy/ Bias	Same as QC Acceptance Limits
MS	One per preparatory batch of 20 or fewer samples of similar matrix.	%R should be within 80-120% of true value (if sample is < 4x spike added).	A post digest spike (PDS) should be analyzed whenever the MS is outside criteria	Analyst, Supervisor	Accuracy/ Bias	Same as QC Acceptance Limits
Sample Duplicate	One per preparatory batch of 20 or fewer samples of similar matrix.	The RPD should be $\leq 20\%$ for duplicate samples for both water and soils.	Narrate any results that are outside control limits.	Analyst, Supervisor	Precision	Same as QC Acceptance Limits
Serial Dilution (Inductively Coupled Plasma (ICP) Only)	One per preparatory batch with sample concentration(s) >50x LOD.	The 5-fold dilution result must agree within $\pm 10\%D$ of the original sample result if result is >50x LOD.	Perform post digestion spike. Data will be qualified when the %D is outside of criteria.	Analyst, Supervisor	Accuracy/ Bias	Same as QC Acceptance Limits

Matrix	Soil					
Analytical Group	Metals					
Analytical Method / SOP Reference	SW-946 6010C/ Empirical SOP-105					
QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
PDS (ICP Only)	One is performed when MS sample is outside criteria or serial dilution fails or target analyte concentration(s) in all samples are < 50x LOD.	The %R must be within 80-120% of expected value to verify the absence of an interference. Spike addition should produce a concentration of 10-100x LOQ.	Flag results for affected analytes for all associated samples with "J".	Analyst, Supervisor	Accuracy/ Bias	Same as QC Acceptance Limits
Results between DL and LOQ	NA.	Apply "J" qualifier to results between DL and LOQ.	NA.	Analyst, Supervisor	Accuracy	Same as QC Acceptance Limits

Matrix	Soil					
Analytical Group	Explosives					
Analytical Method / SOP Reference	SW-846 8330B Empirical SOP-327					
QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One per preparatory batch of 20 or fewer samples.	All target analytes must be $\leq \frac{1}{2}$ LOQ.	If the method blank acceptance criteria are not met, identify and correct the source of contamination, and re-prepare and reanalyze the associated samples.	Analyst, Supervisor	Bias/ Contamination	Same as QC Acceptance Limits
LCS	One per preparatory batch of 20 or fewer samples of similar matrix.	%Rs for aqueous and soil are provided following this table.	Correct problem, then reprepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Contact Client if samples cannot be reanalyzed within hold time.	Analyst, Supervisor	Accuracy/ Bias	Same as QC Acceptance Limits
MS/MSD	One per preparatory batch of 20 or fewer samples of similar matrix.	%Rs for aqueous and soil are provided following this table.	CA will not be taken for samples when %Rs are outside limits and surrogate and LCS criteria are met unless RPDs indicate obvious extraction/ analysis difficulties, then re-prepare and reanalyze MS/MSD.	Analyst, Supervisor	Accuracy/ Bias/ Precision	Same as QC Acceptance Limits
Surrogate	All field and QC samples - one per sample. One surrogate 1-chloro-3-nitrobenzene.	%Rs = 60%-140% for aqueous %Rs = 50-150% for soil	If surrogate %Rs are outside the established limits, verify calculations, dilutions, and standard solutions. Also verify that the instrument performance is acceptable. If the surrogate %R is outside the established limits due to well-documented matrix effects, the results must be flagged and an explanation included in the report narrative.	Analyst, Supervisor	Accuracy	Same as QC Acceptance Limits
Results between DL and LOQ	NA.	Apply "J" qualifier to results detected between DL and LOQ.	NA.	Analyst, Supervisor	Accuracy	Same as QC Acceptance Limits
Second Column Confirmation	All positive results must be confirmed.	Results between primary and secondary column must be RPD $\leq 40\%$.	None. Apply "J" flag if RPD $>40\%$ and discuss in the case narrative.	Analyst, Supervisor	Accuracy	Same as QC Acceptance Limits

Matrix	Method	Analyte	LCS MS/MSD Lower Limit %R	LCS MS/MSD Upper Limit %R	RPD
Solid	8330B	1,3,5-Trinitrobenzene	75	125	20
Solid	8330B	1,3-Dinitrobenzene	80	125	20
Solid	8330B	1-Chloro-3-nitrobenzene (Surrogate)	55	140	
Solid	8330B	2,4,6-Trinitrophenylmethylnitramine (Tetryl)	10	150	20
Solid	8330B	2,4,6-Trinitrotoluene (TNT)	55	140	20
Solid	8330B	2,4-Dinitrotoluene (DNT)	80	125	20
Solid	8330B	2,6-Dinitrotoluene	80	125	20
Solid	8330B	2-Amino-4,6-dinitrotoluene	80	125	20
Solid	8330B	2-Nitrotoluene (ONT)	80	125	20
Solid	8330B	3,5-Dinitroaniline	60	120	20
Solid	8330B	3-Nitrotoluene	75	120	20
Solid	8330B	4-Amino-2,6-dinitrotoluene	80	125	20
Solid	8330B	4-Nitrotoluene (PNT)	75	125	20
Solid	8330B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	70	135	20
Solid	8330B	Nitrobenzene	75	125	20
Solid	8330B	Nitroglycerin	60	120	20
Solid	8330B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	75	125	20
Solid	8330B	PETN	60	120	20
Water	8330B	1,3,5-Trinitrobenzene	65	140	20
Water	8330B	1,3-Dinitrobenzene	45	160	20
Water	8330B	1-Chloro-3-nitrobenzene (Surrogate)	40	145	
Water	8330B	2,4,6-Trinitrophenylmethylnitramine (Tetryl)	20	175	20
Water	8330B	2,4,6-Trinitrotoluene (TNT)	50	145	20
Water	8330B	2,4-Dinitrotoluene (DNT)	60	135	20

Matrix	Method	Analyte	LCS MS/MSD Lower Limit %R	LCS MS/MSD Upper Limit %R	RPD
Water	8330B	2,6-Dinitrotoluene	60	135	20
Water	8330B	2-Amino-4,6-dinitrotoluene	50	155	20
Water	8330B	2-Nitrotoluene (ONT)	45	135	20
Water	8330B	3,5-Dinitroaniline	60	120	20
Water	8330B	3-Nitrotoluene	50	130	20
Water	8330B	4-Amino-2,6-dinitrotoluene	55	155	20
Water	8330B	4-Nitrotoluene (PNT)	50	130	20
Water	8330B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	50	160	20
Water	8330B	Nitrobenzene	50	140	20
Water	8330B	Nitroglycerin	60	120	20
Water	8330B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	80	115	20
Water	8330B	PETN	60	120	20

SAP Worksheet #29 -- Project Documents and Records Table

[\(UFP-QAPP Manual Section 3.5.1\)](#)

Document	Where Maintained
<p><u>Sample Collection Documents and Records</u> Project Personnel Sign-off Records Field logbook (and sampling notes) Field sample forms (e.g., boring logs, sample log sheets, drilling logs, etc.) COC records Sample shipment airbills Equipment calibration logs Photographs FTMR forms SAP Field sampling SOPs</p>	<p>Tetra Tech project file (may include hard-copy as well as electronic information), results will be discussed in subject document.</p>
<p><u>Laboratory Documents and Records</u> Sample receipt/log-in forms Sample storage records Sample preparation logs Standard traceability logs Equipment calibration logs Sample analysis run logs Equipment maintenance, testing, and inspection logs FTMR forms Reported field sample results Reported results for standards, QC checks, and QC samples Data completeness checklists Sample storage and disposal records Telephone logs Extraction/clean-up records Raw data</p> <p><u>Data Assessment Documents and Records</u> Field sampling audit checklist (if an audit is conducted) Analytical audit checklist (if an audit is conducted) Data validation memoranda</p>	<p>Tetra Tech project file (may include hard-copy as well as electronic information), long-term data package storage at third-party professional document storage firm (BRM), results will be discussed in subject document.</p> <p>Tetra Tech project file (may include hard-copy as well as electronic information), results will be discussed in subject document.</p>

Document	Where Maintained
<u>Other Documents</u> HASP All final versions of SAP All final versions of reports, amendments and revisions (e.g., SI, RI, FS, etc.)	Tetra Tech project file (may include hard-copy as well as electronic information)

Tetra Tech storage of archived project documents and records will be secured at a third-party professional document storage firm in the BRM repository located at 651 Mansfield Ave, Pittsburgh, PA 15220.

SAP Worksheet #30 -- Analytical Services Table

[\(UFP-QAPP Manual Section 3.5.2.3\)](#)

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)
Soil	Metals	See Worksheet #18	SW-846 6010C	21 calendar days	Empirical Laboratories 621 Mainstream Drive Suite 270 Nashville, TN 37228 Brian Richard (615)-345-1115 X249	NA
	Explosives		SW-846 8330B			

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 CA (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Laboratory System Audit ¹	Every 2 years	External	DoD ELAP Accrediting Body	DoD ELAP Accrediting Body Auditor	Laboratory QAM or Laboratory Manager, Empirical	Laboratory QAM or Laboratory Manager, Empirical	Laboratory QAM or Laboratory Manager, Empirical

1 Empirical is DoD ELAP and Florida accredited by a recognized accrediting body. Copies of DoD ELAP accreditation letters are included in [Appendix D](#).

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

[\(UFP-QAPP Manual Section 4.1.2\)](#)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Laboratory System Audit	Written audit report	Marcia McGinnity, Laboratory Data Quality Manager (DQM), Empirical	Specified by DoD ELAP Accrediting Body	Letter	DoD ELAP Accrediting Body	Specified by DoD ELAP Accrediting Body

SAP Worksheet #33 -- QA Management Reports Table

[\(UFP QAPP Manual Section 4.2\)](#)

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Data Validation Report	Per sample delivery group (SDG)	Within 3 weeks after receiving the data from the laboratory	Project Chemist or Data Validator, Tetra Tech	TOM, Tetra Tech; project file
Project Monthly Progress Report	Monthly for duration of the project	Monthly	TOM, Tetra Tech	TOM, Tetra Tech; QAM, Tetra Tech; Program Manager, Tetra Tech; Navy RPM; project file
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately upon detection of problem (on the same day)	Laboratory PM, Empirical and Test America	TOM and project file, Tetra Tech

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

[\(UFP-QAPP Manual Section 5.2.1\)](#)

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-Custody Forms	The Tetra Tech FOL or designee will review and sign the chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory and the sample information is accurate. The forms will be signed by the sampler and a copy will be retained for the project file, the Tetra Tech TOM, and the Tetra Tech Data Validators. See SOP-01 .	Internal	Sampler and FOL, Tetra Tech
	The Laboratory Sample Custodian will review the sample shipment for completeness and integrity thereby accepting the shipment. The Tetra Tech Data Validators will check that the chain-of-custody form was signed and dated by the Tetra Tech FOL or designee relinquishing the samples and also by the Laboratory Sample Custodian receiving the samples for analyses.	Internal/ External	1 - Laboratory Sample Custodian, Empirical 2 - Data Validators, Tetra Tech
SAP Sample Tables/ Chain-of-Custody Forms	Verify that all proposed samples listed in the SAP tables have been collected.	Internal	FOL or designee, Tetra Tech
Sample Log Sheets	Verify that information recorded in the log sheets is accurate and complete.	Internal	FOL or designee, Tetra Tech
SAP/Field Logs/ Analytical Data Packages	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and measurement performance criterion MPC have been achieved. Particular attention should be given to verify that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken trail of documented chain-of-custody from sample collection to report generation. Verify that the correct sampling and analytical methods/SOPs were applied. Verify that the sampling plan was implemented and carried out as written and that any deviations are documented.	Internal	TOM or designee, Tetra Tech
SAP/Laboratory SOPs/ Raw Data/ Applicable Control Limits Tables	Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied. 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 QAM will contact the Tetra Tech TOM via telephone or e-mail for guidance prior to report preparation.	Internal	Laboratory QAM, Empirical
SAP/Chain-of-Custody Forms	Check that field QC samples listed in Worksheet #20 were collected as required.	Internal	FOL or designee, Tetra Tech

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Electronic Data Deliverables (EDDs)/ Analytical Data Packages	Each EDD will be verified against the chain-of-custody form and hard copy data package for accuracy and completeness. Laboratory analytical results will be verified and compared to the electronic analytical results for accuracy. Sample results will be evaluated for laboratory contamination and will be qualified for false positives using the laboratory method/preparation blank summaries. Positive results reported between the MDL and the LOQ will be qualified as estimated. Extraneous laboratory qualifiers will be removed from the validation qualifier.	External	Data Validators, Tetra Tech
Analytical Data Packages	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The Laboratory QAM will sign the case narrative for each data package.	Internal	Laboratory QAM, Empirical
	Each data package will be verified for completeness by the Tetra Tech Data Validator. Missing information will be requested by the Tetra Tech Data Validator from the Laboratory PM.	External	Data Validators, Tetra Tech

Notes: Verification includes field data verification and laboratory data verification. Verification inputs as per [Worksheet #34](#) will be checked.

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

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

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	SAP/Sample Log Sheets	Verify that actual sample locations are correct and in accordance with the SAP proposed locations. Document any discrepancies in the final report.	TOM, FOL, or designee, Tetra Tech
IIa	Chain-of-Custody Forms	Ensure that the custody and integrity of the samples was maintained from collection to analysis and the custody records are complete and any deviations are recorded. Review that the samples were shipped and stored at the required temperature and sample pH for chemically-preserved samples meet the requirements listed in Worksheet #19 . Ensure that the analyses were performed within the holding times listed in Worksheet #19 .	Project Chemist or Data Validators, Tetra Tech
IIa/IIb	SAP/Laboratory Data Packages/ EDDs	Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the MPCs listed in Worksheet #12 were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	Project Chemist or Data Validators, Tetra Tech
		Check the field sampling precision by calculating the RPD for field duplicate samples. Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses; MS/MSDs; and LCS/LCSD, if available.	
		Check that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from sample collection to analysis.	
		Review the chain-of-custody forms generated in the field to ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. The Tetra Tech Data Validator will verify that elements of the data package required for validation is present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet #36 .	

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIb	SAP/ Laboratory Data Packages/ EDDs	Ensure that the LOQs listed in Worksheet #15 were achieved.	Project Chemist or Data Validators, Tetra Tech
		Discuss the impact of matrix interferences or sample dilutions performed because of the high concentration of one or more other contaminants, on the other target compounds reported as non-detected.	
		Summarize deviations from methods, procedures, or contracts in the Data Validation Report. If possible determine the impact of any deviation from sampling or analytical methods and SOPs requirements and matrix interferences effect on the analytical results. Qualify data results based on method or QC deviation and explain all the data qualifications.	

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 samples	Explosives	100% limited validation ¹ will be performed using criteria for SW-846 Methods 8330B listed in this SAP and the current DoD QSM. If not included in the aforementioned, the logic outlined in USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review USEPA-540/R-99-008, (USEPA, October 1999) will be used to apply qualifiers to data to the extent possible.	Data Validators ² , Tetra Tech
IIa and IIb	Soil samples	Metals	100% limited validation ¹ will be performed using criteria for SW-846 Method 6010C listed in this SAP and the current DoD QSM. If not included in the aforementioned, the logic outlined in USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (USEPA, October 2004) will be used to apply qualifiers to data to the extent possible.	Data Validators ² , Tetra Tech

- 1) Limits the data review to specific review parameters (Data Completeness/Data Verification, Holding times, Calibrations, Blank Contamination, & Detection limits) to determine gross deficiencies only. The limited data validation is best expressed as a review to preclude the possibility of false negatives and to eliminate false positives. Raw data are not evaluated and sample result verification is not conducted. A formal report, similar to a full data validation report, is prepared.
- 2) Tetra Tech Chemists performing data validation are independent of data generation activities.

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 Tetra Tech TOM and risk assessor will determine whether the deviations compromise the ability to meet project objectives. If they do, the Tetra Tech TOM will consult with the Navy RPM and other Project Team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions.

Precision

- The Project Chemist acting on behalf of the Project Team will determine whether precision goals for field duplicates and laboratory duplicates were met. This will be accomplished by comparing duplicate results to precision goals identified in [Worksheets #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, laboratory Control sample Duplicate (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.

Data Usability Assessment

Representativeness

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

Comparability

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

Sensitivity

- The Project Chemist acting on behalf of the Project Team will determine whether project sensitivity goals listed in [Worksheet #15](#) are achieved. The overall sensitivity and QLs 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 Tetra Tech TOM and designated team members will evaluate whether project assumptions are 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. Statistical tests for outliers will be conducted using standard statistical techniques appropriate for this task. Potential outliers will be removed if a review of the associated indicates that the results have an assignable cause that renders them inconsistent with the rest of the data. During this evaluation, the team will consider whether outliers could be indications of unanticipated site conditions. Consideration will be given to whether outliers represent an unanticipated site condition.

Data Usability Assessment

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

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

For statistical comparisons and mathematical manipulations, non-detected values will be represented by a concentration equal to one-half the sample-specific reporting limit. For duplicate results (original and duplicate), the highest concentration of the original and its duplicate sample will be used to represent the concentration at a particular sampled location.

Identify the personnel responsible for performing the usability assessment:

The Tetra Tech TOM, 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, NAS Pensacola POC, FDEP, and USEPA. If deficiencies affecting the attainment of project objectives are identified, 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.

REFERENCES

Tetra Tech (Tetra Tech NUS, Inc.), 2010. Site Inspection Report for Munitions Response Program Site Inspections at 13 Sites Outlying Landing Fields Bronson, Corry Station, and Saufley Field, Naval Air Station Pensacola, Pensacola, Florida. September.

FDEP (Florida Department of Environmental Protection), 2004. Guidance for the Selection of Analytical Methods for the Evaluation of Practical Quantitation Limits. October.

FDEP, 2005. Final Technical Report: Development of Cleanup Target Levels (CTLs) for Chapter 62-777, F.A.C. Prepared for the Division of Waste Management, Florida Department of Environmental Protection; Prepared by Center for Environmental & Human Toxicology, University of Florida, Gainesville, FL. February 26.

United States Environmental Protection Agency (USEPA), 2011. Regions 3, 6, and 9 Regional Screening Levels for Chemical Contaminants at Superfund Sites. November 2011 RSL Table Update: <http://epa-prgs.ornl.gov/chemicals/index.shtml>



DRAWN BY	DATE
S. STROZ	9/25/09
CHECKED BY	DATE
Y. MARTINEZ	1/25/11
REVISED BY	DATE
MK BOND	9/14/11
SCALE AS NOTED	



AREA LOCATION MAP
 NAS PENSACOLA
 PENSACOLA, FLORIDA

CONTRACT NUMBER 067	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO. 1	REV 0

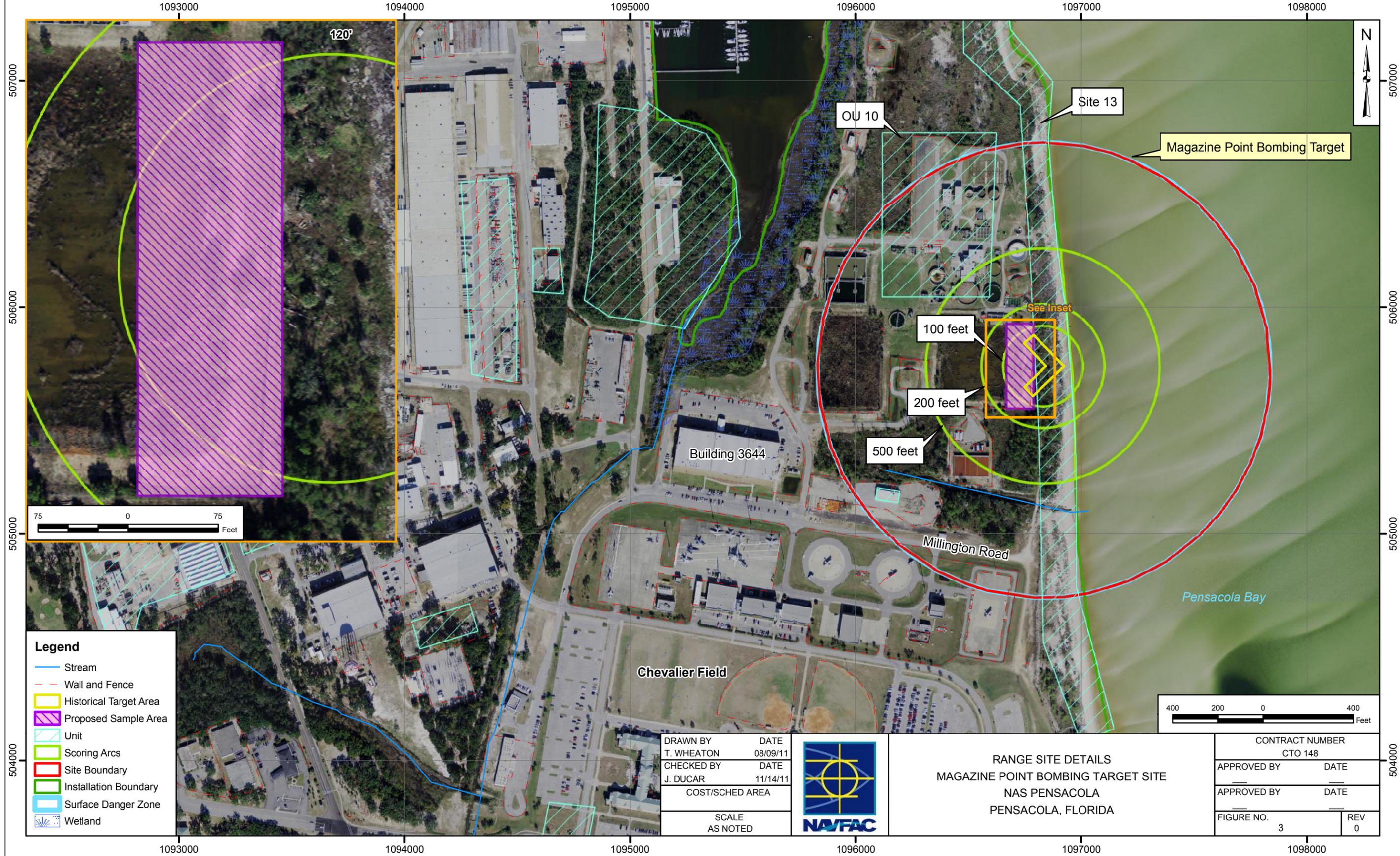


DRAWN BY S. STROZ	DATE 9/25/09
CHECKED BY J. GOERDT	DATE 9/25/09
REVISED BY MK BOND	DATE 9/14/11
SCALE AS NOTED	



SITE LOCATION MAP
NAS PENSACOLA
PENSACOLA, FLORIDA

CONTRACT NUMBER	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO. 2	REV 0



- Legend**
- Stream
 - - - Wall and Fence
 - Historical Target Area
 - Proposed Sample Area
 - Unit
 - Scoring Arcs
 - Site Boundary
 - Installation Boundary
 - Surface Danger Zone
 - ~ Wetland

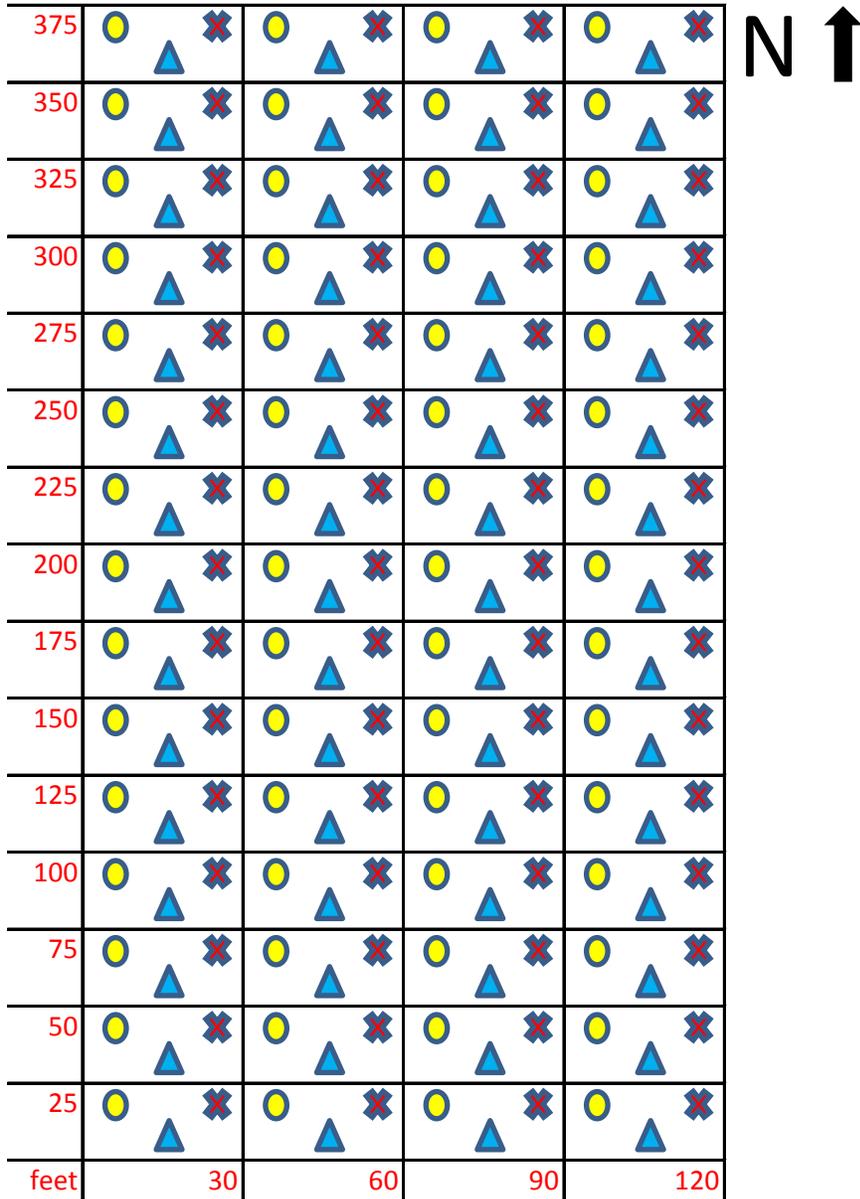
DRAWN BY T. WHEATON	DATE 08/09/11
CHECKED BY J. DUCAR	DATE 11/14/11
COST/SCHED AREA	
SCALE AS NOTED	



RANGE SITE DETAILS
MAGAZINE POINT BOMBING TARGET SITE
NAS PENSACOLA
PENSACOLA, FLORIDA

CONTRACT NUMBER CTO 148	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO. 3	REV 0

Figure 4
Incremental Composite Sample Layout
Magazine Point Bombing Target Site
NAS Pensacola



- = Original Sample (X1-IS-001A0006)
- = Duplicate Sample (X1-IS-002A0006)
- = Triplicate Sample (X1-IS-003A0006)

APPENDIX A

SITE-SPECIFIC FIELD STANDARD OPERATING PROCEDURES

TABLE OF CONTENTS

SOP-01	Sample Labeling
SOP-02	Sample Identification Nomenclature
SOP-03	Sample Custody and Documentation of Field Activities
SOP-04	Sample Preservation, Packaging, and Shipping
SOP-05	Incremental Composite Sampling for Soil and Sediment
SOP-06	Decontamination of Field Sampling Equipment
SOP-07	Global Positioning System
SOP-08	Management of Investigation-Derived Waste

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 identification number (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
- Sample Location ID
- Contract Task Order Number (CTO 148)
- Sample ID

- Sample 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 Air Station (NAS) Pensacola. 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 Florida Department of Environmental Protection (FDEP) must approve any deviations from this procedure.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Pen with indelible ink

Sample tags

Sample container labels

3.0 SAMPLE IDENTIFICATION NOMENCLATURE

3.1 Soil 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 site, sample type, location, and sample depth. For soil samples, the final four tracking numbers will identify the depth in units of inches below ground surface (bgs) at which the sample was collected.

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

AN	AA	NNN	NNNN
Site ID	Matrix	Sample Location Number	Sequential depth interval from freshly exposed surface (inches)

Character Type:

A = Alpha

N = Numeric

Site IDs (AN):

X1 = UXO 01 (Magazine Point Bombing Target)

Matrix Code (AA):

IS = Incremental Soil Sample

Location Number (NNN):

Sequential number beginning with "001"

Depth Interval (NNNN):

For the soil samples, the final four tracking numbers will identify the depth in units of inches.

The depth code is used to note the depth bgs at which a soil sample is collected. The first two numbers of the four-number code specify the top interval, and the third and fourth specify the bottom interval of the sample depth. 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 Surface Soil Sample Nomenclature

The first incremental soil sample collected from Magazine Point Bombing Target at a depth of 0 to 6 inches bgs would be labeled as "X1-IS001A-0006".

3.2 Field Quality Control (QC) Sample Nomenclature

Field QC samples (duplicate and triplicate) are described in the UFP-SAP. These samples will be collected within the same grid as the original sample, but in a separate consistent location within the grid. These QC samples will incorporate the same nomenclature scheme as the original sample in numerical order. For instance, the location ID for the duplicate sample will be "002", and the location ID for the triplicate sample will be "003". No other QC samples are planned for this field event.

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 Boring Log

Soil and Sediment 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 Tetra Tech 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. At a minimum, the following activities and events will be recorded (daily) in the field logbooks:

- Field personnel for activities in the field logbook
- 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

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 field logbook:

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

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. Sample labeling and nomenclature are described in SOP-01 and SOP-02, respectively.

3.4 Chain-of-Custody Form

The Chain-of-Custody Form (COC) is initiated as samples are acquired and accompanies a sample (or group of samples) as it is transferred from person to person. This form must

accompany any samples collected for laboratory chemical analysis. Each COC will be uniquely numbered. 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 Tetra Tech Project Manager (PM). 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 is part of the chain-of-custody process. Custody seals are used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transit to the laboratory. Custody seals will be signed and dated by the samplers and affixed across the opening edges of each cooler (two seals per cooler on opposite sides) containing environmental samples. The laboratory sample custodian will examine the custody seal for evidence of tampering and will notify the Tetra Tech PM 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 the soil. A copy of the sample log sheet is attached at the end of this SOP. A sample log sheet will be prepared for each sample collected and submitted for laboratory analysis.

4.0 ATTACHMENTS

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

STANDARD OPERATING PROCEDURE

SOP-04

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 a temperature not to exceed 6 degrees Celsius (°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 a 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-milliliter 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 NUMBER SOP-05

INCREMENTAL SAMPLING (IS) FOR SOIL AND SEDIMENT

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for collecting incremental sampling (IS) methodology surface soil and/or sediment samples using a hand-operated coring device to support the field investigation at the Magazine Point Bombing Target site located at Naval Air Station (NAS) Pensacola. This SOP complies with United States Environmental Protection Agency (USEPA) SW-846 Method 8330B, Appendix A, and United States Army Corps of Engineers (USACE) guidance documents.

The most widely-known description of IS for environmental use is SW-846 Method 8330B, Appendix A. The specific sample collection and processing procedures described in Method 8330B were based primarily on studies by the USACE Cold Regions Research Engineering Laboratory (CRREL). These studies were designed to demonstrate and develop the methodology for application to the investigation of explosive compounds at active military testing and training ranges.

This technique will assist in estimating mean munitions constituent (MC) concentrations which can be used to assess whether potential MC:

- Are present within the sample area at an average concentration greater than the analytical method detection limit (MDL) or reporting limit (RL).
- May pose an unacceptable risk to human health, or ecological receptors.
- May contribute to significant contaminant concentrations in groundwater.
- Exhibit concentrations that exceed mean background or ambient concentrations unrelated to munitions activities.

A Sampling Unit (SU) [sometimes synonymous with a Decision Unit (DU)] is the area and depth of soil to be represented by the IS samples. SUs must be delineated so that the mean analyte concentrations obtained are directly relevant to well-defined project objectives. Typical SUs could be as small as 3 feet by 3 feet, or as large as 300 feet by 300 feet.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following field forms and equipment are required for IS of soil and/or sediment.

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

Indelible marker

CRREL coring device or equivalent

One coring shoe (size predetermined before sampling commences, based on number of increments, sample depth interval, and required sample size)

Plastic storage bags

Sample labels/tags

Shipping containers with ice (i.e., coolers)

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

Field Forms: Soil and Sediment Sample Log (SOP-03)

3.0 SAMPLING PROCEDURES

Hand coring will be employed to collect incremental soil cores of cohesive soils and/or sediments using a coring device and properly-sized coring shoe (or equivalent). This will ensure the increments of soil or sediment are collected in a consistent manner across the entire SU. This consistency maximizes the representativeness of the IS sample. Although the use of a hand trowel or hand auger may be unavoidable for some material, such tools are not recommended because they do not control the amount of material per increment, and uniform increments are critical to IS. If the soils and/or sediments are not cohesive, this SOP should not be used and an alternate procedure should be followed that ensures a representative sampling scheme that is appropriate for the situation at hand.

Practical limitations and unforeseen field conditions may require modifying the delineation of a SU as defined during planning. Conditions of this nature may include the presence of pavement, buildings, or exposed bedrock surface without soil. In general, significantly changing the total sampling area should be avoided. Changes and their rationale should be fully described in a field task modification request (FTMR) and in the project report.

A 1 to 2 kilogram (dry weight) IS sample usually ensures that sufficient mass has been collected to adequately represent the SU mean concentration.

The sampler will wear clean, disposable, medical-grade gloves and the coring device will undergo decontamination procedures according to SOP-06 at the beginning of each sample day and prior to

collection of subsequent samples. Decontamination of the coring device is not required between collection of the individual increments making up a single IS sample.

3.1 **INCREMENTAL SAMPLING PROCEDURES**

3.1.1 The correct size coring shoe (or equivalent) was determined based on the number of sample increments required, the sampling depth, and the average density of the soil or sediment. Since a 1 to 2 kilogram (dry weight) IS sample is preferred, the following table matches the project-specific need to the correct coring shoe size (or equivalent auger diameter and depth).

Coring Device Size Selection Based on Number of Increments and Sample Depth

Number of Increments	Sample Depth (inches)	Soil Density* (g/cc)	Minimum Corer Diameter - 1,000 grams total (nearest ¼ inch)	Maximum Corer Diameter - 2,000 grams total (nearest ¼ inch)
60 (15 x 4)	6	1.5	0.50	0.50

* Assumed soil density = 1.50 grams per cubic centimeter (g/cc) and assumed percent moisture is less than 10 percent. At the discretion of the Tetra Tech FOL, the coring device size may be adjusted based on site conditions that differ from these assumptions, but must be the same throughout the entire SU.

3.1.2 A hand-held Global Positioning System (GPS) (see SOP-07) meter will be utilized to locate the corners of the SU. Utilizing a measuring tape, grid off the 375 foot long by 120 foot wide SU into 60 increments (cells) of equal size (4 squares wide by 15 squares long, resulting in each grid cell being 30 feet wide and 25 feet long). Stakes, flagging, or other means of clear visual reference should be used so the field sampler can accurately identify each grid.

3.1.3 Starting in one cell (a corner cell is recommended), systematically sample each grid cell as described in Steps 3.1.3.1 through 3.1.3.4. A successful systematic increment collection scheme is to start in one corner of the SU and work in a back-and-forth path, traveling to each successive cell until an increment has been collected from each grid cell.

3.1.3.1 Randomly select a single increment sampling point in the initial grid cell, then collect all subsequent increments from the same relative location within each of the other grid cells.

- 3.1.3.2 Turn or push the coring device into the ground to the desired depth (i.e., 6 inches). Remove the coring device and visually verify that the entire core was retained. If the coring device is not filled with soil, collect remaining soil from the hole to fill the coring device with the intended soil volume.
- 3.1.3.3 Eject the soil increment into a labeled sample container such as a large sealable plastic bag or other suitable container which must be large enough to hold all of the increments to be collected from the SU (between 1 and 2 kilograms).
- 3.1.3.4 Field triplicate samples must be collected at a minimum frequency of 1 per 10 IS samples in order to provide precision data that will be used to support the data evaluation process. Field triplicates for IS are not field split samples; rather, they are independently collected incremental samples from the same SU. At the NAS Pensacola Magazine Point Bombing Target site, this will be accomplished by randomly selecting a single increment sampling point for the duplicate in the initial grid cell that is different than the original sample location, then collecting all subsequent increments from the same relative location within each of the other grid cells. Collect the triplicate sample in the same manner as the original and duplicate samples, but in a random location within the initial cell grid that is different than the original and duplicate sample locations. Collect all subsequent increments from the same relative location within each of the other grid cells.
- 3.1.4 Repeat steps 3.1.3.1 to 3.1.3.4 in the designated SU until the entire sample grid has been sampled.
- 3.1.5 For each of the three replicate ISs collected from the SU, package the entire lot of collected soil in accordance with SOP-04 and ship them to the laboratory via overnight courier for processing and analysis.
- 3.1.6 Complete the required information on the Soil and Sediment Sample Log Sheet (see attachment). Although it is not necessary to record the locations of individual increments with the GPS, the location of the individual increments within the grid will be noted 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 should be visible on the Chain-of-Custody form for review by laboratory personnel.

3.1.7 Decontaminate the coring device in accordance with SOP-06 between collection of each of the three IS samples (original, duplicate, triplicate).

4.0 REFERENCES

GPL Laboratories, LLC. 2009. SOP No. G.22: General Laboratory Multi Incremental Sampling (MIS) sub-sampling procedure. January.

US Army Corps of Engineers (USACE). 2004. Field Sampling Tools for Explosives Residues Developed at CRREL. ERDC/CRREL TN-04-1. April.

USACE. 2007. Protocols for Collection of Surface Soil Samples at Military Training and Testing Ranges for the Characterization of Energetic Munitions Constituents. ERDC/CRREL TR-07-10. July.

USACE. 2009. Interim Guidance 09-02: Implementation of Incremental Sampling (IS) of Soil for the Military Munitions Response Program. July.

United States Environmental Protection Agency (USEPA), 2006. SW-846 Method 8330B, Appendix A. Office of Solid Waste and Emergency Response, Washington, DC. October.

5.0 ATTACHMENTS

1. Soil and Sediment Sample Log Sheet

STANDARD OPERATING PROCEDURE

SOP-06

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)

Liqui-Nox® or Alconox® 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 Liqui-Nox® or Aloconox® detergent. Prepare the detergent wash solution in accordance with the instructions on the detergent container. Collect the 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 detergent 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 and 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-08.

STANDARD OPERATING PROCEDURE

SOP-07

GLOBAL POSITIONING SYSTEM

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide the field personnel with basic instructions for operating a handheld Global Positioning System (GPS) unit allowing them to set GPS parameters in the receiver, record GPS positions on the field device, and update existing Geographic Information System (GIS) data. This SOP is specific to GIS quality data collection for Trimble®-specific hardware and software.

If possible, the Trimble® GeoXM™ or GeoXH™ operators manual should be downloaded onto the operator's personal computer for reference before or while in the field. The manual can be downloaded at <http://trl.trimble.com/docushare/dsweb/Get/Document-311749/TerraSyncReferenceManual.pdf>

Unless the operator is proficient in the setup and operation of the GPS unit, the Project Manager (or designee) should have the GPS unit shipped to the project-specific contact listed below in the Pittsburgh, Pennsylvania, office at least five working days prior to field mobilization so project-specific shape files, data points, background images, and correct coordinate systems can be uploaded into the unit.

Tetra Tech NUS, Inc.
Attn: John Wright
661 Anderson Drive, Bldg #7
Pittsburgh, PA 15220

2.0 REQUIRED EQUIPMENT

The following hardware and software should be utilized for locating and establishing GPS points in the field:

2.1 Required GPS Hardware

- Hand-held GPS unit capable of sub-meter accuracy (i.e. Trimble® GeoXM™ or Trimble® GeoXH™). This includes the docking cradle, A/C adapter, stylus, and USB cable for data transfer.

Optional Accessories:

- External antenna
- Range pole
- Hardware clamp (for mounting GPS unit to range pole)
- GeoBeacon
- Writing utensil (preferably black pen with indelible ink)
- Non-metallic pin flags for temporary marking of positions

2.2 Required GPS Software

The following software is required to transfer data from the handheld GPS unit to a personal computer:

- Trimble® TerraSync version 2.6 or later (pre-loaded onto GPS unit from vendor)
- Microsoft® ActiveSync® version 4.5 or later. Download to personal computer from:
<http://www.microsoft.com/windowsmobile/en-us/downloads/microsoft/activesync-download.mspx>
- Trimble® Data Transfer Utility (freeware version 2.1 or later). Download to personal computer from:
<http://www.trimble.com/datatransfer.shtml>

3.0 START-UP PROCEDURES

Prior to utilizing the GPS in the field, ensure the unit is fully charged. The unit may come charged from the vendor, but an overnight charge is recommended prior to fieldwork.

The Geo-series GPS units require a docking cradle for both charging and data transfer. The Geo-series GPS unit is docked in the cradle by first inserting the domed end in the top of the cradle, then gently seating the contact end into the latch. The power charger is then connected to the cradle at the back end using the twist-lock connector. Attach a USB cable as needed between the cradle (B end) and the laptop/PC (A end).

It is recommended that the user also be familiar and check various Windows Mobile settings. One critical setting is the Power Options. The backlight should be set as needed to conserve power when not in use.

Start Up:

- 1) Power on the GPS unit by pushing the small green button located on the lower right front of the unit.
- 2) Utilizing the stylus that came with the GPS unit, launch **TerraSync** from the Windows Operating System by tapping on the start icon located in the upper left hand corner of the screen and then tap on **TerraSync** from the drop-down list.
- 3) If the unit does not default to the Setup screen, tap the Main Menu (uppermost left tab, just below the Windows icon) and select Setup.
- 4) If the unit was previously shipped to the Pittsburgh office for setup, you can skip directly to Section 4.0. However, to confirm or change settings, continue on to Section 3.1.

3.1 Confirm Setup Settings

Use the Setup section to confirm the TerraSync software settings. To open the Setup section, tap the Main Menu and select Setup.

- 1) Coordinate System
 - a. Tap on the Coordinate System.
 - b. Verify the project specs are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu.
Note: It is always best to utilize the Cancel tab rather than the OK tab if no changes are made since configurations are easily changed by mistake.
 - c. Tap on the Units.
 - d. Verify the user preferences are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu.
 - e. Tap Real-time Settings.
 - f. Verify the Real-time Settings are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu.
 - g. The GPS unit is now configured correctly for your specific project.

4.0 ANTENNA CONNECTION

- 1) If a connection has been properly made with the internal antenna, a satellite icon along with the number of usable satellites will appear at the top of the screen next to the battery icon. If no connection is made (e.g.: no satellite icon), tap on the GPS tab to connect antenna.
- 2) At this point the GPS unit is ready to begin collecting data.

5.0 COLLECTING NEW DATA IN THE FIELD

- 1) From the Main Menu select Data.
- 2) From the Sub Menu (located below the Data tab) select New which will bring up the New Data File menu.
- 3) An auto-generated filename appears and should be edited for your specific project. If the integral keyboard does not appear, tap the small keyboard icon at the bottom of the screen.
- 4) After entering the file name, tap Create to create the new file.
- 5) Confirm antenna height if screen appears. Antenna height is the height that the GPS unit will be held from the ground surface (Typically 3 to 4 feet).
- 6) The Choose Feature screen appears.

5.1 Collecting Features

- 1) If not already open, the Collect Feature screen can be opened by tapping the Main Menu and selecting Data. The Sub Menu should default to Collect.
- 2) **Do not begin the data logging process until you are at the specific location for which you intend to log the data.**
- 3) A known reference or two should be shot at the beginning and at the end of each day in which the GPS unit is being used. This allows for greater accuracy during post-processing of the data.
- 4) Upon arriving at the specific location, tap on Point_generic as the Feature Name.
- 5) Tap Create to begin data logging.
- 6) In the Comment Box enter sample ID or location-specific information.
- 7) Data logging can be confirmed by viewing the writing pencil icon in the upper part of the screen. Also, the logging counter will begin. As a Rule of Thumb, accumulate a minimum of 20 readings on the counter, per point, as indicated by the logging counter before saving the GPS data.
- 8) Once the counter has reached a minimum number of counts (i.e. 20), tap on OK to save the data point to the GPS unit. Confirm the feature. All data points are automatically saved within the GPS unit.

- 9) Repeat steps 2 through 8, giving each data point a unique name or number.

Note: If the small satellite icon or the pencil icon is blinking, this is an indication the GPS unit is not collecting data. A possible problem may be too few satellites. While still in data collection mode, tap on Main Menu in upper left hand corner of the screen and select Status. Skyplot will display as the default showing the number of available satellites. To increase productivity (number of usable satellites) use the stylus to move the pointer on the productivity and precision line to the left. This will decrease precision, but increase productivity. The precision and productivity of the GPS unit can be adjusted as the number of usable satellites changes throughout the day. To determine if GPS is correctly recording data, see Section 5.2.

5.2 Viewing Data or Entering Additional Data Points to the Current File

- 1) To view the stored data points in the current file, tap on the Main Menu and select Map. Stored data points for that particular file will appear. Use the +/- and <-/-> icons in lower left hand corner of screen to zoom in/out and to manipulate current view.
- 2) To return to data collection, tap on the Main Menu and select Data. You are now ready to continue to collect additional data points.

5.3 Viewing Data or Entering Data Points from an Existing File

- 1) To view data points from a previous file, tap on Main Menu and select Data, then select File Manager from the Sub Menu.
- 4) Highlight the file you want to view and select Map from the Main Menu.
- 5) To add data points to this file, tap on Main Menu and select Data. Continue to collect additional data points.

6.0 NAVIGATION

This section provides instructions on navigating to saved data points in an existing file within the GPS unit.

- 1) From the Main Menu select Map.
- 2) Using the Select tool, pick the point on the map to where you want to navigate.
- 3) The location you select will have a box placed around the point.
- 4) From the Options menu, choose the Set Nav Target (aka set navigation target).
- 5) The location will now have double blue flags indicating this point is you navigation target.

- 6) From the Main Menu select Navigation.
- 7) The dial and data on this page will indicate what distance and direction you need to travel to reach the desired target.
- 8) Follow the navigation guide until you reach the point you select.
- 9) Repeat as needed for any map point by going back to Step 1.

7.0 PULLING IN A BACKGROUND FILE

This section provides instructions on pulling in a pre-loaded background file. These files are helpful in visualizing your current location.

- 1) From the Main Menu select Map, then tap on Layers, select the background file from drop down list.
- 2) Select the project-specific background file from the list of available files.
- 3) Once the selected background file appears, the operator can manipulate the screen utilizing the +/- and <-/> functions at the bottom of the screen.
- 4) In operating mode, the operator's location will show up on the background file as a floating "x".

8.0 DATA TRANSFER

This section provides instructions on how to transfer stored data on the handheld GPS unit to a personal computer. Prior to transferring data from the GPS unit to a computer, Microsoft ActiveSync and Trimble Data Transfer Utility software must be downloaded to the computer from the links provided in Section 2.2 (Required GPS Software). If a leased computer is utilized in which the operator cannot download files, see the Note at the end of Section 8.0.

- 1) See Attachment A at the end of this SOP for instructions on how to transfer data from the GPS to a personal computer.

Note: If you are unable to properly transfer data from the GPS unit to a personal computer, the unit should be shipped to the project-specific contact listed in Section 1.0 where the data will be transferred and the GPS unit then shipped back to the vendor.

9.0 SHUTTING DOWN

This section provides instruction for properly shutting down the GPS unit.

- 1) When shutting down the GPS unit for the day, first click on the "X" in the upper right hand corner.
- 2) You will be prompted to ensure you want to exit TerraSync. Select Yes.
- 3) Power off the GPS unit by pushing the small green button located on the bottom face of the unit.
- 4) Place the GPS unit in its cradle to recharge the battery overnight. Ensure the green charge light is visible on the charging cradle.

ATTACHMENT A

How to Transfer Trimble GPS Data between Data Collector and PC
original 11/21/06 (5/1/08 update) – John Wright

Remember – Coordinate System, Datum, and Units are critical!!!

Trimble Data Collection Devices:

Standard rental systems include the Trimble® ProXR/XRS backpack and the newer handheld GeoXT™ or GeoXH™ units. Some of the older backpack system may come with either a RECON “PDA-style” or a TSCe or TSC1 alpha-numeric style data collector.

The software on all of the above units should be Trimble® TerraSync (v 2.53 or higher – current version is 3.20) and to the user should basically look and function similar. The newer units and software versions (which should always be requested when renting) include enhancements for data processing, real-time display functions, and other features.

Data Transfer:

Trimble provides a free transfer utility program to aid in the transfer of GIS and field data. The Data Transfer Utility is a standalone program that will run on a standard office PC or laptop.

To connect a field data collector such as a RECON, GeoXM, GeoXT, GeoXH, or ProXH, you must first have Microsoft® ActiveSync® installed to allow the PC and the data collector to talk to one another. A standard USB cable is also needed to connect the two devices.

A CD or USB drive is provided with the data collector for use in data transfer. If needed, these programs are also available without charge via the web at:

- **Trimble Data Transfer Utility** (v 1.38) program to download the RECON or GeoXH field data to your PC: <http://www.trimble.com/datatransfer.shtml>

- **ActiveSync** from Microsoft to connect the data collector to the PC. The latest version (v4.5) can be found at: <http://www.microsoft.com/windowsmobile/en-us/downloads/microsoft/activesync-download.msp>

(see page 2 for data transfer instructions)

To Transfer Data Collected in the Field:

- Install the Data Transfer and ActiveSync software installed on your PC
- Connect the RECON or GeoXH to your PC via an A/B USB cable (blade end and square end type "HP printer" style)
- ActiveSync should auto-detect the connection and recognize the data collector
- Make sure the data file desired is CLOSED in TerraSync prior to transfer
- Connect via ActiveSync as a guest (not a partnership)
- Run the Trimble Data Transfer Utility program on your PC
- Select "**GIS Datalogger on Windows CE**" or similar selection
- Hit the green connect icon to the right - the far right area should say "**Connected to**" if successful
- Select the "**Receive**" data tab (under device)
- Select "**Data**" from file types on the right
- Find the file(s) needed for data transfer. You can sort the data files by clicking on the date/time header
- Select or browse to a C-drive folder you can put this file for emailing
- When the file appears on the list, hit the "**Transfer All**"
- Go to your Outlook or other email, send a message to: John.Wright@tetrattech.com (or GIS department)
- Attach the file(s) you downloaded from your C-drive. For each TerraSync data file created you should have a packet of multiple data files. All need to be sent as a group – make sure you attach all files (the number of files may vary – examples include: ssf, obx, obs, gix, giw, gis, gip, gic, dd, and car)

To Transfer GIS Data from PC to the Field Device (must be converted in Pathfinder Office):

- Obtain GIS file(s) desired from GIS Department and have converted to Trimble extension
- Contact John Wright (John.Wright@tetrattech.com) if needed for file conversion and upload support
- The GIS file(s) can be quickly converted if requested and sent back to the field user in the needed "Trimble xxx.imp" extension via email – then quickly downloaded from Outlook to your PC for transfer
- Install the Data Transfer and ActiveSync software installed on your PC
- Connect the RECON or GeoXH to your PC via an A/B USB cable (blade end and square end type "HP printer" style)
- ActiveSync should auto-detect the connection and recognize the data collector
- Connect via ActiveSync as a guest (not a partnership)
- Run the Trimble Data Transfer Utility program on your PC
- Select "**GIS Datalogger on Windows CE**" or similar selection
- Hit the green connect icon to the right - the far right area should say "**Connected to**" if successful
- Select the "**Send**" data tab (under device)
- Select "**Data**" from file types on the right (you can also send background files)
- Browse to the location of the data on your PC (obtain the file from Pathfinder Office or from the person who converted the data for field use)
- Select the options as appropriate for the name and location of the data file to go on the data collector (usually you can choose main memory or a data storage card)
- When the file(s) appears on the list, hit the "**Transfer All**"
- Run TerraSync on the field device and open the existing data files. Your transferred file should appear (make sure you have selected Main Memory, Default, or Storage Card as appropriate)

STANDARD OPERATING PROCEDURE

SOP-08

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 Naval Air Station (NAS) Pensacola. The following types of IDW may be generated during this investigation:

- Decontamination solutions
- Personal protective equipment (PPE) and clothing
- Miscellaneous trash and incidental items

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Health and safety equipment (with PPE)

CRREL coring device or similar Bucket (with collected development/purge water)

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 may be generated during the site activities include decontamination solutions from sampling equipment. These wastes will be collected and containerized in a central location at NAS Pensacola for proper disposal.

3.2 Solid Wastes

No solid wastes are expected to be generated during this investigation.

3.3 PPE and Incidental Trash

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

APPENDIX B

**EMPIRICAL LABORATORIES SOPs AND
DoD ELAP ACCREDITATION**

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

INORGANICS: SOP100 REVISION #: 22 EFFECTIVE DATE: 20101117

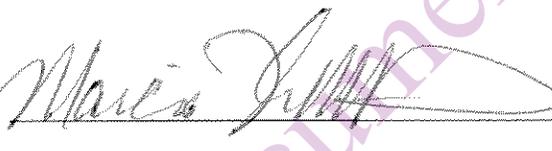
METALS DIGESTION/PREPARATION

References:

Methods 3005A/USEPA CLPILM0 4.1 Aqueous, 3010A, 3030C, 3050B
USEPA CLPILM0 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C 21st
See Addendum for USEPA CLPILM 05.2 (Aqueous & Soil/Sediment)

APPROVALS:

Lab Director:  Date: 11/19/10

Data Quality Manager:  Date: 11/19/10

Section Supervisor:  Date: 11/19/10

Changes Summary

Revision 22, 11/17/10

- The SOP is an update from Revision 21 dated 9/1/10
- Revised to add the need for matrix spike duplicates to be digested and analyzed for TCLP extracts.
- Requirement to hold samples 24 hours after in-house preservation was added to section III.

Revision 21, 9/1/10

- The SOP is an update from Revision 20 dated 04/27/10
- The SOP has been found to be up-to-date with Standard Methods 21st edition.
- Reference to adjusting filtrate volume for method 3030C has been removed.
- References to bound logbooks have been replaced with LIMS references.

Revision 20, 4/27/10

- The SOP is an update from Revision 19 dated 04/20/09.
- References to oil sample preparation have been removed.
- Extraction volumes for TCLP have been updated.

METALS DIGESTION/PREPARATION

References:

**Methods 3005A/USEPA CLPILM0 4.1 Aqueous, 3010A, 3030C, 3050B
USEPA CLPILM0 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C
See Addendum for USEPA CLPILM 05.2 (Aqueous & Soil/Sediment)**

I. SCOPE AND APPLICATION

A. AQUEOUS

1. Method 3005A and USEPA CLP ILM0 4.1, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP Spectroscopy".
 - a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
2. Method 200.7, "Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry"
 - a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
3. Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy".
 - a. This method is used to prepare aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for analysis by ICP. The procedure is used to determine total metals.
4. Method 3030C (Standard methods), "Preliminary Treatment for Acid-Extractable Metals".
 - a. This method is used to prepare ground water samples from North Carolina for analysis by ICP.

B. SOLIDS

1. Method 3050B, "Acid Digestion of Sediments, Sludges and Soils".
 - a. This method is used to prepare sediments, sludges and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.
 - b. It should be noted that some metals could be biased high with the soil digestion when dilution is necessary. Take necessary measures to ensure that dilutions are made as accurately as possible.
2. USEPA CLP ILM0 4.1, "Acid Digestion of Soil/Sediment"
 - a. This method is used to prepare sediments and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.

D. NOTES:

1. "Total Metals" includes all metals, inorganically and organically bound and both dissolved and particulate.
2. "Dissolved metals" includes all metals present in a sample after filtration through a 0.45 micron filter followed by digestion.

II. SUMMARY OF METHODS

A. A representative sample of water or soil is put into an acid medium and exposed to heat for a certain amount of time. This allows for reduction of interferences by organic matter and converts metals bound to particulates to form the free metal that can be determined by ICP-Atomic Emission Spectrometry.

NOTE: When a reporting limit is required for a project lower than is customary, a four times concentration or alternate soil digestion ratio must be used in order to reach that lower level. Care must be taken to matrix match this concentrated aliquot. A blank and laboratory control sample (at a reduced concentration) are required with this concentration. A matrix spike (not at reduced concentration) and duplicate or matrix spike and matrix spike duplicate is needed per 20 samples or per batch.

III. SAMPLE HANDLING AND PRESERVATION

A. AQUEOUS

1. Samples are taken in high density polyethylene, one liter bottles. Samples should be preserved with concentrated HNO₃ to a pH <2 immediately upon sampling. If dissolved metals are to be analyzed the sample should be filtered before the HNO₃ is added. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months. Note – samples received unpreserved and preserved in-house must be held 24 hours prior to preparation.

B. SOLIDS

1. Samples are taken in high density polyethylene (CLP only) or glass bottles. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

IV. INTERFERENCES

A. AQUEOUS

1. Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

B. SOLIDS

1. Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether this method is applicable to a given waste.

V. SAFETY

A. Normal accepted laboratory safety practices should be followed while performing this analysis.

B. Be certain the exhaust hood is functioning before you begin the digestion procedure.

C. Hot acids can be extremely corrosive. Avoid inhalation or contact with skin.

VI. EQUIPMENT/APPARATUS

A. Fume hood, Labconco or equivalent.

B. Hot plate, Thermolyne cimarec-3 or equivalent source for use at 95°C. The temperature of the hot plate must be monitored via the use of a temperature blank.

- C. Thermometer capable of reading 80 to 120 degrees C – ERTCO cat# 611-3-SC or equivalent.
 - D. Vacuum pump for filtering dissolved metals- Gast or equivalent.
 - E. Analytical balance capable of weighing to 0.01 gram. Mettler model BB300 or equivalent.
 - F. Beckman CS-6R centrifuge.
 - G. Various class A volumetric glassware and ribbed watchglasses, Pyrex or equivalent.
 - H. Whatman No. 41 filter paper or equivalent.
 - I. Whatman No. 42 filter paper or equivalent.
 - J. Whatman 0.45 micron filter paper or equivalent.
 - K. 250 mL beaker or other appropriate vessel such as polypropylene block digester tubes, watch glasses and caps.
 - L. Stirring device, e.g. magnetic stirrer, glass rod or equivalent.
 - M. Manual Sample Mill
 - N. Wiley Sample Mill
 - O. Clippers for cutting vegetation
- NOTE:** All glassware should be acid washed.

VII. REAGENTS AND STANDARD PREPARATION

A. REAGENTS

1. Metals grade Nitric acid (HNO₃). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
2. Metals grade Hydrochloric acid (HCl). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
3. 30% hydrogen peroxide reagent, ACS Grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
4. Metals grade Sulfuric acid (H₂SO₄). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
5. Reagent water (Deionized water).
6. Potassium Permanganate - Ultra pure grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
7. Ammonium hydroxide, concentrated, reagent grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
8. Ammonium phosphate, reagent grade- Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

B. STANDARDS

1. Traceability

- a. A LIMS record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the LIMS as well as on the container's label.
- b. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date and the information is recorded in LIMS. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in LIMS. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with

sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

c. The analyst must initial and date each entry made in LIMS.

2. PREPARATION

A. Laboratory control sample

1. Aqueous

- a. This solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO₃, 1 mL of CLP-CAL-1, Solution A, 1 mL of CLP-CAL-1 Solution B, 0.25 mL of CLP-CAL-2, and 0.25 mL of CLP-CAL-3 diluted to 1 L in a volumetric flask. Use 50 mL (100 mL for strict CLPILM0 4.1) for digestion. This solution is given a unique identifier and recorded in sample LIMS.
- b. For four times concentrated samples: The solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO₃, 1mL CLPP-SPK-4 (Inorganic Ventures) (This solution contains 10 mg/L Selenium, 100 mg/L Antimony, 50 mg/L Cadmium and Thallium, 40 mg/L Arsenic and 20 mg/L Lead) to 1 L in a volumetric flask. This solution is given a unique identifier. Use 12.5 mLs to 50 mLs and prepare two aliquots. Heat at 90 to 95°C to reduce the volume in each vessel to ten mLs and then combine each 10 mL aliquot into one vessel and take to a final volume of 25 mLs. Take care to matrix match acids so that the final 25 mL portion will contain 2% HNO₃ and 5% HCl. Use 0.125 mLs HNO₃ and 0.3125 mLs HCl to each 50 mL vessel.

2. Solids:

- a. 1.0 ±0.02 (or 2.0 ±0.02) gram aliquot of teflon chips is weighed and spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier according to the Lot# for the teflon chips used and when digested is given the descriptor. i.e. BS1 and then BS2 etc. plus the unique identifier number assigned. Alternatively a solid matrix standard reference material is obtained from the manufacturer. This sample is given a unique identifier and the weight is recorded in a bound logbook and transferred to LIMS.

B. Spiking solution

1. Sample is spiked using 0.1 mL of CLP-CAL-1, Solution A, 0.1 mL of CLP-CAL-1 Solution B, 0.025 mL of CLP-CAL-2 and 0.025 mL of CLP-CAL-3 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers. Record the amount spiked and the unique identifier of the standard.
2. CLP sample is spiked using 0.1 mL CLPP-SPK-1 and 0.1 mL CLPP-SPK-4 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers.
3. For samples that require four times concentration, the sample is spiked using 0.0125 mLs of CLPP-SPK-4 to each of two vessels with 50 mLs of sample in each. The volume of each of the vessels is lowered to less than 10 mLs and combined and the final volume of this concentrated sample is 25mLs.

VIII. CALIBRATION

- A. The temperature of the samples must be maintained at 95°C and monitored via a temperature blank. Record in temperature logbook for later transfer into LIMS.

IX. PROCEDURE

- A. Glassware preparation for digestion or when the hot-block can not be used:
 1. Wash glassware with hot soapy water and rinse thoroughly. (Beakers must be washed as soon as possible after being used, dirty beakers must not be allowed to sit overnight.)
 2. Rinse glassware with reagent water that contains 5% HNO₃ and 5% HCl followed by a rinse with reagent water.
 3. Prior to use, all glassware must be confirmed clean via a glassware check. Otherwise, repeat step "2" until the glassware check passes.
- B. Aqueous sample filtration (for dissolved metals):
 1. Thoroughly clean a flask and funnel with hot soapy water. Next, rinse the flask and funnel with 1:5 HNO₃ followed by a thorough D.I. water rinsing. This step is very important because the filters contain some metals (namely Zn) which could contaminate the samples.
 2. Rinse a 0.45 micron filter with 1:5 HNO₃ thoroughly, followed by D.I. water.
 3. Filter the unpreserved sample. If dissolved Hg analysis is requested for the sample, filter at least 200 mL.
 4. Discard the first 50 to 100 mL.
 5. A preparation blank must be taken through the filtration step and analyzed with the sample.
 6. Preserve the sample with HNO₃ to pH<2.
 7. Soluble samples that are clean and clear do not have to be digested. Use 100 mL sample, add 5 mL of concentrated HCl and 2 mL of concentrated HNO₃. **Samples must be digested unless approval for analysis without digestion is received from the project manager.**
- C. Aqueous sample preparation
 1. Method 3005A and USEPA CLP ILM0 4.1, "**Acid digestion procedure for total recoverable or dissolved metals for analysis by ICP**".
 - a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a digestion vessel. For samples which require concentration pour 50 mLs of the well-mixed sample into two digestion vessels.
 - b. Add 0.50 mL (1 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO₃ to the sample. For samples which require concentration, add 0.125 mL (0.25 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO₃ to the sample.
 - c. Add 2.5 mL (5 mL of 1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample. For samples which require concentration, add 0.3125 mL (0.625 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample.
 - d. Cover the sample with a ribbed watch glass or equivalent source.
 - e. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank will assure correct temperature. The temperature must be

- recorded in the temperature logbook. Take the volume down to between 5 to 10 mL, (12 to 25 mLs when strict CLP ILM0 4.1 is required) **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes.** Remove the sample from the hot plate and cool
- f. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
 - g. Bring sample to its predigestion volume (or when samples require concentration, to a volume four times lower then what was started with) with DI water in the digestion vessel. The final volume must be recorded in the LIMS.
 - h. The sample is now ready for analysis.
 - i. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards plus identification #'s for standards used for spiking and the volume spiked into the sample.
2. Method 200.7, "**Acid digestion procedure for total recoverable metals**".
- a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into the digestion vessel. If sample contains undissolved solids >1% refer to Section 11.3 of Method 200.7 for subsequent procedures.
 - b. Add 1.0 mL concentrated HNO₃ to the sample.
 - c. Add 2.50 mL concentrated HCl to the sample.
 - d. Cover the sample with a ribbed watch glass or equivalent source.
 - e. Transfer the digestion vessel to a pre-heated hot plate or equivalent source at 85°C. Take the volume down to between 10 to 15 mL, **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes.**
 - f. Leave sample on hot plate and gently reflux for 30 minutes. Remove from hot plate and cool.
 - g. Bring sample to its predigestion volume with DI water in the digestion vessel.
 - h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
 - i. The sample is now ready for analysis.
 - j. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.
3. Method 3010A, "**Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy**".
- a. Shake sample thoroughly and pour 50 mL (5ml diluted to 50mL for TCLP, full 50ml volume for SPLP) of the well-mixed sample into the digestion vessel.
 - b. Add 1.5 mL concentrated HNO₃ to the sample.
 - c. Cover the sample with a ribbed watch glass.
 - d. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank must be used, with the temperature being recorded in the temperature logbook. Take the volume down to a low volume (~5 mL), **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes. Also make certain that no portion of**

the bottom of the digestion vessel is allowed to go dry. This may lead to low recoveries. Remove the sample from the hot plate and cool.

- e. Add another 1.5 mL portion of concentrated HNO₃ to the sample.
 - f. Cover the sample with a ribbed watch glass.
 - g. Transfer the vessel to the hotblock or equivalent source. Increase the temperature so a gentle reflux occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).
 - h. Uncover the vessel and evaporate to a low volume (~3 mL) **making certain that no portion of the bottom of the digestion vessel is allowed to go dry.** Remove and cool.
 - i. Add 2.5 ml of 1:1 HCl (10 mL/100 mL of final solution).
 - j. Cover the digestion vessel and reflux for an additional 15 minutes.
 - k. Bring sample to its predigestion volume in digestion vessel.
 - l. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
Note: When preparing DoD project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.
 - m. The sample is now ready for analysis.
 - n. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.
- 4 Method 3030C (Standard Methods), "**Preliminary treatment for Acid-Extractable Metals**"
- a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a 50 mL digestion vessel.
 - b. Add 2.5 mL 1:1 HCl to the sample.
 - c. Heat 15 minutes in a hot bath.
 - d. Filter through a membrane filter.
 - e. Transfer to ICP analyst.

D. Solid sample preparation

It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

- *The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

This procedure should be repeated several times until the sample is adequately mixed.

NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.

Grinding of Vegetation Samples

Remove sample from shipping container and brush off dirt particles. Chop sample into about half inch pieces with clippers or other cutting tool. Place the sample in an aluminum pan and air-dry in an exhaust hood to the appropriate dryness for grinding. It should be dry enough where it won't stick to the inside of the mill. Grind the dried sample to fineness in either the manual sample mill or the Wiley mill or both if needed. Place the ground sample in a container and label immediately.

1. USEPA CLP ILM0 4.1, "**Acid digestion of Soil/Sediment**"
 - a. Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh (to the nearest 0.01 g) a 1.0 to 1.5 g portion of sample and transfer to a digestion vessel.
 - b. Add 10 mL of 1:1 nitric acid (HNO_3), mix the slurry, and cover with a watch glass or equivalent source. Heat the sample to 92 to 95°C and reflux for 10 minutes without boiling. Allow the sample to cool, add 5.0 mL of concentrated HNO_3 , replace with watch glass or equivalent source, as appropriate, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the heating vessel.
 - c. After the second reflux step has been completed and the sample has cooled, add 2 mL of Type II water and 3.0 mL of 30% hydrogen peroxide (H_2O_2). Return the heating vessel to the hot plate or equivalent heating source for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides, and cool the heating vessel.
 - d. Continue to add 30% H_2O_2 in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 mL 30% H_2O_2 .)
 - e. If the sample is being prepared for ICP analysis of Al, As, Sb, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, and Zn, add 5 mL of 1:1 HCl and 10 mL of Type II water, return the covered heating vessel to the hot plate or equivalent heating source, and heat for an additional 10 minutes. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 50 mL with Type II water. NOTE: In place of filtering, the sample (after dilution and mixing) may be centrifuged or allowed to settle by gravity overnight to remove insoluble material. Dilute the digestate to 144 mL with DI water, add 5 mLs concentrated HCl and 1 mL of concentrated HNO_3 , mix well and place into the appropriate container. The diluted sample has an approximate acid concentration of 2.5% (v/v) HCl and 5% (v/v) HNO_3 . The sample is now ready for analysis.
 - f. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards and ID of matrix spikes and the amounts used for spiking.
2. Method 3050B, "**Acid digestion of Sediments, Sludges and Soils**"

- a. Mix the sample thoroughly for 5 minutes using a plastic spatula or Teflon coated spatula in a glass or plastic weigh boat to achieve homogeneity.
- b. Weigh approximately (to the nearest 0.01 g) a 1 to 1.5 g portion of the sample directly into a digestion vessel. For samples with low percent solids a larger sample size may be used as long as digestion is completed. Record the exact mass in the LIMS.

NOTE: To achieve the lowest reporting limit possible, use a 2.0 g portion of sample with an ending volume of 100 mLs.

- c. Add 5 mL D.I. water and 5 mL concentrated $\text{HNO}_3(1:1)$, mix the slurry and cover with a watch glass. Place the sample in a preheated hot block and reflux at 95°C for 10 to 15 minutes being certain that the sample does not boil. Record temperature in temperature logbook
- d. Allow the sample to cool. Add 5 mL concentrated HNO_3 , replace the watch glass and heat/reflux again 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) over and over until no brown fumes are given off by the sample indicating the complete reaction with HNO_3 . Using a watch glass or equivalent allow the solution to evaporate to approximately 5 mL without boiling at $95^\circ\text{C} \pm 5^\circ\text{C}$ for approximately two hours. Maintain a covering of solution over the bottom of the vessel at all times. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the beaker. If the volume does get low, add 2.5 mL of D.I. water to bring volume back up.
- e. Take the sample off the hot block and allow it to cool. Next, add 2 mL of D.I. water and 3 mL of 30% Hydrogen Peroxide. (The sample will bubble upon the addition of H_2O_2 if it is still warm.) Cover the vessel with a watch glass and return the sample to the hot block or equivalent source and heat until the bubbling subsides. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker. Add two more 3 mL portions of H_2O_2 to the sample in the same manner as before. (NOTE: Do not add more than a total of 10 mL 30% H_2O_2 .)
- f. Cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate at $95^\circ\text{C} \pm 5^\circ\text{C}$ without boiling for approximately two hours until the volume has been reduced to approximately 2.5 mL. Maintain covering of solution over the bottom of the vessel at all times.
- g. Add 2.5 mL of DI water and 2.5 mL of concentrated HCl and 10 mL of DI water, cover the sample with a ribbed watch glass and continue refluxing for an additional 10 minutes without boiling
- h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
- i. Bring sample up to 50 mL with D.I. water in the vessel. Add 150 ml of DI water to a 250 ml sample bottle. Invert the 50 ml sample digestion vessel several times to mix the sample and pour sample into the 150 ml of the sample bottle. Pour some sample back into the 50 ml sample digestion vessel to rinse and pour back into the 250 ml sample bottle and cap and mix.

NOTE1: When preparing DoD project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.

NOTE2: To achieve the lowest reporting limit possible use 2.0 grams of sample with an ending volume of 100 mLs.

- j. The sample is now ready for analysis.
- k. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

X. CALCULATIONS

- A. The analyst must be supplied with both beginning sample masses/volumes and final digestate volumes. This information must be recorded in the digestion log.

XI. QUALITY CONTROL

A. Digestion

1. Temperature blank

- a. The temperature of the hot plate/hot block must be monitored for temperature during the digestion process.
- b. The thermometer must be tagged with annual calibration information. Record the thermometer reading, correction factor and the corrected temperature in the digestion log.

2. Blanks

- a. Digest a blank with every batch of samples digested (20 sample maximum). The blank is prepared by adding all the same reagents added to the samples to a clean dry beaker and taking it through the same process as the samples.
- b. Also, there must be a blank for every different method of digestion that is set up that day, every 20 samples.
- c. There must also be a blank for every different matrix of samples that is to be digested, every 20 samples.
- d. Sample is given a unique identifier in the digestion log.

3. Laboratory Control Samples

- a. For water samples, one LCS is digested with every batch of samples digested (20 sample maximum).
- b. For water samples, a LCS is digested every day for each type of digestion, every 20 samples.
- c. For soil/sediment samples, a soil matrix standard reference material (SRM) must be digested per batch (20 samples maximum) or alternatively a spiked teflon chip sample.
- d. Sample is given a unique identifier in the digestion log.

4. Duplicates

- a. A duplicate is prepared every 20 samples. This usually takes the form of a matrix spike duplicate.
NOTE: Certain projects require a sample duplicate and a matrix spike duplicate with each set of twenty samples.

5. Blank Spike

- a. This is required for certain projects.

B. Sample Matrix

NOTE: Field blanks/duplicates, trip blanks, or equipment blanks are not to be used for sample matrix QC samples.

1. Matrix spike

- a. Digest a spike and spike duplicate every 20 samples where sample volume is adequate to do so. Choose a sample (if possible) that has a lot of metals requested to be analyzed.

NOTE: For some projects, a sample duplicate and sample spike may be required instead of a spike and spike duplicate. Your supervisor should make you aware of these projects.

- b. The following metals do not get digested spikes when using CLP spike.

Calcium

Magnesium

Sodium

Potassium

- c. For TCLP samples, a spike **and a spike duplicate** must be digested for every matrix. You should inspect the sample (original sample prior to extraction) or check the log book to determine matrix type. (Also the matrix spike aliquots must be added to the extracts after filtration but before preservation.)

- d. **The CLH project requires that a high and a low spike be prepared and analyzed. Spikes should be prepared at 40 mg/Kg and 400 mg/Kg for soil samples and 200 ug/L and 2000 ug/L for aqueous samples.**

XII. CORRECTIVE ACTIONS

A. Sample boils during digestion.

1. Redigest another sample aliquot.

B. Sample goes dry or portion of beaker bottom is exposed due to excess evaporation during digestion.

1. Redigest another sample aliquot.
2. Glass beaker dry for an extended period of time? Discard beaker.

XIII. SPECIAL NOTES

A. **Never** take for granted how a sample should be digested. If the sample looks strange or unusual, or if you are not sure what metals the sample gets, what detection limits are required, whether the sample is total or dissolved, or even what method of digestion should be used, always ask your supervisor or the person who is to analyze the sample. How metals need to be digested changes too often to take it for granted.

B. **Antimony (Sb) soils** should be analyzed within 48 hours of digestion whenever possible. When a soil requesting Antimony analysis is received, you must coordinate with the person who will be analyzing it to be sure that they can analyze it on the same day that it is digested.

C. Labels for the digested sample must be written in a neat and legible manner. The labels must include such information as sample number, client name, the date digested, and the volume or mass digested.

D. There are several precautions that must be taken to minimize the possibility of contamination.

1. All metals glassware must be kept separate from all other laboratory glassware.

2. Metals glassware must be washed as soon as possible after being used. **Dirty metals beakers must not be left overnight.**
 3. Acid to be used for metals digestions must be kept separate from all other laboratory acid.
- E. Samples must be digested in a timely manner to ensure ICP analysis remains on schedule for data generation. Samples received on or before Wednesday of week X must be prepared for ICP digestion by the end of week X. Your supervisor must be consulted if this schedule can not be met at a particular time.
- F. Please consult Waste Disposal SOP-QS14, for information concerning disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

Addendum for USEPA CLPILM 05.2 AQUEOUS & SOIL/SEDIMENT

The following is a list of changes for sample preparation when the 5.2 statement of work is required:

1. Soluble samples are required to be digested unless the chain of custody specifically states that digestion is not required. An MDL study must be done on the unprepared MDL solution in order to provide MDL levels for samples that are not digested. When digestion is not required an LCSW and post digestion spike are not required.
2. Digestates must be stored until 365 days after delivery of a complete, reconciled data package.
3. Preparation codes are used on form 13's. They are found in the 5.2 statement of work page B-39 3.4.12.2.4.

DEFINITIONS – Refer to SOP-QS08 for common environmental laboratory definitions.

EMPIRICAL LABS, LLC.

Record of SOP Review and Implementation

Rev 22

TRAINING TOPIC SOP 100 - Metals Digestion/Preparation Methods 3005A, 3010A, 3020A, 3030, 3040A, 3050B USEPA CLPILMO 04.1

AQUEOUS & Soil/Sediment USEPA CLPILMO 05.2 Aqueous & Soil/Sediment, USEPA Method 200.7 (Standard Methods) 3030C

Group: Betty Deville

ATTENDEES:					
NAMES (print)	SIGNATURE	REMARK	DATE	TIME	INSTRUCTOR
1 Kendra Gentry	<i>Kendra Gentry</i>		1/17/11	14:54	BLD
2 Royer Buer	<i>Roy B</i>		1-17-2011	15:11	BLD
3 Fran Hula	<i>Fran Hula</i>		1/17/2011	15:17	KH
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**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

METALS: SOP 105

REVISION #: 17

EFFECTIVE DATE: 20110516

**METALS
BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION
SPECTROMETRY (ICP-AES) TECHNIQUE**

**SW846 6010B, SW846 6010C, EPA 200.7, SM 2340B (19th 20th and 21st Edition) for Hardness
Calculation, (USEPA CLP) ILMO 4.1 (NJDEP does not accept CLPILM 04.1 after June,
2003) and Addendum for USEPA CLPILM 05.2.**

APPROVALS:

Lab Director:  Date: 5/16/11

Data Quality Manager:  Date: 5/16/11

Section Supervisor: Betty Quillen Date: 5/16/11

Changes Summary

Revision 17, 20110516

- This is an update of SOP revision 16 dated 4/11/2010.
- Change all limit statements to include “after rounding to the nearest whole number”.
- Add procedure for recording digestates filtered prior to analysis within section 14.2.
- Training SOP reference updated to QS03 in section 14.6.
- References to DoD QSM 4.1 have been updated to DoD QSM 4.2.

Revision 16, 04/11/10

- The SOP is an update from Revision 15 dated 05/08/09
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory’s revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

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1. Identification of the Test Method

This SOP is compliant with methods – SW846 6010B, SW846 6010C, EPA 200.7, SM 2340B (19th 20th and 21st Edition) for Hardness Calculation, (USEPA CLP) ILMO 4.1 (NJDEP does not accept CLPILM 04.1 after June, 2003) and Addendum for USEPA CLPILM 05.2.

2. Applicable Matrix or Matrices

This SOP is applicable to all matrices, including ground water, aqueous samples, TCLP, SPLP and EP extracts, industrial and organic wastes, soils, sludge samples, sediments, and other solid wastes, require digestion prior to analysis.

3. Detection Limit: Detection limits are found in **Table 1** of this SOP. Sensitivity and optimum ranges of the metals may be found in the ICP method file.

4. Scope of Application, Including components to be Analyzed

Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Method Detection Limit and the Reporting Limit (also defined as the Limit of Quantitation).

5. Summary of the Test Method

- 5.1 Prior to analysis, samples must be solubilized or digested using appropriate. Sample Preparation Methods (e.g., Methods 3005-3050 and SOW ILM 04.1/05.2). When analyzing for dissolved constituents, acid digestion is not always necessary if the samples are filtered and acid preserved prior to analysis. If particulates form after filtration and preservation the sample must be digested prior to analysis.

NOTE: When selenium is required soluble samples must always be digested.

- 5.2 This method describes the simultaneous multi-elemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the large droplets are removed by a spray chamber and the small droplets then pass through to the plasma. The solvent is evaporated. The residual sample decomposed to atoms and ions that become excited and emit characteristic light which is measured, giving a measurement of the concentration of each element type in the original sample. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analytic wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. Control of the spectrometer is provided by PC based *iTEVA* software.

5.3 Inductively Coupled Argon Plasma (ICAP) primary advantage is that it allows simultaneous determination of any elements in a short time. The primary disadvantage of ICP is background radiation from other elements and the plasma gases. Although all ICP instruments utilize high-resolution optics and background correction to minimize these interferences, analysis for traces of metals in the presence of a large excess of a single metal is difficult. Examples would be traces of metals in an alloy or traces of metals in a limed (high calcium) waste. ICP and Flame AA have comparable detection limits (within a factor of 4) except that ICP exhibits greater sensitivity for refractories (Al, Ba, etc.). Furnace AA, in general, will exhibit lower detection limits than either ICP or FAA.

5.4 It is standard procedure to use an internal standard (scandium) with samples to increase the stability of the instrument as recommended by the manufacturer (Thermo Fisher). (When samples are suspected of containing scandium, internal standard cannot be used.)

6. Definitions

Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.

Additional definitions specific to this SOP are listed below:

- 6.1 **ICP or ICAP**- Inductively Coupled Plasma or Inductively Coupled Argon Plasma.
- 6.2 **Inter-element correction (IEC)**- Defined as a correction factor applied by the instrument when there is an overlap of the spectrum from the plasma gases or from another metal into the spectrum of another metal causing that metals concentration to either be inflated or deflated.

7. Interferences

7.1. Spectral interferences are caused by background contribution from continuum or recombination phenomena, stray light from the line emission of high-concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

7.1.1. Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same

change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans should be included in the correction algorithm. Off-line interferences are handled by including spectra on interfering species in the algorithm.

- 7.1.2. To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line inter-element spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a 200 mg/L or 500 mg/L concentration near the upper analytical range limit.
- 7.1.3. Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for inter-element contributions. Instruments that use equations for inter-element correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply inter-element correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelength are listed in the method in table 2. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.
- 7.1.4. When using inter-element correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that Arsenic is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Aluminum. According to Table 2 from the method, 100 mg/L of Aluminum would yield a false signal for Arsenic equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Aluminum would result in a false signal for Arsenic equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interferences than that shown in Table 2 from the method. The interference effects must be evaluated for each individual instrument since the intensities will vary.

- 7.1.5. Inter-element corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Inter-element corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Inter-element corrections that constitute a major portion of an emission signal may not yield accurate data. Users should not forget that some samples may contain uncommon elements that could contribute spectral interferences.
- 7.1.6. The interference effects must be evaluated for each individual instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences as well as any other suspected interferences that may be specific to the instrument or matrix. The instrument utilizes a computer routine for automatic correction on all analyses.
- 7.1.7. If the correction routine is operating properly, the determined, apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.
- 7.1.8. When inter-element corrections are applied, their accuracy should be verified, daily, by analyzing spectral interference check solutions (IFA/IFB). If the correction factors or multivariate correction matrices tested on a daily basis are found to be within 20% criteria for 5 consecutive days, the required verification frequency of those factors in compliance may be extended to a weekly basis. Also, if the nature of the samples analyzed is such they do not contain concentrations of the interfering elements at \pm one reporting limit from zero, daily verification is not required. All inter-element spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation-change, such as in the torch, nebulizer, injector, or plasma conditions occurs. Standard solution should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

7.2. Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If

physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump, by using an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers.

- 7.3. Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build-up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the elements and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized.
- 7.4. Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. When the instrument displays negative values, dilution of the samples may be necessary.

8. Safety

Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed lab-wide.

- 8.1 Normal accepted laboratory safety practices should be followed while performing this analysis.
 - 8.1.1. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of appropriate safety gloves and lab coats is highly recommended.
 - 8.1.2 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
 - 8.1.3MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves in the Quality Assurance Officers office.

9. Equipment & Supplies

- 9.1. Inductively coupled argon plasma emission spectrometer: Thermo Scientific 6500 DUO.
- 9.2. Computer-controlled emission spectrometer with background correction: Thermo Scientific 6500 DUO or equivalent.
- 9.3. Radio frequency generator compliant with FCC regulations: Thermo Fisher or equivalent.
- 9.4. Auto-sampler: Thermo Fisher or equivalent.

- 9.5. Printer capable of printing results every 4 minutes.
- 9.6. Cooling Water recycler.
- 9.7. Iteva software.
- 9.8. Argon gas supply – Liquid Argon
- 9.9. Class A volumetric flasks
- 9.10. Analytical balance - capable of accurate measurement to a minimum of three significant figures (0.001gm).
- 9.11. Variable Eppendorf Pipettes 1000 μ L; 5000 μ L
- 9.12. Disposable beakers 10, 20 and 50 mL size.
- 9.13. Hood system capable of venting the heat from the system off of the instrument during analysis.

10. Reagents and Standards

The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

- 10.1. Reagent Water. All references to water in the method refer to reagent grade water unless otherwise specified. Reagent water will be interference free.
- 10.2. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.
- 10.3. Hydrochloric acid (concentrated), HCl. A method blank is digested and analyzed before a new lot number of HCl is put into use, to ascertain purity. The lot # is logged into Element and the data kept on file.
- 10.4. Nitric acid (concentrated), HNO₃. A method blank is digested and analyzed before a new lot number of HNO₃ is put into use, to ascertain purity. The lot # is logged into Element and the data kept on file.
- 10.5. Calibration standards

- 10.5.1. All standards have an acid matrix of 2% HNO₃ and 5% HCl and should be prepared using class A volumetric flasks and calibrated Eppendorfs).
- 10.5.2. CAL1 is the calibration blank: Reagent grade water matrix matched as in
Note: when this standard is analyzed the intensities should be compared to a previous run to make sure that no contamination has occurred. Prepare this solution fresh daily.
- 10.5.3. Stock QC21 solution: (100 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals - Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, Se, Sr, Tl, Ti, V, and Zn.
- 10.5.4. Stock QC7 solution: Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals- (50 ug/mL)- silver; (100 ug/mL)- aluminum, boron, barium and sodium; (1000 ug/mL)- potassium; (500 ug/mL or 100 ug/mL note we use two sources of this standard and each have different concentrations for Si) –Silica.
- 10.5.5. Boron solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.6. Stock Tin solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.7. Stock Silver solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.8. Stock Aluminum solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.9. Stock Calcium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier. Note: Two sources are needed.
- 10.5.10. Stock Magnesium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.11. Stock Iron solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

- 10.5.12. Stock Potassium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.13. Stock Barium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.14. Stock Sodium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.15. Stock Arsenic solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.16. Stock Cobalt solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.17. Stock Chromium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.18. Stock Copper solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.19. Stock Manganese solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.20. Stock Nickel solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.21. Stock Lead solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.22. Stock Selenium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.23. Stock Thallium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.24. Stock Beryllium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.25. Stock Cadmium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.26. Stock Antimony solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

- 10.5.27. Stock Molybdenum solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.28. Stock Strontium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.29. Stock Titanium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.30. Stock Vanadium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.31. Stock Zinc solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.32. Stock Scandium solution (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.6. Calibration and Calibration Verification standards

- 10.6.1. The calibration standards and calibration verification standards preparations are recorded in Element. Please find method of preparation in Appendix I.
- 10.6.2. The CRL solution is analyzed to check the accuracy of the instrument at the reporting limit. The stock standard solutions A and B are prepared from single element standards listed in 10.5 above. Please find method of preparation in Appendix I. This solution is stable for 6 months. The working solutions are made up as needed or every 3 months as follows: Prepared by adding 1.0 ml of RL Stock solution A and 1.0 ml of RL Stock Solution B to de-ionized water with 2% HNO₃ and 5% HCL matrix and diluting to 100 mLs , mix well. This solution is stable for 3 months.
- 10.6.3. The interference check standard solutions (IFA and IFB) are prepared to provide an adequate test of the IECs. A purchased solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe is diluted 10x to prepare the IFA. The IFB is prepared by diluting 100x a purchased solution containing 10 ug/mL of As and Tl; 20 ug/mL Ag; 50 ug/mL Ba, Be, Cr, Co, Cu, Mn, and V; 100 ug/mL Cd, Ni and Zn; 5 ug/mL Pb and Se; and 60 ug/L Sb. Add to this a purchased solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe diluted 10x. These solutions are prepared as needed or monthly and assigned an Element # for traceability.

10.7 Digestion standards

10.7.1 The Blank Spike (BS) is prepared from High Purity solutions CLP-CAL-1 solution A and B; CLP-CAL-2 and CLP-CAL-3. 0.50 mL of CLP-CAL-1 A and B; and 0.50 mLs of the 1000 ug/mL single element standards for Molybdenum, Boron, Titanium and Strontium is diluted to 500 mL with 0.125 mL of CLP-CAL-2 and CLP-CAL-3 and 0.050 mLs of 10000 ug/mL Tin. 25 mL of HCl and 10 mL of HNO₃ are added for preservation. This solution is stored in a Teflon bottle. A portion is reserved in case of a problem with digestion. When there is a problem with the analysis of the BS the solution is checked first before action is taken to make sure that it was made properly and has not deteriorated since it was made up. This solution is given a unique identifier within Element. The BS is prepared from a source independent from that used in the calibration standards. This solution is prepared daily or as needed. 50 mLs of this solution is used for digestion for normal level water samples and the sample is brought back to 50 mLs after digestion. Low level water samples start with two 50 mLs vials with only 1.0 mL of the stock blank spike solution in each taken to 50 mLs. The samples are cooked down to below 25 mLs and combined and then cooked down to below 25 mLs again and then brought back to 25 mLs. This low level BS is given a unique identifier in Element.

10.7.2. The solid BS used with soil samples is prepared by weighing up 1.0 gram of Teflon chips for regular level and 2.0 grams of Teflon chips for low level and spiking using the same spiking solutions used to spike the sample matrix. This standard is given a unique identifier i.e. Batch #-BS1. Note: Amount of spiking solution used varies according to whether the samples are being digested for normal level or low level soils. See spiking solutions in 10.7.3.1 for how to prepare the BS for a solid sample, it is prepared the same way that a soil spike is prepared only the known amounts of metals are added to laboratory water.

10.7.3. The spiking solutions are prepared as follows:

10.7.3.1. Stock Multi-element Spiking Solutions: High Purity CLP-CAL-1 solution A: 2000 ug/mL Al and Ba; 50 ug/mL Be; 200 ug/mL Cr; 500 ug/mL Co, Mn, Ni, V and Zn; 250 ug/mL Cu; 1000 ug/mL Fe; 5000 ug/mL Ca, Mg, K and Na; solution B: 250 ug/mL Ag; CLP-CAL-2: 1000 ug/L Sb; CLP-CAL-3: 1000 ug/mL As, Pb, Se, Tl; 500 ug/mL Cd. Order from the manufacturer already prepared. These solutions are given a unique identifier within Element. Add 0.050 mL for water samples and 0.20 mL for normal level soil samples and 0.10 for low level soil samples of CLP-CAL-1 solutions A and B, and 0.0125 mL for water samples and 0.05 mLs for normal level soil samples and 0.025 mLs for low level soil samples of CLP-CAL-2 and 3 to 50 mL of sample for water samples and 1 gram of sample for normal level soils and 2 grams of sample for low level soils for the following spike values: 2000 ug/L Al and Ba; 50 ug/L Be; 200 ug/L Cr; 500 ug/L Co, Mn, Ni, V and Zn; 250 ug/L Cu; 1000 ug/L Fe; 5.0 mg/L Ca, Mg, K and Na, 250 ug/L Ag, Sb, As, Pb, Se and Tl; 125 ug/L Cd. A blank

spike should be prepared at the time the samples are spiked to check the actual spike value and accuracy.

10.7.3.2. TCLP Spiking Solution: Use 0.50 mL diluted to 50 mL for digestion:

2.5 mL 10000 mg/L Ba stock standard diluted to 100 mL; 2.5 mL Cr, Pb and As 1000 mg/L stock standard diluted to 100 mL; 0.50 mL Cd and Se diluted to 100 mL. Store in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 2500 ug/L Ba; 250 ug/L Cr, Pb and As; and 50 ug/L of Cd and Se. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed.

10.7.3.3. TCLP Silver Spiking Solution: Use 5.0 mL diluted to 50 mL for digestion:

0.40 mL of 1000 mg/L stock Ag solution diluted to 200 mL. Store this solution in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 200 ug/L. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed. Also this solution is not very stable and may require fresh preparation at least weekly.

11. Sample Collection, Preservation, Shipment, and Storage

Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.1. Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples which have been pre-filtered and acidified will not need acid digestion as long as the samples and standards are matrix matched and particulates do not form after the filtration and preservation take place. Solubilization and digestion procedures are presented in Sample Preparation Methods (Methods 3005A-3050A).

11.2. Sample digestates are stored at room temperature for at least 2 months unless a longer time is requested by the client. The samples contain an acid matrix of 3:1. All metal samples are neutralized before disposal in the receiving section of the laboratory.

11.3. **The appropriate SOPs should be consulted regarding sample preparation.** The following is a brief summary of the methods we use for metals preparation.

11.3.1. Method 3005A prepares groundwater and surface water samples for total recoverable and dissolved metals determination by ICP. The unfiltered or filtered sample is heated with dilute HCl and HNO₃ prior to metal determination.

11.3.2. Method 3010A prepares waste samples for total metal determination by ICP. The samples are vigorously digested with a mixture of nitric acid and hydrochloric acid followed by dilution with laboratory water. The method is applicable to aqueous samples, TCLP and mobility-procedure extracts.

11.3.3. Standard Methods Method 3030C prepares ground-waters and surface water samples for acid extractable metals: (lead and chromium.) This preparation has a holding time of 72 hours. The samples are preserved at collection with 5mL/L of HNO₃, in the laboratory 5 mL/100mL of 1+1 HCl is added and the sample is heated for 15 minutes in a block digester. The sample is filtered through a membrane filter and the filtrate is carefully transferred to a volumetric flask and brought back to 100 mLs.

11.3.4. Method 3050B prepares wastes samples for total metals determination by ICP. The samples are vigorously digested in nitric acid and hydrogen peroxide followed by dilution with either laboratory water or hydrochloric acid and laboratory water. The method is applicable to soils, sludges, and solid waste samples.

12. Quality Control

Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.

12.1. Daily run and batch QC

12.1.1. Calibration is required daily. Either a blank and a high standard or a client specific three standard concentration points and a blank calibration is required daily.

12.1.2. IEC correction standards for aluminum and iron are required daily.

12.1.3. ICV within $\pm 5\%$ for 200.7 and within $\pm 10\%$ for all other methods.

12.1.4. ICB/CCB less than two times \pm MDL or less than \pm LOD for DOD. The ICB/CCB must immediately follow the ICV/CCV.

12.1.5. RL standard run against the curve within $\pm 20\%$ initially and client specific requirement of $\pm 30\%$ at the end of the analysis.

12.1.6. IFA/IFB analyzed daily. IFA must be less than two times \pm MDL or less than \pm LOD unless verified standard contamination for DOD. The IFB must recover within $\pm 20\%$ for all analytes in the IFB standard solution. If the IFA/IFB solution is not within the required limits- if possible reanalyze all associated samples, if not possible to reanalyze all associated samples must be flagged with an “Q” on the final report for DOD.

12.1.7. CCV must be analyzed every ten samples or at the end of the analysis within $\pm 10\%$ or the samples are reanalyzed if possible. If samples cannot be reanalyzed, all samples are flagged with a "Q" for DOD.

12.1.8. CCB must be analyzed every ten samples immediately following the CCV or at the end of the analysis less than two times $\pm \text{MDL}$ or $\leq \text{LOD}$ for DOD. If the CCB is out of the allowable range the samples are flagged with "B".

12.1.9. *The following should be analyzed with each preparation batch containing a matrix spike.*

- Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 50 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution (volumetric glassware must be used) should agree within $\pm 10\%$ of the original determination. If not, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.
- Post digestion spike addition: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75% to 125% of the known value for SW6010B and 80 to 120% for SW6010C and is required especially if the pre-digestion matrix spike is outside of control limits. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected. Run all associated samples in the preparatory batch by method of standard additions (MSA) or apply "J" flag. The analyst and or section manager must note this situation on the final analytical report. Apply "J" flag if the post spike is outside the range of 75% to 125% for 6010B or 80% to 120% for 6010C.

12.2 Quarterly and/or every six months

12.2.1. Linear range standards must be analyzed at a frequency no less than once every six months. The linear range standard is required for verification that samples are actually linear to the degree claimed. The analyst is responsible for completing this task in a timely manner. The linear range standard must be within $\pm 10\%$ of true value. This standard can be analyzed as the linear dynamic range.

12.2.2. The inter-element correction factors (IEC) should be verified at the time the linear range standards are analyzed or whenever there is any question about whether an IEC is correcting correctly.

12.2.3. IDL's, linear range and IEC checks must be performed quarterly if straight CLP work is required.

12.3. Digested Batch QC

- 12.3.1. All quality control data should be maintained and available for easy reference or inspection.
- 12.3.2. Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank (BLK), sometimes referred to as the preparation blank is a volume of reagent water acidified with the same amounts of acids as were the standards and samples. These blanks are taken through the same digestion/preparation steps as the sample being tested. The result for the method blank should not indicate contamination greater than $\pm \frac{1}{2}$ RL for DOD or \pm RL/CRDL for other or CLP. If exceeded, the impact upon the data should be evaluated and the associated sample(s) should be either re-digested or the data should be qualified. The extracted blank associated with TCLP batches must be less than 100 X the regulatory limit for barium.
- 12.3.3. Employ a minimum of one blank spike (BS) for aqueous samples or one Teflon chip spiked sample per sample batch to verify the digestion procedure. These blank spikes are taken through the same digestion/preparation steps as the sample being tested. The control limits are $\pm 15\%$ method 200.7 - aqueous and soil samples or $\pm 20\%$ for all other methods aqueous and soil samples. If the BS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be re-digested. Consult your supervisor for further action. Qualifying the associated data may not be permissible for some clients.

12.4. Sample

- 12.4.1. Analyze one replicate sample for every twenty samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample replicate. Project specific requirements will take precedence in these situations. NJDEP demands that this requirement be met with a client specific duplicate rather than a spike duplicate. The control limits are less than or equal to 20% RPD (if both are $>5x$ RL) or \pm the RL (if either are $<5x$ RL). Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation. Apply "J" flag for DOD if acceptance criteria are not met. Apply "*" flag for CLP and other work if acceptance criteria are not met.
- 12.4.2. Analyze a minimum of one spiked sample and/or spiked sample duplicate for every twenty samples or per analytical batch, whichever is more frequent. Project specific requirements will take precedence in determining whether a matrix spike duplicate is employed in these situations. If the analyte level in the sample is not greater than 4X the spiking level, the spike recoveries should be within $\pm 20\%$ of the true value. If not, and sufficient sample volume exist, a post digestion spike should be analyzed. Apply "J" flag for DOD if acceptance

criteria are not met. Apply “N” flag or CLP and other work if acceptance criteria are not met.

13. Calibration and Standardization

Quality Systems **SOP QS08** “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

- 13.1. Set up the instrument with proper operating parameters. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).
- 13.2. Operating conditions - **The instrument settings can be found in method file within the iTEVA software.** For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on that particular instrument. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.
- 13.3. Auto-peak when some change has been made to the introductory system and calibrate the instrument according to the instrument manufacturers recommended procedures, using the specified calibration standard solutions. Flush the system with 2% HNO₃ / 5% HCl between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve consists of a blank and three standards ($r \geq 0.998$). If a three point calibration curve is not required for the client samples being analyzed by Empirical Laboratories may use a blank and one standard as referenced in USEPA - CLP protocols.
- 13.4. Before beginning the sample run, analyze single element Iron and Aluminum standards at their linear range to check for IEC drifts. Analyze these standards first as QC samples with an IEC check table and action taken should be to calculate IECs using the iTEVA software. Make sure to rinse thoroughly after running these linear range standards, they can cause carry over into the initial QC samples which are analyzed next. The analysis order follows as: ICV ($\pm 10\%$) for 200.7 ($\pm 5\%$) and ICB ($< \pm 2 \times \text{MDL}$, $< \pm \text{LOD-DOD}$ or $\pm \text{RL/CRDL}$ for others or CLP, first, then analyze a reporting limit standard (a standard at the concentration of the reporting limit). This standard should be within $\pm 20\%$ for DOD projects and $\pm 30\%$ for samples analyzed for 6010C. Then reanalyze the highest mixed calibration standard(s) as if it were a sample. Concentration values obtained should not deviate from the actual values by more than 5%. If they do, follow the recommendations of the instrument manufacturer to correct for this condition. Note: Supervisor must be notified if the control limit is

not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.

- 13.5. For **CLP projects**, verify the validity of the curve in the region of 2x the contract required detection limit (CRDL) before and after each batch of 20 samples in the specific order of CRI, ICSA, ICSAB, CCV and CCB (CCB criteria: $< \pm\text{MDL}$ or $\pm\text{RL}/\text{CRDL}$ for others or CLP, or twice during every 8-hour work shift, whichever is more frequent. Results should be within $\pm 20\%$. Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation. (For Internal QC)
- 13.6. Verify the inter-element and background correction factors at the beginning of the sequence in the specific order of IFA, IFB, CCV and CCB (IFA criteria: non-spiked analytes $< \pm 2\text{xMDL}$ or $< \text{LOD}$ for DOD beginning of sequence. Do this by analyzing the interference check solution IFA and IFB. Absolute value of concentration for all non-spiked analytes in the IFA must be $< \text{LOD}$ (unless they are verified trace impurity from one of the spiked analytes) for DOD. Results must be within $\pm 20\%$ of the true value for IFB. If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS. (CRI, ICSA and ICSAB required at the end for CLP projects only).

Note: Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.

13.7. The instrument must be calibrated once every 24 hours.

13.8. Instrument Autosampler Report example:

Calibration Rack (used by instrument software to insert QC)

- 1) Cal Std 1 (blank)
- 2) Cal Std 2 (Low Cal)
- 3) Cal Std 3 (Mid Cal)
- 4) Cal Std 4 (Ba @ 5000 ppb)
- 5) Cal Std 5 (QC5)
- 6) Cal Std 6 (QC 21)
- 7) Cal Std 7 (NAK 100)
- 8) Cal Std 8 (QC3)
- 9) Cal Std 9 (Ag)
- 10) Al IEC-(correction using ITEVA software)
- 11) Fe IEC-(correction using ITEVA software)

Sample Sequence RACK 1

- 1) SEQ-ICV

- 2) SEQ-ICB
- 3) SEQ-CRL1-reporting limit standard 1
- 4) SEQ-CRL2-reporting limit standard 2
- 5) Ba@ 5000 ppb (readback)
- 6) QC5
- 7) NAK High-(readback)
- 8) QC 21 High-(readback)
- 9) Salt Cal at 500 ppm (readback)
- 10) Rinse
- 11) SEQ-IFA1
- 12) SEQ-IFB1
- 13) Rinse
- 14) SEQ-CCV
- 15) SEQ-CCB
- 16) Method Blank (*Batch # -BLK1*)
- 17) Blank Spike (*Batch # -BS1*)
- 18) Sample 1
- 19) Sample 2
- 20) Sample 3
- 21) Sample 4
- 22) Sample 5
- 23) Sample 6
- 24) Sample 7
- 25) Sample 8
- 26) Sample 9
- 27) Sample 10
- 28) SEQ-CCV
- 29) SEQ-CCB
- 30) Sample 11
- 31) Sample 12
- 32) Sample 13
- 33) Sample 14
- 34) Sample 15
- 35) Sample 16
- 36) Sample 17
- 37) Sample 18
- 38) Sample 19
- 39) Sample 20
- 40) Sample matrix spike (*batch#- MS1*)
- 41) Sample matrix spike duplicate (*batch# -MSD1*)
- 42) Sample post digestion spike (*batch# -PS1*)
- 43) Sample serial dilution (*batch# -DUP1*)
- 44) SEQ-CCV
- 45) SEQ-CCB
- 46) Preparation Blank (*batch# -BLK1*)
- 47) Blank Spike (*batch# -BS1*)
- 48) Sample 1
- 49) Sample 2

- 50) Sample 3
- 51) Sample 4
- 52) Sample 5
- 53) Sample 6
- 54) Sample 7
- 55) Sample 8
- 56) Sample 9
- 57) Sample 10
- 58) SEQ-CCV
- 59) SEQ-CCB
- 60) Sample 11

RACK 2

- 1) Sample 12
- 2) Sample 13
- Etcetera...

Each rack holds 60 samples and there are 4 racks that are used for samples, CCVs and CCBs and run QC.

14. Procedure

- 14.1. Once the instrument has been calibrated, begin the analysis of samples.
- 14.2. If particulates are visible in the digestate, the sample must be filtered prior to analysis. If filtration is required, a filter blank must be prepared by filtering reagent grade water which has been properly acidified. The sample requiring filtration must be recorded on the bench sheet and added to the bench sheet comments in the LIMS. **In the event USACE samples are filtered, all USACE samples and the QC samples in that QC batch must be filtered. All USACE solid samples and their associated batch QC samples must be filtered prior to analysis.**
- 14.3. Flush the system with 2% HNO₃ / 5% HCl for at least 1 minute before the analysis of each sample.
- 14.4. Dilute and reanalyze samples that are more concentrated than the linear calibration limit or, for 200.7, $\pm 10\%$ of the linear range standard. **In the case of USACE samples, the criterion changes and requires dilution and reanalysis of all samples which produce a concentration that exceeds the highest calibration standard. Sample results detected between the MDL and LOQ are flagged as estimated with a "J" flag.**
- 14.5. Verify calibration every 10 samples or every 2 hours, whichever is more frequent and at the end of the analytical run, using a continuing calibration verification (CCV) sample and a continuing calibration blank (CCB) sample.

- 14.5.1. The results of the CCV are to agree within $\pm 10\%$ for 6010 (5% for 200.7) on initial verification of the expected value, with relative standard deviation (RSD) $< 5\%$ from 3 replicates (minimum of three integrations). If not, terminate the analysis, correct the problem, and reanalyze the previous ten samples. The analyst may continue the analytical run, and after conferring with the section manager it may be necessary to reanalyze a group of samples. The analyst must notify the section manager within 24 hours.
- 14.5.2. The results of the calibration blank (this is not the method/preparation blank) are to be $< 2x \pm MDL$, for CLP $< RL$, for **DOD no analytes detected** $> \pm LOD$. If the calibration blank is not in control, evaluate the impact upon the previous 10 samples. Reanalysis may be required after an evaluation of the data. If the blank $< 1/10$ the concentration of the action level of interest and no sample is within 10% of the action limit, samples need not be reanalyzed. One must also evaluate the reporting limit (RL) as it relates to 3X the IDL/MDL. If the RL is significantly above 3X IDL or MDL then reanalysis may not be required (Na, K, Mg and Ca are good examples of this situation).
- 14.6. Demonstration of Capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP QS03 for guidance.

15. Data Analysis and Calculations

Quality Systems SOP QS09 “General and commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.

- 15.1. Total hardness is reported from HNO_3 preserved sample. The final concentration is calculated from the calcium and magnesium results as follows: $Ca \text{ mg/L} \times 2.5 + Mg \text{ mg/L} \times 4.1 = \text{total Hardness in mg/L as } CaCO_3$.
- 15.2. The instrument will generate data results in mg/L or $\mu\text{g/L}$ (labeled appropriately). Each result represents an average of three individual readings per metal channel.
- 15.3. For aqueous samples, if a post/pre-digestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution.
- 15.4. For solid samples, if a post-digestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution. Also, the result must be converted to reporting units which are usually mg/kg.

$$SR \text{ (}\mu\text{g/g or mg/kg)} = IR * DF * FED / SM$$

- SR = Sample result
 IR = Instrument result ($\mu\text{g/L}$)
 DF = Dilution factor (post digestion)

FED = Final volume of digestate (L)
SM = Sample mass digested (g)

16. Method Performance

Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality Manual is completed by each analyst and then provided to the supervisor for further processing and approval.

DOC LCS Preparation: See BS preparation under 10.7.1 through 10.7.3 above.

DOC Accuracy and Precision Criteria: The LOD is analyzed at 2 times the MDL and must result in a concentration 3 times the noise. The LOQ is analyzed at the RL or 2 times the RL and must be recovered within $\pm 50\%$.

17. Pollution Prevention:

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18. Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. Table 2 of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. Table 2 within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

CORRECTIVE ACTIONS

19.1. INSTRUMENT RELATED

- 19.1.1. ICV not within $\pm 10\%$ or $\pm 5\%$ for 200.7
 - a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.
 - b. Is the problem with the calibration?
 - i. Recalibrate through analysis of appropriate standards and recheck ICV.

19.1.2. ICB not \pm MDL or within \pm 3X IDL or CRDL for CLP, **DOD no analytes detected >LOD**

- a. Is the problem with the solution?
 - i. Re-prepare
- b. Is the problem with the calibration?
 - i. Recalibrate with the blank solution or the low level standard. Restart analysis with the ICV.

19.1.3. Check standards not within \pm 5%

- a. Is the problem with the solution?
 - i. Re-pour, re-prepare or obtain new stock.
- b. Is the problem with the calibration?
 - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.4. CLP only-CRI not within \pm 20% (Internal QC, only required for CLP work).

- a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.
- b. Is the problem with the calibration?
 - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.5. IFA metals not present are not less than the detection limit for that metal, **for IFA DOD, absolute value of concentration for all non-spiked analytes $<\pm$ LOD.**

- a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.
- b. Is the problem with the calibration?
 - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.6. IFB not within \pm 20%

- a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.
- b. Is the problem with the calibration?
 - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.7. CCV not within \pm 10%

- a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.
- b. Is the problem with the calibration?
 - i. If appropriate, continue the analysis. Discuss effect of the out of control situation with your supervisor. The samples will be reanalyzed or the data will be qualified.

19.1.8. CCB not \pm 2xMDL or CRDL for CLP, DOD no analytes detected $>\pm$ LOD.

- a. Is the problem with the solution?

- i. Re-prepare
- b. Is the problem with the calibration?
 - i. Re-calibrate and reanalyze.

19.2. DIGESTION RELATED

- 19.2.1. Preparation blank (BLK) not within $\pm \frac{1}{2}$ RL and \pm RL for common contaminants DOD or RL/CRDL for other or CLP
 - a. Is the problem with the instrument?
 - i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
 - b. Is the problem with the digestion?
 - i. If associated samples are less than 10X the level of the preparation blank but above the RL, the sample must be re-digested or the data must be qualified on the final report.
- 19.2.2. BS not within control limits
 - a. Is the problem with the instrument?
 - i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
 - b. Is the problem with the digestion?
 - i. If biased low, associated samples must be re-digested.
 - ii. If biased high, the impact upon the data user must be evaluated. The samples will be re-digested or the data will be qualified on the final report.

19.3. SAMPLE MATRIX RELATED

- 19.3.1. Replicate analysis RPD not within $\pm 20\%$ (if both are $>5X$ CRDL) or \pm the CRDL (if either are $<5X$ CRDL).
 - a. The associated sample data must be qualified on the final report.
- 19.3.2. Spike analysis recovery not within $\pm 20\%$.
 - a. Is the analyte level in the sample greater than 4X the spiking level?
 - i. If yes, the spike recovery is not evaluated.
 - ii. If no, a post digestion spike must be analyzed and the associated sample data must be qualified on the final report.
- 19.3.3. When required, post digestion spike analysis recovery not within $\pm 25\%$ for SW6010B, DOD or $\pm 20\%$ SW6010C.
 - a. The associated sample data must be qualified on the final report.
 - b. For USACE analysis by MSA is required.
- 19.3.4. Serial dilution analysis percent difference not within $\pm 10\%$
 - a. Is the analyte concentration a factor of 50 above the instrumental detection limit after dilution?
 - i. If no, the serial dilution data can not be evaluated.

- ii. If yes, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.

20. Waste Management

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21. References

21.1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 6010B and Method 6010C.*

21.2. *USEPA Code of Federal Regulations, 40, CH 1, PT 136; Method 200.7; APX-B.*

21.3. *USEPA Contract Laboratory Program (CLP) for Inorganics ILM04.1; ILM05.2*

21.4. DOD Quality Systems Manual for Environmental Laboratories Version 4.2. (Based on NELAC Voted Revision June 5, 2003. 10/25/2010)

22. Tables, Diagrams, Flowcharts and Validation Data

Table 1 contains all applicable parameters with the applicable RL/LOQ, LOD and Detection Limit.

Table 1A, contains a list of the wavelengths used for each analyte.

Table 2, for all technical methods, contains the QA/QC summary table.

Table 3, Technical Completeness / Accuracy Checklist

Table 4, Data Reviewers Checklist

Table 1 Water				
Analyte	MDL	LOD	MRL	Units
Aluminum	50.0	100	200	ug/L
Antimony	5.00	8.00	15.0	ug/L
Arsenic	3.00	6.00	10.0	ug/L
Barium	5.00	10.0	40.0	ug/L
Beryllium	1.00	2.00	5.00	ug/L
Boron	10.0	20.0	30.0	ug/L
Cadmium	1.00	2.00	5.00	ug/L
Calcium	1000	2000	5000	ug/L
Chromium	2.00	4.00	10.0	ug/L
Cobalt	5.00	10.0	12.5	ug/L
Copper	4.00	8.00	10.0	ug/L
Iron	30.0	60.0	100	ug/L
Lead	1.50	3.00	3.00	ug/L
Magnesium	1000	3000	5000	ug/L
Manganese	3.00	6.00	15.0	ug/L
Molybdenum	5.00	10.0	15.0	ug/L
Nickel	3.00	6.00	10.0	ug/L
Potassium	1000	3000	5000	ug/L
Selenium	3.00	5.00	6.00	ug/L
Silver	1.00	2.00	10.0	ug/L
Sodium	1000	3000	5000	ug/L
Thallium	3.00	4.00	8.00	ug/L
Tin	10.0	20.0	30.0	ug/L
Titanium	5.00	10.0	15.0	ug/L
Vanadium	5.00	10.0	12.5	ug/L
Zinc	5.00	10.0	20.0	ug/L

Table 1 TCLP				
Analyte	MDL	LOD	MRL	Units
Antimony	0.00500	0.00800	0.0150	mg/L
Arsenic	0.00300	0.00600	0.0100	mg/L
Barium	0.00500	0.0100	0.0400	mg/L
Cadmium	0.00100	0.00200	0.00500	mg/L
Chromium	0.00200	0.00400	0.0100	mg/L
Copper	0.00400	0.00800	0.0100	mg/L
Lead	0.00150	0.00300	0.00300	mg/L
Selenium	0.00300	0.00500	0.00600	mg/L
Silver	0.00100	0.00200	0.0100	mg/L

Table 1 Soil				
Analyte	MDL	LOD	MRL	Units
Aluminum	10.0	20.0	40.0	mg/Kg
Antimony	1.00	1.60	3.00	mg/Kg
Arsenic	0.600	1.20	2.00	mg/Kg
Barium	1.00	2.00	8.00	mg/Kg
Beryllium	0.200	0.400	1.00	mg/Kg
Boron	2.00	4.00	6.00	mg/Kg
Cadmium	0.200	0.400	1.00	mg/Kg
Calcium	200	400	1000	mg/Kg
Chromium	0.400	0.800	2.00	mg/Kg
Cobalt	1.00	2.00	2.50	mg/Kg
Copper	0.800	1.60	2.00	mg/Kg
Iron	6.00	12.0	20.0	mg/Kg
Lead	0.300	0.600	0.600	mg/Kg
Magnesium	200	600	1000	mg/Kg
Manganese	0.600	1.20	3.00	mg/Kg
Molybdenum	1.00	2.00	3.00	mg/Kg
Nickel	0.600	1.20	2.00	mg/Kg
Potassium	200	600	1000	mg/Kg
Selenium	0.600	1.00	1.20	mg/Kg
Silver	0.200	0.400	2.00	mg/Kg
Sodium	200	600	1000	mg/Kg
Thallium	0.600	0.800	1.60	mg/Kg
Tin	2.00	4.00	6.00	mg/Kg
Titanium	1.00	2.00	3.00	mg/Kg
Vanadium	1.00	2.00	2.50	mg/Kg
Zinc	1.00	2.00	4.00	mg/Kg

TABLE 1A

METAL	WAVELENGTH
Aluminum	396.1
Antimony	206.8
Arsenic	189.0
Barium	233.5
Beryllium	313.0
Boron	249.7
Cadmium	228.8
Calcium	317.9
Chromium	267.7
Cobalt	228.6
Copper	324.7
Iron	261.1
Lead	220.3
Magnesium	279.0
Manganese	257.6
Molybdenum	202.0
Nickel	231.6
Potassium	766.4
Selenium	196.0
Silver	328.0
Sodium	589.5
Strontium	421.5
Thallium	190.8
Tin	189.9
Titanium	334.9
Vanadium	292.4
Zinc	206.2

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Interference Check	<ul style="list-style-type: none"> once per calibration 	<ul style="list-style-type: none"> IFA less than LOD if not verified contamination of standard. IFB must be within $\pm 20\%$. 	<ul style="list-style-type: none"> Check IEC corrections for metals in the IFA.
Calibration Curve	<ul style="list-style-type: none"> Prior to analyzing any samples A minimum of a blank and 3-points for linear fits client specific requirement or a blank and high standard. Low standard at the RL level run against the curve within 20% initially and within 30% for subsequent analysis (6010C). 	<ul style="list-style-type: none"> Linear calibration Corr. of 0.998 Must follow curve processing requirements from SOP QS08 	<ul style="list-style-type: none"> Re-evaluate curve mix and makeup Re-run curve Check instrument for maintenance needs Re-prepare the curve standards <p>Samples cannot be analyzed until there is a passing calibration</p>
ICB	At the beginning of every sequence	Must meet the $\leq \pm \text{LOD}$ for DOD or $< 2 \times \text{MDL}$	Re-run
ICV	Alternate source standard to be analyzed after every calibration curve	<ul style="list-style-type: none"> Must be in the range 90 to 110% for 6010B&C, or 95 to 115% for 200.7. 	<ul style="list-style-type: none"> Re-analyze an ICV from a different source Re-prepare and re-analyze the ICV Re-calibrate and verify standard preps and sources
CCV	<ul style="list-style-type: none"> At the beginning of every sequence For every 10-client samples 	<ul style="list-style-type: none"> Must be in the range 90 to 110% 	<ul style="list-style-type: none"> Samples must be reanalyzed if possible, if not samples are flagged with a "Q".
Closing CCV	<ul style="list-style-type: none"> At the end of every sequence 	<ul style="list-style-type: none"> Must be in the range 90 to 110% 	<ul style="list-style-type: none"> Samples must be reanalyzed if possible, if not samples are flagged with a "Q".
BLK	One per prep batch	<ul style="list-style-type: none"> Must be less than $\frac{1}{2} \pm \text{RL}$. 	<ul style="list-style-type: none"> Re-analysis to confirm the positive value Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action Re-prepare of samples associated with the MB NCR will be required for data reported Final Report data flagging will be required

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
BS	One per prep batch	Must be in the range of 80 to 120% for 6010B, DOD; or 85 to 115% for 200.7.	<ul style="list-style-type: none"> • Rerun to confirm problem. • All samples associated with the LCS must be re-digested, reanalyzed if possible. • NCR will be required for data reported • If samples cannot be re-digested or re-analyzed Final Report data flagging will be required
MS	One per prep batch	Must be in the range of 80 to 120%	Final Report data flagging will be required
MSD	One per prep batch	Must be in the range of 80 to 120%	Final Report data flagging will be required
Sample Duplicate	One per prep batch	20%	Flag samples
Post Digestion Spike	One per batch	±25% for DOD/6010B, ±20% 6010C	If possible MSA required, Flag samples
DOC Study	<ul style="list-style-type: none"> • Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples 	<ul style="list-style-type: none"> • Must meet the criteria of the BS for average accuracy 	<ul style="list-style-type: none"> • Re-prep and / or • Re-analysis
MDL Study	Once per year		
LOD Verification	Every quarter		
LOQ Verification	Every quarter		
Linear Dynamic Range Study (LDR)	Every six months		

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were dilution factors applied correctly
5. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
6. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
7. Were proper data qualifiers applied to the data in LIMS
8. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual calculations verified

ANALYST DATA REVIEW CHECKLIST Sample Number(s):				
Batch Number(s):				
Method: 6010B or 6010C (ICP)				

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?	_____	_____	_____	_____
2. Was initial calibration curve QC criteria met?	_____	_____	_____	_____
3. Was all continuing calibration criteria in control?	_____	_____	_____	_____
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)	_____	_____	_____	_____
5. Did BS or blank spike meet control limits?	_____	_____	_____	_____
6. Did MS/MSD meet control limits?	_____	_____	_____	_____
7. Was the preparation (Method) Blank (BLK) below the project required detection limits?	_____	_____	_____	_____
8. Did you return samples back to cold storage immediately after use?	_____	_____	_____	_____
9. Was hot plate temperature monitored/documented and did you apply the thermometer correction factor?	_____	_____	_____	_____
10. Sample preparation information is correct and complete.	_____	_____	_____	_____
11. Analytical results are correct and complete.	_____	_____	_____	_____
12. The appropriate SOP's have been used and followed.	_____	_____	_____	_____
14. "Raw data" including all manual integration's have been correctly interpreted.	_____	_____	_____	_____
15. "Special" sample preparation and analytical requirements have been met.	_____	_____	_____	_____
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete.	_____	_____	_____	_____

Comments on any "No" response:

Analyst: _____ Date: _____

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

ORGANICS: SOP 327

REVISION #: 21

EFFECTIVE DATE: 20111031

**NITROAROMATICS AND NITRAMINES BY
HIGH PERFORMANCE
LIQUID CHROMATOGRAPHY (HPLC)
METHOD 8330, 8330A, 8330B and 8332**

APPROVALS:

Lab Director:



Date: 11/01/11

Data Quality Manager:



Date: 11/01/11

Section Supervisor:



Betty Quillen

Date: 11/01/11

Section Supervisor:



Jade Holliman

Date: 11/01/11

Changes Summary

Revision 21, 10/31/2011

- Equipment and supplies list updated to include 500g weight. Also referenced in section 14.6.
- Chilled shaker temperature requirement added to reflect <30°C as per method 8330B.
- 8330B sample bench sheets added in section 14.6 with updated process for weighing 8330B discrete and KG samples.
- Drying room temperature indicated as documented with pendant recorder in section 14.6.
- Extraction process for 10 gram added as section 14.10.
- Added information concerning eluent make-up/documentation to section 10.
- References to biphenyl column updated to reflect Aromax column.

Revision 20, 5/14/2011

- 8330B constant drying requirement updated from +/- 1% to +/- 4% in section 14.6.2 based on Standard Methods SM2540.
- Removed reference to chilled sonication bath and updated reference to chilled shaker to indicate validation by SW846 team as included in method 8330B.
- Added a requirement to monitor the temperature of the chilled shaker in section 14.8.2.
- Added a reference to the CRM for 8330B in section 10.6 and 14.7.6
- Added 30mesh sieve to list of equipment in section 9.4.10.
- Removed Nitroguanidine from Table 1.
- Updated references to Table 2 to reflect Table 2 or 3 where appropriate.
- Removed reference to section 8.6 calculations within section 15.

Revision 19, 10/04/2010

- 8330B drying equipment has been added
- 8330B drying process has been updated
- Standard expiration dates have been clarified
- 8330B DL/LOD/LOQ have been added to Table 1.

Revision 18, 9/7/2010

- The SOP is an update from Revision 17 dated 02/12/10
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.
- **8330B requirements updated with addition of Table 3 and Table 6.**

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Uncontrolled Document if Printed

1.0 Identification of the Test Method

This SOP, based on SW-846 Method 8330, 8330A, 8330B and 8332, is used for the trace analysis of explosives residues by high performance liquid chromatography (HPLC) using a UV-VIS detector in water, solid, or sediment matrices..

2.0 Applicable Matrix or Matrices

This SOP is used for the trace analysis of explosives residues by high performance liquid chromatography (HPLC) using a UV-VIS detector in water, solid, or sediment matrices.

3.0 Detection Limit

See **Table 1**.

4.0 Scope of Application, Including Components to Be Analyzed

4.1 Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. When applicable, surrogates are listed and indicated as such within this table.

4.2 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds and all handling of standards should be done in a hood. These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

5.1 Samples are analyzed after appropriate sample preparation using HPLC with identification at 210nm on a C-18 reverse phase column and confirmation at 210nm on a Aromax column. The preparation is performed using a solid phase extraction method, SW846 method 3535 for low concentrations of explosives residues in water. Dilution and filtration prepare high concentration water samples for direct injection. Extraction with acetonitrile in an ultrasonic bath or shaker followed by filtration prepares soil and sediment samples.

6.0 Definitions

6.1 Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.

7.0 Interferences

7.1 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences.

7.2 2-Am-DNT and 4-Am-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 should be reported.

7.3 Tetryl decomposes rapidly in methanol/water solutions, as well as with heat. Degradation products of tetryl appear as a shoulder on the 2,4,6-TNT peak, peak heights rather than peak areas should be used when tetryl is present in concentrations that are significant relative to the concentration of 2,4,6-TNT.

8.0 Safety

8.1 Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be followed labwide.

- 8.2 Standard precautionary measures used for handling other organic compounds should be sufficient for the safe handling of the analytes targeted by Method 8330A. The only extra caution that should be taken is when handling the analytical standard neat material for the explosives themselves and in rare cases where oil or waste samples are highly contaminated with the explosives. The HMX, RDX, Tetryl, and 2,4,6-TNT are explosives and the neat material should be handled carefully. Drying at ambient temperature requires several days. Do not dry at heated temperatures!
- 8.3 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.
- 8.4 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.
- 8.5 MSDS sheets are available for all reagents and standards which have been purchased. These are located in the office next to the technical director.

9.0 Equipment & Supplies

- 9.1 Instrumentation
- 9.1.1 Agilent Series 1100 HPLC System
 - 9.1.2 Agilent G1311A Quaternary Pump
 - 9.1.3 Agilent G1379A Degasser
 - 9.1.4 Agilent 1313A 100 Position Autosampler
 - 9.1.5 Agilent G1316A Column Compartment
 - 9.1.6 Agilent G1314A Variable Wavelength Detector
 - 9.1.7 Agilent HPLC Chemstation
 - 9.1.8 Primary Column: Restek Ultra C-18, or equivalent
 - 9.1.9 Secondary Column: Restek Ultra Aromax, or equivalent
- 9.2 Hewlett Packard Series 1050 HPLC System
- 9.2.1 Hewlett Packard Model 79852 1050 Quaternary HPLC Pump
 - 9.2.2 Hewlett Packard Model 79853 Variable Wavelength UV-VIS Detector
 - 9.2.3 Hewlett Packard 1050 79855A 21 Position Autosampler
 - 9.2.4 Hewlett Packard 1050 100 Position Autosampler Upgrade
 - 9.2.5 Hewlett Packard Model 79856A 1050 Solvent Module
 - 9.2.6 Hewlett Packard Model G1303A Vacuum Degassing Module
 - 9.2.7 Dell OptiPlex 933 GX150 Pentium III Computer
 - 9.2.8 Hewlett Packard PC Communication for HP Chemstation
 - 9.2.9 Hewlett Packard HPLC Chemstation
 - 9.2.10 SideWinder Temperature Control Module
 - 9.2.11 Primary Column: Restek Ultra C-18, or equivalent
 - 9.2.12 Secondary Column: Restek Ultra Aromax, or equivalent
- 9.3 Solid-phase extraction system consisting of:
- 9.3.1 Manifold Station, *J.T. Baker spe-12G*, or equivalent
 - 9.3.2 Tubing and connectors
 - 9.3.3 SFE extraction cartridges, *Porapak®RDX Cartridges* or equivalent
 - 9.3.4 Vacuum system capable of maintaining 18 inches of mercury
 - 9.3.5 Balance ± 0.01 g.
 - 9.3.6 Vortex mixer.

- 9.4 Other Components:
- 9.4.1 Chilled Shaker (validation by SW846 team as included in method 8330B.)
 - 9.4.2 Disposable cartridge filters - 0.45 mm PTFE filter.
 - 9.4.3 Scintillation Vials - 20 mL, glass.
 - 9.4.4 Vials - 15 mL, glass, Teflon-lined cap.
 - 9.4.5 Vials - 40 mL, glass, Teflon-lined cap.
 - 9.4.6 Disposable syringes - Plastipak, 3 mL and 10 mL or equivalent.
 - 9.4.7 Volumetric flask with ground glass stopper - 100 mL and 1000 mL.
 - 9.4.8 Vacuum desiccator - Glass.
 - 9.4.9 Mortar and pestle - Steel.
 - 9.4.10 Sieve – 10 mesh, 30 mesh & 200 mesh
 - 9.4.11 Graduated cylinders - 10 mL, 25 mL, 250 mL, 1000 mL.
 - 9.4.12 Pasteur pipet - length 9 ".
 - 9.4.13 Manual Sample Mill.
 - 9.4.14 Wiley Sample Mill.
 - 9.4.15 ESSA model LM-2P pulverizing mill (specified in 8330B)
 - 9.4.16 Clippers for cutting vegetation
 - 9.4.17 Drying cabinet & shelving inside
 - 9.4.18 500g Class I weight
 - 9.4.19 Pendant temperature recorder for monitoring 8330B drying room temperature.
 - 9.4.20 Heavy duty zip-lock bag
 - 9.4.21 Tamper

10.0 Reagents and Standards

- 10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:
- 10.2 HPLC grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Certified stock standards are purchased from Restek, Protocol, Ultra and other vendors depending on their availability. The date they are received is noted on the label or container they are received in and in the LIMS system. The date the standards are opened they are recorded and given a sequential number in the LIMS system. All stock standards are stored at 4°C.
- 10.3 List of Reagents
- Acetonitrile, CH₃CN - HPLC grade.
 - Methanol, CH₃OH - HPLC grade.
 - Calcium chloride, CaCl₂ - Reagent grade. Prepare an aqueous solution of 5g/L. This is for use with soil/sediment samples.
 - Organic-free reagent water. Obtained from the Nano Pure Water System in the GC lab.
 - Sodium Chloride solution - 325 g NaCl per 1000 mL reagent water.
- 10.4 Eluents are made up daily, using the reagents listed above, as required for the analytical columns used. Eluent make-up is documented on the daily sequence log/analytical run log,
- 10.5 Stock standards are purchased in mixtures from reputable vendors. The date they are received and opened is recorded in LIMs along with their lot number and vendor. Each standard that is prepared is recorded in the LIMs and given a sequential number. Each standards label is completed with the standard number, name, concentration, expiration date, and analyst initials. All standards are stored in the refrigerator in the dark at a temperature of 4°C ± 2°C or less from

the date they are received/prepared. The refrigerator temperature is monitored daily with an annually calibrated thermometer and recorded with calibration correction in the GC refrigerator/freezer logbook or the Extraction temperature/calibration logbook. Makeup of some common standards is detailed below. The makeup of other standards can be found in the LIMs. In general, stock standards expire 6 months from the date opened. If the manufacturer expiration date is earlier then the earlier date is used. If the manufacturer expiration date lists month/year, the last day of the month is used.

10.6 Calibration Solutions - The 8330 calibration standards are prepared as follows using solutions purchased from Restek or an equivalent vendor.

10.6.1 Explosive (Mix#1,#2 and Surrogate) Calibration Stock Solutions: Using a 100 μ L syringe, 100 μ L of Mix # 1, Mix # 2, and Surrogate (Restek at 1000 μ g/mL) are injected into a 10mL volumetric flask containing approximately 9.5mL 1:1 acetonitrile and water and diluted to volume with same to make a 10 μ g/mL standard. After capping and inverting several times, the solution is transferred into a labeled, 12ml, teflon-lined, screw-capped vial and stored in the refrigerator in the dark at 4°C or less for up to 30 days. These standards are used to make the calibration curve standards in 1:1 acetonitrile and water at concentrations of 10, 5.0, 1.0, 0.50, 0.10 and 0.025 μ g/mL. The lowest calibration standard can be extended down to 0.010 μ g/ml, if required.

10.6.2 Mix # 3 Calibration Stock Solution: Using a 500 μ L syringe, 500 μ L of Mix # 3 (Restek PETN & Nitroglycerin, NG, at 1000 μ g/mL) is injected into a 10mL volumetric flask containing approximately 9.0mL 1:1 acetonitrile and water and diluted to volume with same to make a 10 μ g/mL standard. After capping and inverting several times, the solutions are transferred into a labeled, 12ml, teflon-lined, screw-capped vial and stored in the refrigerator in the dark at 4°C or less for up to 30 days. These standards are used to make the calibration curve standards in 1:1 acetonitrile and water at concentrations of 50, 25, 5, 2.5, 0.50 and 0.25 μ g/mL. The working calibration standards must be prepared fresh the day of use.

10.6.3 Matrix/LCS Spike Standard - The 8330 spiking solution is prepared by adding 0.5mL of a solution purchased from Ultra (Combined Stock Solution) in 50mL of acetonitrile for a final concentration of 10 μ g/mL. Samples are spiked with 1 mL of this solution.

10.6.4 Second Source Calibration Solution - The 8330 second source standard is prepared as follows using a solution purchased from Ultra(Combined Stock Solution) and Accustandard Inc.(Mix #3) or an equivalent vendor.

10.6.5 Second Source Stock Solutions: Using a 100 μ L syringe, 100 μ L of Combined Stock Solution (Ultra at 1000 μ g/mL) is injected into a 10mL volumetric flask containing approximately 9.5mL 1:1 acetonitrile and water and diluted to volume with same to make a 10 μ g/mL standard. After capping and inverting several times, the solution is transferred into a labeled, 12ml, teflon-lined, screw-capped vial and stored in the refrigerator in the dark at 4°C or less for up to 30 days. This standard is used to make a second source check standard 1:1 acetonitrile and water at 1.0 μ g/mL.

Using a 500 μ L syringe, 500 μ L of Mix #3(Accustandard at 1000 μ g/mL) is injected into a 10mL volumetric flask containing approximately 9.0mL 1:1 acetonitrile and water and diluted to volume with same to make a 10 μ g/mL standard. After capping and inverting several times, the solution is transferred into a labeled, 12ml, teflon-lined, screw-capped vial and stored in the refrigerator in the dark at 4°C or less for up to 30 days. This standard is used to make a second source check standard 1:1 acetonitrile and water at 5.0 μ g/mL.

10.6.6 Surrogate Spike Solution- The 1,2-dinitrobenzene solution (1-Chloro-3-Nitrobenzene may be used as an alternate) is prepared as follows using a standard purchased from Restek or an equivalent vendor.

Using a 250µL syringe, 200µL of 1,2-dinitrobenzene, (or 1-Chloro-3-Nitrobenzene), (Restek at 1000 µg/mL) is injected into a 100mL volumetric flask containing approximately 95mL acetonitrile and diluted to volume with same to make a 2.0 µg/mL standard. After capping and inverting several times, the solution is transferred into several labeled, 40ml, teflon-lined, screw-capped vial and stored in the refrigerator in the dark at 4°C or less for up to 30 days. Samples are spiked with 1 mL of this solution.

- 10.6 Certified Reference Material – 8330B reference materials are purchased from reputable vendors such as Wibby. The date they are received and opened is recorded in LIMS along with their lot number, vendor and a scanned copy of their COA. Alternatively, Soil PT study samples may be purchased and used once the results are returned with the true values reported (report used as COA in LIMS).

11.0 Sample Collection, Preservation, Shipment, and Storage

- 11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.
- 11.2 Samples and sample extracts are stored in the dark at 4°C. Samples are stored in the sample storage walk-in coolers. Extracts are stored in the Hobart in the Semivolatiles laboratory. The holding time for samples is 7 days for waters and 14 days for soils. The holding time for extracts is 40 days.
- 11.3 RT Windows
Retention time (RT) windows are determined for each compound through the analysis of 3 standards over a 72 hour period. The standard deviation of the standard retention times is calculated and the RT windows are determined to be $\pm 3x$ this standard deviation. New in-house retention time windows are established after every major change to the system (new column or flow) and after a new initial calibration using the mid-point standard. Retention times of each analyte in each CCV are compared to the established retention time window. Each analyte must fall within its respective RT window. If this criterion is not met, the system must be adjusted to allow another CCV to meet criteria, or a new initial calibration performed and new retention time windows established.

12.0 Quality Control

- 12.1 Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.
- 12.2 An initial demonstration must be performed by each analyst performing this method. See [Table 2 or 3](#) for acceptance criteria.
- 12.3 Surrogate - All samples and QC are spiked with the surrogate. See [Table 2 or 3](#) for acceptance criteria and corrective action.
- 12.4 LCS Sample - The LCS is analyzed at the frequency required by the regulatory agency or client (every batch or 20 samples). To prepare the LCS, a blank is spiked with the LCS mix standards. See [Table 2 or 3](#) for acceptance criteria and corrective action..
- 12.5 Method Blanks - The Method Blank is extracted 1 per extraction batch of up to 20 samples. See [Table 2 or 3](#) for acceptance criteria and corrective action.
- 12.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD, if sample is available. If no sample is available, an LCS must be extracted to provide precision results. See [Table 2 or 3](#) for acceptance criteria and corrective action. MS data evaluation must include the consideration of the following factors.
- 12.7 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. Corrective action must be taken in the form of reanalysis if a method problem is indicated.

- 12.8 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was two or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.
- 12.9 MS vs. MSD - If a spiked compound has a similar problem in the MS and the MSD which is not traceable to the execution of the method, no further corrective action may be necessary other than the generation of a corrective action report to document the problem as matrix effect.
- 12.10 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.

13.0 Calibration and Standardization

- 13.1 Quality Systems **SOP QS08** "Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.
- 13.2 Calibration of HPLC
 - 13.2.1 Upon initial startup of the pump, flow is increased to 5.0mL/min after opening the purge valve to bleed air from the solvent/water lines. When all air bubbles have been removed, the flow is reduced to normal run conditions and the purge valve is closed. The instrument is then pumped with 100% solvent for 45 minutes (or less) and then pumped with the appropriate solvent/water mixture for an additional 45 minutes (or longer).
 - 13.2.2 Initial Calibration: Injections of each calibration standard over the concentration range of interest are sequentially injected into the HPLC. Peak areas or heights are obtained for each analyte. (Peak height may be used instead of peak area for 2,4,6-TNT because degradation products of tetryl appear as a shoulder on the 2,4,6-TNT peak.) The calibration curve should be linear. However, some target analytes may be difficult to optimize without application of quadratic or higher order mathematical functions. Linearity may be determined using linear regression analysis for each target analyte by calculating the correlation coefficient r . Another term used to describe the goodness of fit of the line is coefficient of determination r^2 (the square correlation coefficient). The resulting line would normally not be forced through the origin or use the origin as a calibration point unless it is demonstrated that the intercept of the regression line is not statistically different from zero at 95% level of confidence. See **Table 2 or 3** for acceptance criteria and corrective action.
 - 13.2.3 Due to the lack of resolution between 2-Am-DNT and 4-AM-DNT, calibration of these compounds can be based on "isomeric pairs". Improved resolution may be obtained using a Supelco C-18 column with eluent of 57%/43% (v/v) methanol and water at 1.0 mL/min.
 - 13.2.4 A visual inspection of the calibration curve should also be used as a diagnostic tool when nonlinear behavior is observed to verify if there is a large percentage error in any particular portion of the calibration curve. If the visual inspection indicates problems, or if one criteria is not met, then evaluate the following items for implementation based on an understanding of the detector response/contaminant concentration relationship.
 - 13.2.5 Check the instrument operating conditions or the initial calibration standards used and make adjustments to achieve a linear calibration curve.
 - 13.2.6 Narrow the calibration range using the same number of standards. Generally the highest standard is lowered first. The consequences of all actions taken must also be evaluated, i.e., reduction of the calibration range, raising of the Method Quantitation Limit, MQL, etc.

- 13.2.7 Evaluate the use of a nonlinear curve, when applicable. When nonlinear calibration models are used, the resultant line should not be forced through the origin and the origin should not be used as a calibration point. No higher than a third-order (cubic) calibration model shall be used. When a nonlinear calibration model is employed, more data points are needed to maintain at least three degrees of freedom. For example, use of a quadratic function requires at least a six-point initial calibration curve. The resulting r^2 should be greater than or equal to 0.99 for this to be considered acceptable.
- 13.2.8 Use of alternative techniques (e.g., relative standard error (RSE) outlined in the USEPA Memorandum, "Clarification Regarding Use of SW-846 Methods" (EPA/SW-846).
- 13.2.9 The standards used to make the calibration curve are verified to be accurate using a standard obtained from a second source, initial calibration verification (ICV). See Table 2 for acceptance criteria.
- 13.2.10 Daily Calibration: Continuing calibration verification (CCV) standards must be analyzed, at a minimum, at the beginning of the day, after every 20 samples and at the end of the sequence. See **Table 2 or 3** for acceptance criteria and corrective action.

14.0 Procedure

- 14.1 All waters have a 7-day holding time and soils have a 14-day holding time. Determine the samples necessary to extract as follows:
 - 14.1.1 Each day a backlog report will be provided indicating sample numbers with the respective analysis required. Line through all the extractions that have been completed and plan to do the remaining analysis within the required holding time.
 - 14.1.2 Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel will generally communicate this need.
 - 14.1.3 Check with log-in throughout the day and examine the COC (chain of custody) forms that arrive with each set of samples. If an analysis is ongoing, extra QC may be avoided by picking up those extractions on the same day.
 - 14.1.4 Wearing lab coat, gloves and safety glasses, get samples from cooler. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass(soil)/glass amber jar(water) and have a Teflon lid. Find out if any special dilutions or screens need to be made for this client.
- 14.2 Before extraction, all glassware must be prepared as instructed in SOP-306. Before weighing, the balance must be calibrated with ASTM Class I weights which bracket the amount to be weighed and recorded in the Extraction temperature/calibration logbook. If a heavy container is to be used for weighing, place a representative container on the balance, tare the balance and then calibrate the balance with the chosen weights. During extraction, all pertinent information (glassware, amounts, reagent lots, standards, etc.) is recorded in the HPLC 8330 extraction logbook so as to allow reconstruction of the extraction in the future.
- 14.3 High Level Aqueous Extraction Method
 - 14.3.1 Before extraction begins get out enough scintillation vials for each sample, method blank, laboratory control sample, matrix spike and matrix spike duplicate. Place an Avery label on each vial containing the following information: Lab #, Client name, Type of Analysis, Initial Volume - Final Volume, and the Lab Prep Batch Code.
 - 14.3.2 Place a 5 mL aliquot of each water sample in an appropriately labeled scintillation vial and add 4 mL of acetonitrile (3 mL for MS/MSD samples). Add 1.0 mL of the surrogate standard (2.0 µg/mL) using a 1.0 mL syringe to each sample, method blank and QC sample. Add 1.0 ml of 8330 standard spiking solution to each appropriate QC sample (LCS, MS&MSD). Shake samples thoroughly, and filter through a 0.45mm PTFE filter using a disposable syringe. Discard the first 3 mL of filtrate, and retain the remainder in

a Teflon-capped vial for HPLC analysis. HMX quantitation can be improved with the use of methanol rather than acetonitrile for dilution before filtration. For screening purposes, 1 ml of sample is placed in a 4 ml vial along with 0.5 ml of acetonitrile and 0.5 ml of water.

14.4 Solid-Phase Extraction

This extraction method may not be appropriate for aqueous samples with greater than 1% suspended solids. Consult SW-846 Method 3535 for additional information.

14.4.1 Mark the outside of the sample container at the sample meniscus with "white-out". This mark will be used to determine the initial sample volume after processing the contents. Add 1.0 mL of the surrogate standard (2.0 µg/mL) using a 1.0 mL syringe to each sample, method blank and QC sample. Add 1.0 ml of standard spiking solution to each appropriate QC sample (LCS, MS&MSD).

14.4.2 Assemble the manifold for multiple extractions with SPE cartridges.

14.4.3 Wash the cartridges with 6 mL acetonitrile 3 times and 6 mL reagent water 6 times with gravity flow, do not let cartridge go dry. If it goes dry, you must start over.

14.4.4 Add sample to the cartridge and attach connectors and tubing.

14.4.5 Turn on the vacuum pump and begin drawing sample through the cartridge, while adjusting the flow to 10mL/min.

14.4.6 Empty the water trap as needed.

14.4.7 After the sample extraction is complete draw air through the cartridge for 15 minutes to dry.

14.4.8 Add 4 mL of acetonitrile to the cartridge and allow it to pass through with gravity flow collecting it in a 12 mL vial. Note: the volume of acetonitrile may be reduced to 3ml to lower detection limits. Place an Avery label on each tube containing the following information: Lab #, Client name, Type of Analysis, Initial Volume - Final Volume, and the Lab Prep Batch Code. Bring extract up to 4 ml with acetonitrile. Sample extracts are diluted 2x with DI water prior to analysis. Record this volume in the HPLC extraction logbook. The extract is ready for analysis, proceed to Section 8.0.

14.4.9 Determine the original sample volume by refilling the sample bottle to the mark made with "white out". Transfer the liquid to a plastic 1000-mL graduated cylinder and record the sample volume in the LIMS bench sheet to the nearest 10-mL.

14.5 Soil, Sediment and Nonaqueous Samples

14.5.1 Dry representative soil samples at room temperature, normally overnight, being careful not to expose the samples to direct sunlight. Grind and homogenize the dried sample thoroughly in an acetonitrile rinsed mortar so it will pass through a 30 mesh sieve. In other words, grind to a fine-dust like particle size. If one grinds the sample down to this small of a partial size, then the sieve would not be required.

14.5.2 NOTE : Soil samples may be screened by a commercially available test kit prior to grinding in a mortar and pestle. Visually observe the sample for lumps of material that have a chemical appearance. These lumps should be suspect and not ground. Explosives are generally a very finely ground grayish-white material. Soil samples as high as 2% 2,4,6-TNT have been safely ground. Samples containing higher concentrations should not be ground in the mortar and pestle. 2,4,6-TNT is the analyte most often detected in high concentration in soil samples.

14.6 Sample Drying (8330B)

14.6.1 Samples must be dried to a constant weight (<4% difference). As this requires weighing large sample aliquots, the balance calibration must include the 500g weight to bracket sample weights being measured.

- 14.6.1.1 Discrete samples are thoroughly mixed then placed in aluminum pie pans (labeled with lab ID) for drying. In “Totals_Oversized” bench sheet, record the weight of the pie pan then add the entire sample and record the weight of the pie pan plus the sample in “Beginning Weight”.
- 14.6.1.2 Kg samples are thoroughly mixed then split into aliquots of approximately 500g. The balance is tared with a large disposable aluminum cookie sheet then sample aliquots are placed on large sheets of aluminum foil (labeled with lab ID and aliquot designation) for weighing. Record the weight of the sample aliquot in “Splits” bench sheet.
- 14.6.2 Samples are placed on shelves that are located in the 8330B drying cabinet. Make sure that the cabinet exhaust fan has been turned on before you place the samples in the cabinet.
- 14.6.3 Room temperature is monitored using a pendant data recorder from which data is downloaded monthly. Verify pendant is blinking to confirm monitoring and provide pendant to Data Quality Manager for download following sample drying. Charts are maintained via PDF with most recent chart printed and placed on the bench next to the drying cabinet.
- 14.6.3 Dry the entire sample at room temperature overnight (minimum), to a maximum of two (2) days, being careful to avoid direct sunlight. After the initial drying, weigh the associated aliquots and record on associated bench sheets (Totals_Oversized for discrete and Splits for Kg samples). Replace in the drying cabinet and wait at least 2 hours to measure second set of weights. Check percent difference from first dry weight to second dry weight to determine if additional drying time is required (%D >4%).
- 14.6.4 When sample has come to a constant weight, sieve the entire sample with a 10 mesh sieve. Break up pieces of soil (especially clay) with gloved hands. If sample is particularly difficult to break up, place sample in heavy duty ziplock bag and use tamper to perform initial break up. Do not include vegetation in the portion of the sample that passes through the sieve unless this has been identified for inclusion as a project specific requirement.
- 14.6.5 Collect and weigh any portion unable to pass through the sieve. Record weight on “Totals_Oversized” bench sheet as “Oversized Portion”.

8330B Split Weight Log

SAMPLE ID:	Weight A	Weight B	Weight C	Weight D	TOTAL 1	DATE/TIME	Analyst	4%	RANGE	Weight A	Weight B	Weight C	Weight D	TOTAL 2	DATE/TIME	Analyst	4%	RANGE	Weight A	Weight B	Weight C	Weight D	TOTAL 3	DATE/TIME	Analyst	
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8830B Total_Oversized Weight Log

	Sample ID:	Date/ Time	Analyst	Pan Weight (discrete)	Beginning Weight (no tare):	Date/ Time	Analyst	2nd Weight (no tare):	%D	Date/ Time	Analyst	3rd Weight (no tare):	%D	Calculated Final Weight	Oversized Portion:	Description of Oversized Portion:	Analyst/Comments:
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2																	
3																	
4																	
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Samples must be dried to a constant weight, + - 4%

Oversized portion is anything that does not pass through the 10-mesh sieve. Description of oversized portion must be documented. Oversized portions are not ground and used in the analysis.

Umc

- 14.7 Sample Grinding (8330B)
- 14.7.1 Sample grinding for soil samples from ammunition plants, depots, and firing ranges.
 - 14.7.2 A grinder blank (between each sample), using Ottawa sand, must be prepped (ground and sub-sampled) and analyzed in the exact same manner as a field sample. Each analytical batch will have a grinder blank sample. See **Table 3** for acceptance criteria and corrective action.
 - 14.7.3 Soil Sub-sampling Procedure: Each ground sample is mixed and spread out on a large flat surface like a baking tray or pie pan, and 30 or more randomly located increments are removed from the entire depth to obtain the sum of at least a 10g sample.
 - 14.7.4 Soil Sample Triplicate Determination: At the subsampling step, one sample per batch (cannot be a blank) will undergo triplicate analysis. Three 10g subsamples are taken from a sample expected to contain the highest level of explosives. If this information is not available, a random sample will be picked. See **Table 3** for acceptance criteria and corrective action.
 - 14.7.5 Certified Reference Material: A solid CRM must be extracted with every batch of 8330B soil samples. See **Table 3** for acceptance criteria and corrective action.
- 14.8 Grinding of Vegetation Samples: Remove sample from shipping container and brush off dirt particles. Chop sample into about half inch pieces with clippers or other cutting tool. Place the sample in an aluminum pan and air-dry in an exhaust hood to the appropriate dryness for grinding. It should be dry enough where it won't stick to the inside of the mill. Grind the dried sample to fineness in either the manual sample mill or the Wiley mill or both if needed. Place the ground sample in a container and label immediately. Use 6g for extraction.
- 14.9 2g Sample Extraction: Get out enough 20ml scintillation vials for each sample, method blank and QC sample to be extracted. Place an Avery label on each vial containing the following information: Lab #, Client name, Type of Analysis, Initial Weight - Final Volume, and the Lab Prep Batch Code. Weigh-out a 2.0 – 2.3 g subsample of each soil (use a blank matrix soil for each method blank and LCS) into the appropriately labeled vial. To each spiked QC sample, (LCS, MS&MSD), add 8.0 mL of acetonitrile. Then add 1.0 mL of surrogate (2.0µg/mL) standard and 1.0 ml of 8330 standard spiking solution. To each sample and method blank add 9.0 mL of acetonitrile and 1.0 mL of surrogate (2.0 µg/mL) standard using a 1.0 mL syringe. Cap each vial and place in chilled shaker for 16-18 hours. Reset the chilled shaker Min/Max thermometer at the start of the 16-18 hours and record the Min/Max temperature of the chilled shaker when removing samples from the shaker. Chilled shaker is required to maintain a temperature below 30°C.
- 14.9.1 After shaking, allow the sample to settle (10-15 minutes should be adequate). Add 10mL of calcium chloride and centrifuge samples for 10 minutes. (The calcium chloride solution is added to the samples to coagulate suspended particles and remove them from the supernatant.) Make sure the sample is labeled correctly.
- 14.10 10g Sample Extraction: Get out enough 40 mL vials for each sample, method blank and QC sample to be extracted. Place an Avery label on each vial containing the following information: Lab #, Client name, Type of Analysis, Initial Weight - Final Volume, and the Lab Prep Batch Code. Weigh-out a 10.0 – 10.5 g subsample of each soil (use a blank matrix soil for each method blank and LCS) into the appropriately labeled vial. To each spiked QC sample, (LCS, MS&MSD), add 8.0 mL of acetonitrile. Then add 1.0 mL of surrogate (2.0µg/mL) standard and 1.0 ml of 8330 standard spiking solution. To each sample, CRM and method blank add 9.0 mL of acetonitrile and 1.0 mL of surrogate (2.0 µg/mL) standard using a 1.0 mL syringe. Cap each vial with a Teflon-lined cap and place in chilled shaker for 16-18 hours. Reset the chilled shaker

Min/Max thermometer at the start of the 16-18 hours and record the Min/Max temperature of the chilled shaker when removing samples from the shaker. Chilled shaker is required to maintain a temperature below 30°C.

14.10.1 After shaking, allow the sample to settle (10-15 minutes should be adequate). Add 10mL of calcium chloride and centrifuge samples for 10 minutes. (The calcium chloride solution is added to the samples to coagulate suspended particles and remove them from the supernatant.) Make sure the sample is labeled correctly.

14.11 Sample Analysis

14.11.1 Samples will be prepared, analyzed and reported in batches and will be traceable to their respective batches. Quality control, QC, samples are required with each batch. A method blank, matrix spike/matrix spike duplicate and laboratory control sample is required for each sample matrix batch (normally sets of 20 samples).

14.11.2 Analyze the samples using the same conditions as the standards. Compounds identified on the C-18 column must be confirmed by injection on the Aromax column with an RPD limit of 40%. If the RPD exceeds 40%, results should be evaluated to determine if coelution or matrix is causing the exceedence and the reason noted. In cases of coelution or obvious matrix interference, the lower concentration may need to be reported. If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the primary column results.

14.11.3 Identification of a compound is made if a peak is found within the RT window on the C-18 column and then confirmed on the Aromax column. Column temperature control is employed so retention time shifts should not be a problem.

15.0 Data Analysis and Calculations

15.1 Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2 Data Reduction/Evaluation

15.2.1 Each sample analysis sequence is documented in the HPLC run log. After the samples have been analyzed, the data is reduced using Target. The following must be checked to determine if the sample will need any reanalysis or dilution:

15.2.2 The initial CCV must be within $\pm 15\%$ difference of the calibration curve. See **Table 2** for acceptance criteria and corrective action.

15.2.3 Analyte concentration must be within the range of the calibration curve. If an analyte exceeds the curve, a dilution must be performed.

15.2.4 Surrogate recovery should be within the limits established by the laboratory of 40-145% for water and 55-140% for solids/project sample matrix. See **Table 2** for acceptance criteria and corrective action.

15.2.5 After the data has been reduced and determined to be acceptable, it is uploaded into the LIMS and reviewed. Any manual integrations are documented by inclusion of the integrated signals (before and after manual integration) initialed, reason and dated with the quantitation report and chromatogram. Refer to QS07 for guidance.

15.3 Calculations

15.3.1 Calculate the calibration factor for each analyte at each concentration as:

$$CF = \frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

15.3.2 The mean CF is calculated as follows:

$$\overline{CF} = \frac{\sum_{i=1}^n CF_i}{n}$$

13.3.3 The standard deviation (SD) and the relative standard deviation (RSD) of the calibration factors for each analyte are calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^n (CF_i - \overline{CF})^2}{n - 1}}$$

$$RSD = \frac{SD}{\overline{CF}} \times 100$$

15.3.4 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\% \text{ Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{(\text{CCV CF} - \text{Average CF}) * 100}{\text{Average CF}}$$

15.3.5 External standard calibration - The concentration of each analyte in the sample may be determined by calculating the amount of standard injected, from the peak response, using the calibration curve. The concentration of a specific analyte is calculated as follows:

A. Aqueous Samples:

$$\text{Concentration } (\mu\text{g/L}) = \frac{[(A_s) (V_t) (D)]}{[(CF) (V_i) (\overline{D})]}$$

where:

A_s = Response for the analyte in the sample, units may be in area counts or peak height.

V_t = Total volume of the concentrated extract..

D = Dilution factor, if dilution was made on the sample prior to analysis. If no dilution was made, D = 1.

\overline{CF} = Mean calibration factor from initial calibration (area/ng)

V_i = Volume of extract injected, μL .

V_s = Volume of aqueous sample extracted, mL.

Using the units specified here for these terms will result in concentration units of ng/mL, which is $\mu\text{g/L}$.

B. Nonaqueous Samples:

$$\text{Concentration } (\mu\text{g/kg}) = \frac{[(A_s) (V_t) (D)]}{[(\overline{CF}) (V_i) (W_s)]}$$

where:

W_s = Weight of sample extracted, g. The wet weight or dry weight may be used, depending upon the specific applications of the data.

A_s , V_t , D, CF and V_i have the same definition as for aqueous samples.

16.0 Method Performance

Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. See **Table 2** for acceptance criteria and corrective action.

Initial grinding demonstration: The lab must initially show that the mechanical grinder is capable of reducing the particle sieve to < 75 microns by passing representative portions of ground sample through a 200 mesh sieve. The data for this demonstration will be kept on file with the DQM.

17.0 Pollution Prevention

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, “Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results”, provides details on handling out of control data. **Table 2 and 3** within this SOP also list corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20.0 Waste Management

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.0 References

- 21.1 *40 CFR, Part 136; Appendix A*
- 21.2 *Test Methods for Evaluating Solid Waste, SW-846, Third Edition*
- 21.3 National Environmental Laboratory Accreditation Conference; Chap. 5, 2003
- 21.4 DOD Quality Systems Manual for Environmental Laboratories, Ver. 3, Jan. 2006.
- 21.5 DOD Quality Systems Manual for Environmental Laboratories, Ver. 4.1, April, 2009.

22.0 Tables, Diagrams, Flowcharts and Validation Data

- 22.1 Table 1, all applicable parameters, including the surrogates and internals with the applicable RL and lowest calibration standard.
- 22.2 Table 2, 8330/8330A QA/QC summary table.
- 22.3 Table 3, 8330B QA/QC summary table.
- 22.4 Table 4, Technical Completeness / Accuracy Checklist
- 22.5 Table 5, Data Reviewers Checklist 8330/8330A
- 22.6 Table 6, Data Reviewers Checklist 8330B

TABLE 1 (DL/LOD/LOQ).

Analyte	MDL/DL	LOD	MRL/LOQ	Units
1,3,5-Trinitrobenzene 8330A	0.100	0.200	0.400	mg/Kg
1,3-Dinitrobenzene 8330A	0.100	0.200	0.400	mg/Kg
2,4,6-Trinitrophenylmethylnitramine (Tetryl) 8330A	0.100	0.200	0.400	mg/Kg
2,4,6-Trinitrotoluene (TNT) 8330A	0.100	0.200	0.400	mg/Kg
2,4-Dinitrotoluene (DNT) 8330A	0.100	0.200	0.400	mg/Kg
2,6-Dinitrotoluene 8330A	0.100	0.200	0.400	mg/Kg
2-Amino-4,6-dinitrotoluene 8330A	0.100	0.200	0.400	mg/Kg
2-Nitrotoluene (ONT) 8330A	0.100	0.200	0.400	mg/Kg
3,5-Dinitroaniline 8330A	0.100	0.200	0.400	mg/Kg
3-Nitrotoluene 8330A	0.100	0.200	0.400	mg/Kg
4-Amino-2,6-dinitrotoluene 8330A	0.100	0.200	0.400	mg/Kg
4-Nitrotoluene (PNT) 8330A	0.100	0.200	0.400	mg/Kg
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) 8330A	0.100	0.200	0.400	mg/Kg
Nitrobenzene 8330A	0.100	0.200	0.400	mg/Kg
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) 8330A	0.250	0.500	1.00	mg/Kg
Nitroglycerin 8330A	0.100	0.200	0.400	mg/Kg
PETN 8330A	0.250	0.500	1.00	mg/Kg
1,3,5-Trinitrobenzene 8330A/B	0.0800	0.160	0.320	ug/L
1,3-Dinitrobenzene 8330A/B	0.0800	0.160	0.320	ug/L
2,4,6-Trinitrophenylmethylnitramine (Tetryl) 8330A/B	0.0800	0.160	0.320	ug/L
2,4,6-Trinitrotoluene (TNT) 8330A/B	0.0800	0.160	0.320	ug/L
2,4-Dinitrotoluene (DNT) 8330A/B	0.0800	0.160	0.320	ug/L
2,6-Dinitrotoluene 8330A/B	0.0800	0.160	0.320	ug/L
2-Amino-4,6-dinitrotoluene 8330A/B	0.0800	0.160	0.320	ug/L
2-Nitrotoluene (ONT) 8330A/B	0.0800	0.160	0.320	ug/L
3,5-Dinitroaniline 8330A/B	0.0800	0.160	0.320	ug/L
3-Nitrotoluene 8330A/B	0.0800	0.160	0.320	ug/L
4-Amino-2,6-dinitrotoluene 8330A/B	0.0800	0.160	0.320	ug/L
4-Nitrotoluene (PNT) 8330A/B	0.0800	0.160	0.320	ug/L
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) 8330A/B	0.0800	0.160	0.320	ug/L
Nitrobenzene 8330A/B	0.0800	0.160	0.320	ug/L
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) 8330A/B	0.200	0.400	0.800	ug/L
Nitroglycerin 8330A/B	0.0800	0.160	0.320	ug/L
PETN 8330A/B	0.200	0.400	0.800	ug/L
1,3,5-Trinitrobenzene 8330B	0.080	0.040	0.020	mg/Kg
1,3-Dinitrobenzene 8330B	0.080	0.040	0.020	mg/Kg
2,4,6-Trinitrophenylmethylnitramine (Tetryl) 8330B	0.080	0.040	0.020	mg/Kg
2,4,6-Trinitrotoluene (TNT) 8330B	0.080	0.040	0.020	mg/Kg
2,4-Dinitrotoluene (DNT) 8330B	0.080	0.040	0.020	mg/Kg
2,6-Dinitrotoluene 8330B	0.080	0.040	0.020	mg/Kg
2-Amino-4,6-dinitrotoluene 8330B	0.080	0.040	0.020	mg/Kg
2-Nitrotoluene (ONT) 8330B	0.080	0.040	0.020	mg/Kg
3,5-Dinitroaniline 8330B	0.080	0.040	0.020	mg/Kg
3-Nitrotoluene 8330B	0.080	0.040	0.020	mg/Kg
4-Amino-2,6-dinitrotoluene 8330B	0.080	0.040	0.020	mg/Kg
4-Nitrotoluene (PNT) 8330B	0.080	0.040	0.020	mg/Kg
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) 8330B	0.080	0.040	0.020	mg/Kg
Nitrobenzene 8330B	0.080	0.040	0.020	mg/Kg
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) 8330B	0.080	0.040	0.020	mg/Kg
Nitroglycerin 8330B	0.400	0.200	0.100	mg/Kg
PETN 8330B	0.400	0.200	0.100	mg/Kg

Table 2. Organic Analysis by High-Performance Liquid Chromatography (Method 8330 and 8330A)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f of DoD QSM 4.1).	Not Applicable (NA).	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL determination	Initial method demonstration required for some states – not required for DoD	Refer to SOP QS09.			
LOD determination and verification	Prior to initial analysis then quarterly verification.	See Box D-13 of DoD QSM 4.1			
LOQ establishment and verification	Prior to initial analysis then quarterly verification.	See Box D-14 of DoD QSM 4.1			
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from a 72-hour study.	NA.	NA.	
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	One of the options below: Option 1: RSD for each analyte $\leq 20\%$ Option 2: linear least squares regression: $r \geq 0.995$ Option 3: non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin for DoD projects.
Retention time window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	

Table 2. Organic Analysis by High-Performance Liquid Chromatography (Method 8330 and 8330A)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second source calibration verification (ICV)	Following ICAL, prior to sample analysis.	All project analytes within established retention time windows. <u>HPLC methods:</u> All project analytes within $\pm 15\%$ of expected value from the ICAL.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples should be run until calibration has been verified.
Continuing calibration verification (CCV)	Prior to sample analysis, after every 10 field samples (maximum of 20 for non-DoD projects), and at the end of the analysis sequence.	All project analytes within established retention time windows. <u>HPLC methods:</u> All project analytes within $\pm 15\%$ of expected value from the ICAL.	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results should not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method blank	One per preparatory batch.	No analytes detected $> \frac{1}{2}$ RL and $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by client or DoD (appendix G), if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery.	Correct problem. Reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed. In general, the LCS is analyzed on the primary column only.

Table 2. Organic Analysis by High-Performance Liquid Chromatography (Method 8330 and 8330A)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix.	For matrix evaluation, use LCS acceptance criteria above.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error. In general, the MS is analyzed on the primary column only.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix.	MSD: For matrix evaluation, use LCS acceptance criteria above. MSD or sample duplicate: RPD \leq 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference. In general, the MSD is analyzed on the primary column only.
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply qualifier to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column or second detector)	All positive results must be confirmed.	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD \leq 40%.	NA.	Apply P-flag if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method reporting requirements; otherwise, report the result from the primary column. In general, all spiked batch QC is analyzed on the primary column only.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

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Table 3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (Method 8330B)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f of DoD QSM 4.1).	Flagging criteria are not appropriate.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Prior to initial analysis then quarterly verification.	See Box D-13 of DoD QSM 4.1			
LOQ establishment and verification	Prior to initial analysis then quarterly verification.	See Box D-14 of DoD QSM 4.1			
Soil drying procedure	Each sample and batch LCS.	Laboratory must have a procedure to determine when the sample is dry to constant weight. Record date, time, and ambient temperature on a daily basis while drying samples.	NA.	Flagging criteria are not appropriate.	
Soil sieving procedure	Each sample and batch LCS.	Weigh entire sample. Sieve entire sample with a 10 mesh sieve. Breakup pieces of soil (especially clay) with gloved hands. Do not intentionally include vegetation in the portion of the sample that passes through the sieve unless this is a project specific requirement. Collect and weigh any portion unable to pass through the sieve.	NA.	Flagging criteria are not appropriate.	

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Soil grinding procedure	Initial demonstration.	The laboratory must initially demonstrate that the grinding procedure is capable of reducing the particle size to < 75 µm by passing representative portions of ground sample through a 200 mesh sieve (ASTM E11).	NA.	Flagging criteria are not appropriate.	
Soil grinding blank	Between each sample.	A grinding blank using clean solid matrix (such as Ottawa sand) must be prepared (e.g., ground and subsampled) and analyzed in the same manner as a field sample. Grinding blanks can be analyzed individually or composited. No target analytes detected greater than 1/2 Reporting Limit (RL).	All blank results must be reported and the affected samples must be flagged accordingly if blank criteria is not met.	If the composite grinding blank exceeds the acceptance criteria, apply B-flag to all samples associated with the grinding composite. If any individual grinding blank is found to exceed the acceptance criteria, apply B-flag to the sample following that blank.	
Soil subsampling process	Each sample, duplicate, and batch LCS.	Entire ground sample is mixed, spread out on a large flat surface (e.g., baking tray), and 30 or more randomly located increments are removed from the entire depth to sum a ~10 g subsample.	NA.	Flagging criteria are not appropriate.	
Soil sample triplicate	At the subsampling step, one sample per batch. Cannot be performed on any type of blank sample.	Three 10 g subsamples are taken from a sample expected to contain the highest levels of explosives within the Quantitation Range of the method. The RSD for results above the RL must not exceed 20%.	Corrective action must be taken if this criterion is not met (e.g., the grinding process should be investigated to ensure that the samples are being reduced to a sufficiently small particle size).	Apply J-flag if corrective action does not solve problem and no sample available.	

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Aqueous sample preparation	Each sample.	Solid phase extraction (SPE) using resin-based solid phase disks or cartridges is required.	NA.	Flagging criteria are not appropriate.	
Initial calibration (ICAL)	Minimum of 5 calibration standards with the lowest standard concentration at or below the RL. Once calibration curve or line is generated, the lowest calibration standard must be re-analyzed.	The apparent signal-to-noise ratio at the RL must be at least 5:1. If linear regression is used, $r \geq 0.995$. If using Internal Standardization, $RSD \leq 15\%$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	No samples can be run without a valid ICAL. Analysis by HPLC UV, LC/MS, or LC/MS/MS is allowed.
Second source calibration verification (ICV)	Following ICAL, prior to sample analysis.	All analyte(s) and surrogates within $\pm 20\%$ of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples should be run until calibration has been verified.
Continuing calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All target analytes and surrogates within $\pm 20\%$ of the expected value from the ICAL.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results should not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method blank	One per preparatory batch.	No analytes detected $> \frac{1}{2}$ RL and greater than $\frac{1}{10}$ the amount measured in any sample or $\frac{1}{10}$ the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
LCS containing all analytes to be reported	One per preparatory batch.	A solid reference material containing all reported analytes must be prepared (e.g., ground and subsampled) and analyzed in exactly the same manner as a field sample. Recoveries for the LCS must demonstrate the laboratory's ability to meet the project's MQOs.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix.	For matrix evaluation only, therefore is taken post grinding from same ground sample as parent subsample is taken. Percent recovery should be evaluated against LCS limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix.	For matrix evaluation only, therefore is taken post grinding from same ground sample as parent subsample is taken. Percent recovery should be evaluated against LCS limits and relative percent difference (RPD) < 20%.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Confirmation analysis	When target analytes are detected on the primary column using the UV Detector (HPLC) at concentrations exceeding the Limit of Detection (LOD).	Calibration and QC criteria are the same as for initial or primary column analysis. Results between primary and second column RPD \leq 40%.	Report from both columns.	If there is a > 40% RPD between the two column results, data must be P-flagged accordingly.	Confirmation analysis is not needed if LC/MS or LC/MS/MS was used for the primary analysis. Secondary column – Must be capable of resolving (separating) all of the analytes of interest and must have a different retention time order relative to the primary column. Any HPLC column used for confirmation analysis must be able to resolve and quantify all project analytes. Detection by HPLC UV, LC/MS or LC/MS/MS. Calibration and calibration verification acceptance criteria is the same as for the primary analysis.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

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Table 4, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 or 3 of this SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were all manual integrations signed
5. Were dilution factors applied correctly
6. Was there supervisory approval for manual integrations on standards and QC samples
7. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
8. If the data was entered into LIMS manually, was a check of all entered values performed
9. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
10. Were proper data qualifiers applied to the data in LIMS
11. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

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Table 5, ANALYST DATA REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: 8330 and 8330A

QA/QC Item	Yes	No	NA	Second Level Review
A. Initial Calibration				
1. Does the curve consist of at least five Calibration Standards?	_____	_____	_____	_____
2. Is the low RL standard in the calibration curve?	_____	_____	_____	_____
3. Are the % RSDs within QC limits for all analytes?	_____	_____	_____	_____
B. Second Source Verification				
1. Was the initial calibration curve verified by a second source calibration standard (ICV) and have QC criteria been met?	_____	_____	_____	_____
C. Continuing Calibration				
1. Are the Continuing Calibration Verification (CCV) standards analyzed every 20 samples and at the end of the sequence?	_____	_____	_____	_____
2. Are the % differences within QC limits for all analytes?	_____	_____	_____	_____
D. Sample Analysis				
1. Are all sample holding times met?	_____	_____	_____	_____
2. Was pH checked and recorded for all water samples?	_____	_____	_____	_____
3. Are all samples with concentrations > the highest standard used for initial calibration diluted and reanalyzed?	_____	_____	_____	_____
4. Are all compounds identified on the primary column confirmed on the secondary column?	_____	_____	_____	_____
5. Are Surrogate recoveries within QC limits?	_____	_____	_____	_____

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**ANALYST DATA REVIEW CHECKLIST
8330 and 8330A (Explosives)**

QA/QC Item	Yes	No	NA	Second Level Review
E. QC Samples				
1. Is the Method Blank extracted at the desired frequency and is its concentration for target analytes less than 1/2 the LOQ?	_____	_____	_____	_____
2. Is the Laboratory Control Sample and its percent recovery within QC limits?	_____	_____	_____	_____
3. Is the Matrix Spike/Matrix Spike Duplicate extracted at the desired frequency and is the percent recovery/RPD within QC limits?	_____	_____	_____	_____
F. Others				
1. Are all nonconformances included and noted?	_____	_____	_____	_____
2. Are all calculations checked at the minimum frequency?	_____	_____	_____	_____
3. Did analyst initial/date the appropriate printouts and report sheets?	_____	_____	_____	_____
4. Are all sample ID and units checked for transcription errors?	_____	_____	_____	_____
5. Are all manual integrations checked by a second reviewer to verify why they were performed?	_____	_____	_____	_____

Comments on any "No" response:

Analyst: _____

Second Level Review: _____

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Table 6, ANALYST DATA REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: 8330B

QA/QC Item	Yes	No	NA	Second Level Review
A. Initial Calibration				
1. Does the curve consist of at least five Calibration Standards?	_____	_____	_____	_____
2. Is the low RL standard in the calibration curve?	_____	_____	_____	_____
3. Are the % RSDs within QC limits for all analytes?	_____	_____	_____	_____
B. Second Source Verification				
1. Was the initial calibration curve verified by a second source calibration standard (ICV) and have QC criteria been met?	_____	_____	_____	_____
C. Continuing Calibration				
1. Are the Continuing Calibration Verification (CCV) standards analyzed every 20 samples and at the end of the sequence?	_____	_____	_____	_____
2. Are the % differences within QC limits for all analytes?	_____	_____	_____	_____
D. Sample Analysis				
1. Are all sample holding times met?	_____	_____	_____	_____
2. Was pH checked and recorded for all water samples?	_____	_____	_____	_____
3. Are all samples with concentrations > the highest standard used for initial calibration diluted and reanalyzed?	_____	_____	_____	_____
4. Are all compounds identified on the primary column confirmed on the secondary column?	_____	_____	_____	_____
5. Are Surrogate recoveries within QC limits?	_____	_____	_____	_____

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**ANALYST DATA REVIEW CHECKLIST
8330B (Explosives)**

QA/QC Item	Yes	No	NA	Second Level Review
E. QC Samples				
1. Is the Grinding Blank extracted at the desired frequency and is its concentration for target analytes less than ½ the LOQ?	_____	_____	_____	_____
2. Is the Method Blank extracted at the desired frequency and is its concentration for target analytes less than ½ the LOQ?	_____	_____	_____	_____
3. Is the Laboratory Control Sample and its percent recovery within QC limits?	_____	_____	_____	_____
4. Is the Matrix Spike/Matrix Spike Duplicate extracted at the desired frequency and is the percent recovery/RPD within QC limits?	_____	_____	_____	_____
5. Are the soils triplicates (DUP1/DUP2), if applicable, extracted at the desired frequency and is the RSD within QC limits?	_____	_____	_____	_____
F. Others				
1. Are all nonconformances included and noted?	_____	_____	_____	_____
2. Are all calculations checked at the minimum frequency?	_____	_____	_____	_____
3. Did analyst initial/date the appropriate printouts and report sheets?	_____	_____	_____	_____
4. Are all sample ID and units checked for transcription errors?	_____	_____	_____	_____
5. Are all manual integrations checked by a second reviewer to verify why they were performed?	_____	_____	_____	_____

Comments on any "No" response:

Analyst: _____

Second Level Review: _____



**LABORATORY
ACCREDITATION
BUREAU**

Certificate of Accreditation

ISO/IEC 17025:2005

Certificate Number L2226

Empirical Laboratories, LLC

621 Mainstream Drive, Suite 270
Nashville, TN 37228

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

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

Accreditation Granted through: November 30, 2012

A handwritten signature in black ink, appearing to read 'R.D.L.', positioned above a horizontal line.

**R. Douglas Leonard, Jr., Managing Director
Laboratory Accreditation Bureau
Presented the 30th of November 2009**

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

Scope of Accreditation For Empirical Laboratories, LLC

621 Mainstream Drive, Suite 270
Nashville, TN 37228
Marcia K. McGinnity
877-345-1113

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

Accreditation granted through: **November 30, 2012**

Testing - Environmental

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	EPA 8260B	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	EPA 8260B	1,1,2-Trichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	EPA 8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	EPA 8260B	1,1-Dichloropropene
GC/MS	EPA 8260B	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B	1,2,3-Trichloropropane
GC/MS	EPA 8260B	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260B	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260B	1,2-Dichlorobenzene
GC/MS	EPA 8260B	1,2-Dichloroethane (EDC)
GC/MS	EPA 8260B	1,2-Dichloropropane
GC/MS	EPA 8260B	1,3,5-Trimethylbenzene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B	1,3-Dichlorobenzene
GC/MS	EPA 8260B	1,3-Dichloropropane
GC/MS	EPA 8260B	1,4-Dichlorobenzene
GC/MS	EPA 8260B	1,4-Dioxane
GC/MS	EPA 8260B	1-Chlorohexane
GC/MS	EPA 8260B	2,2-Dichloropropane
GC/MS	EPA 8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	EPA 8260B	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B	2-Chlorotoluene
GC/MS	EPA 8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	EPA 8260B	4-Chlorotoluene
GC/MS	EPA 8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	EPA 8260B	Acetone
GC/MS	EPA 8260B	Acetonirile
GC/MS	EPA 8260B	Acrolein
GC/MS	EPA 8260B	Acrylonitrile
GC/MS	EPA 8260B	Allyl chloride
GC/MS	EPA 8260B	Benzene
GC/MS	EPA 8260B	Bromobenzene
GC/MS	EPA 8260B	Bromochloromethane
GC/MS	EPA 8260B	Bromodichloromethane
GC/MS	EPA 8260B	Bromoform
GC/MS	EPA 8260B	Bromomethane
GC/MS	EPA 8260B	Carbon Disulfide
GC/MS	EPA 8260B	Carbon Tetrachloride
GC/MS	EPA 8260B	Chlorobenzene
GC/MS	EPA 8260B	Chloroethane
GC/MS	EPA 8260B	Chloroform
GC/MS	EPA 8260B	Chloromethane
GC/MS	EPA 8260B	Chloroprene
GC/MS	EPA 8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	EPA 8260B	cis-1,3-Dichloropropene
GC/MS	EPA 8260B	cis-1,4-Dichloro-2-butene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B	Cyclohexane
GC/MS	EPA 8260B	Dibromochloromethane
GC/MS	EPA 8260B	Dibromomethane
GC/MS	EPA 8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	EPA 8260B	Di-isopropyl ether
GC/MS	EPA 8260B	ETBE
GC/MS	EPA 8260B	Ethyl methacrylate
GC/MS	EPA 8260B	Ethylbenzene
GC/MS	EPA 8260B	Hexachlorobutadiene
GC/MS	EPA 8260B	Hexane
GC/MS	EPA 8260B	Iodomethane
GC/MS	EPA 8260B	Isobutyl alcohol
GC/MS	EPA 8260B	Isopropylbenzene (Cumene)
GC/MS	EPA 8260B	Methacrylonitrile
GC/MS	EPA 8260B	Methyl Acetate
GC/MS	EPA 8260B	Methyl methacrylate
GC/MS	EPA 8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	EPA 8260B	Methylcyclohexane
GC/MS	EPA 8260B	Methylene Chloride, or Dichloromethane
GC/MS	EPA 8260B	Naphthalene
GC/MS	EPA 8260B	n-Butylbenzene
GC/MS	EPA 8260B	n-Propylbenzene
GC/MS	EPA 8260B	p-Isopropyltoluene
GC/MS	EPA 8260B	Propionitrile
GC/MS	EPA 8260B	sec-Butylbenzene
GC/MS	EPA 8260B	Styrene
GC/MS	EPA 8260B	t-Butyl alcohol
GC/MS	EPA 8260B	tert-Amyl methyl ether
GC/MS	EPA 8260B	tert-Butylbenzene
GC/MS	EPA 8260B	Tetrachloroethene (PCE; PERC)
GC/MS	EPA 8260B	Tetrahydrofuran
GC/MS	EPA 8260B	Toluene
GC/MS	EPA 8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260B	trans-1,3-Dichloropropene
GC/MS	EPA 8260B	trans-1,4-Dichloro-2-butene
GC/MS	EPA 8260B	Trichloroethene (TCE)
GC/MS	EPA 8260B	Trichlorofluoromethane (CFC-11)
GC/MS	EPA 8260B	Vinyl acetate
GC/MS	EPA 8260B	Vinyl Chloride (VC)
GC/MS	EPA 8260B	Xylenes (Total)
GC/MS	EPA 8270C/D	1,1'-Biphenyl
GC/MS	EPA 8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270C/D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/D	1,2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dioxane
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	EPA 8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	EPA 8270C/D	2,4-Dimethylphenol
GC/MS	EPA 8270C/D	2,4-Dinitrophenol
GC/MS	EPA 8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	EPA 8270C/D	2,6-Dichlorophenol
GC/MS	EPA 8270C/D	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol (o-Cresol)
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 8270C/D	2-Nitrophenol (ONP)
GC/MS	EPA 8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	EPA 8270C/D	3-Methylphenol

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 8270C/D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Methylphenol (p-Cresol)
GC/MS	EPA 8270C/D	4-Nitroaniline (PNA)
GC/MS	EPA 8270C/D	4-Nitrophenol (PNP)
GC/MS	EPA 8270C/D	7,12-Dimethylbenz(a)anthracene
GC/MS	EPA 8270C/D	Acenaphthene
GC/MS	EPA 8270C/D	Acenaphthylene
GC/MS	EPA 8270C/D	Acetaphenone
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 8270C/D	Benzdine
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)pyrene
GC/MS	EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270C/D	bis(2-Chloroethyl)ether (BCEE)
GC/MS	EPA 8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	EPA 8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	EPA 8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/D	Dibenzofuran (DBF)
GC/MS	EPA 8270C/D	Diethyl phthalate (DEP)
GC/MS	EPA 8270C/D	Dimethyl phthalate (DMP)
GC/MS	EPA 8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	EPA 8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	EPA 8270C/D	Fluoranthene
GC/MS	EPA 8270C/D	Fluorene
GC/MS	EPA 8270C/D	Hexachlorobenzene (HCB)
GC/MS	EPA 8270C/D	Hexachlorobutadiene (HCBD)
GC/MS	EPA 8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	EPA 8270C/D	Hexachloroethane (HCE)
GC/MS	EPA 8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Naphthalene
GC/MS	EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	N-Nitrosodimethylamine
GC/MS	EPA 8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	EPA 8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	EPA 8270C/D	Pentachlorophenol
GC/MS	EPA 8270C/D	Phenanthrene
GC/MS	EPA 8270C/D	Phenol
GC/MS	EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/ECD	EPA 8081A/B	4,4'-DDD
GC/ECD	EPA 8081A/B	4,4'-DDE
GC/ECD	EPA 8081A/B	4,4'-DDT
GC/ECD	EPA 8081A/B	Aldrin
GC/ECD	EPA 8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	EPA 8081A/B	alpha-Chlordane
GC/ECD	EPA 8081A/B	beta-BHC (beta-HCH)
GC/ECD	EPA 8081A/B	delta-BHC (delta-HCH)

Non-Potable Water		
Technology	Method	Analyte
GC/ECD	EPA 8081A/B	Dieldrin
GC/ECD	EPA 8081A/B	Endosulfan I
GC/ECD	EPA 8081A/B	Endosulfan II
GC/ECD	EPA 8081A/B	Endosulfan sulfate
GC/ECD	EPA 8081A/B	Endrin
GC/ECD	EPA 8081A/B	Endrin aldehyde
GC/ECD	EPA 8081A/B	Endrin ketone
GC/ECD	EPA 8081A/B	gamma-BHC (Lindane; gamma-HCH)
GC/ECD	EPA 8081A/B	gamma-Chlordane
GC/ECD	EPA 8081A/B	Heptachlor
GC/ECD	EPA 8081A/B	Heptachlor epoxide
GC/ECD	EPA 8081A/B	Methoxychlor
GC/ECD	EPA 8081A/B	Chlordane
GC/ECD	EPA 8081A/B	Toxaphene
GC/ECD	EPA 8082 /A	Aroclor-1016
GC/ECD	EPA 8082 /A	Aroclor-1221
GC/ECD	EPA 8082 /A	Aroclor-1232
GC/ECD	EPA 8082 /A	Aroclor-1242
GC/ECD	EPA 8082 /A	Aroclor-1248
GC/ECD	EPA 8082 /A	Aroclor-1254
GC/ECD	EPA 8082 /A	Aroclor-1260
GC/ECD	EPA 8082 /A	Aroclor-1262
GC/ECD	EPA 8082 /A	Aroclor-1268
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4,5-TP (Silvex)
GC/ECD	EPA 8151A	2,4-D
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichlorprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP (Mecoprop)

Non-Potable Water		
Technology	Method	Analyte
HPLC/UV	EPA 8330A/B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A/B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A/B	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	EPA 8330A/B	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	EPA 8330A/B	2,4-Dinitrotoluene (DNT)
HPLC/UV	EPA 8330A/B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Nitrotoluene (ONT)
HPLC/UV	EPA 8330A/B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/B	3-Nitrotoluene
HPLC/UV	EPA 8330A/B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A/B	4-Nitrotoluene (PNT)
HPLC/UV	EPA 8330A/B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	EPA 8330A/B	Nitrobenzene
HPLC/UV	EPA 8330A/B	Nitroglycerin
HPLC/UV	EPA 8330A/B	Nitroguanidine
HPLC/UV	EPA 8330A/B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	EPA 8330A/B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/B	PETN
GC/FID	FLPRO	Petroleum Range Organics
GC/FID	EPA 8015B	TPH DRO
GC/FID	EPA 8015B	TPH GRO
GC/FID	RSK-175	Methane
GC/FID	RSK-175	Ethane
GC/FID	RSK-175	Ethene
GC/ECD	EPA 8011	1,2-Dibromoethane (EDB)
GC/ECD	EPA 8011	1,2-Dibromo-3-chloropropane (DBCP)
HPLC/MS	EPA 6850	Perchlorate
ICP	EPA 6010B/C	Aluminum
ICP	EPA 6010B/C	Antimony
ICP	EPA 6010B/C	Arsenic
ICP	EPA 6010B/C	Barium
ICP	EPA 6010B/C	Beryllium

Non-Potable Water		
Technology	Method	Analyte
ICP	EPA 6010B/C	Boron
ICP	EPA 6010B/C	Cadmium
ICP	EPA 6010B/C	Calcium
ICP	EPA 6010B/C	Chromium, total
ICP	EPA 6010B/C	Cobalt
ICP	EPA 6010B/C	Copper
ICP	EPA 6010B/C	Iron
ICP	EPA 6010B/C	Lead
ICP	EPA 6010B/C	Magnesium
ICP	EPA 6010B/C	Manganese
CVAA	EPA 7470A	Mercury
ICP	EPA 6010B/C	Molybdenum
ICP	EPA 6010B/C	Nickel
ICP	EPA 6010B/C	Potassium
ICP	EPA 6010B/C	Selenium
ICP	EPA 6010B/C	Silver
ICP	EPA 6010B/C	Sodium
ICP	EPA 6010B/C	Strontium
ICP	EPA 6010B/C	Thallium
ICP	EPA 6010B/C	Tin
ICP	EPA 6010B/C	Titanium
ICP	EPA 6010B/C	Vanadium
ICP	EPA 6010B/C	Zinc
IC	EPA 300.0	Chloride
IC	EPA 300.0	Fluoride
IC	EPA 300.0	Nitrate
IC	EPA 300.0	Nitrite
IC	EPA 300.0	Sulfate
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate
IC	EPA 9056A	Nitrite
IC	EPA 9056A	Sulfate

Non-Potable Water		
Technology	Method	Analyte
Titration	SM 2320B 20 th /21 st edition	Alkalinity
Colorimetric	SM 4500 B, G, 20 th /21 st edition	Ammonia
Colorimetric	EPA 410.4	COD
UV/Vis	EPA 7196A	Hexavalent Chromium
Colorimetric	EPA 353.2	Nitrocellulose
Colorimetric	EPA 353.2	Nitrate/Nitrite
Gravimetric	EPA 1664A	O&G
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	SM 4500 S-2CF, 20 th /21 st edition	Sulfide
UV/Vis	SM 4500 P B5, E, 20 th /21 st edition	Total Phosphorus (as P)
UV/Vis	SM 4500 PE, 20 th /21 st edition	Ortho-Phosphate (as P)
TOC	9060A/SM5310C, 20 th /21 st edition	Total Organic Carbon
Gravimetric	SM 2540C, 20 th /21 st edition	TDS
Gravimetric	SM 2540D, 20 th /21 st edition	TSS
Colorimetric	EPA 9012A/B	Cyanide
Physical	EPA 1010A	Ignitability
Physical	EPA 9095B	Paint Filter
Probe	EPA 9040B/C	pH
Preparation	Method	Type
Preparation	EPA 1311	TCLP
Preparation	EPA 3005A	Metals digestion
Preparation	EPA 3010A	Metals digestion
Preparation	EPA 3510C	Organics Liquid Extraction
Preparation	EPA 5030A/B	Purge and Trap Water

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260B	1,1,1-Trichloroethane (1,1,1-TCA)

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260B	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	EPA 8260B	1,1,2-Trichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	EPA 8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	EPA 8260B	1,1-Dichloropropene
GC/MS	EPA 8260B	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B	1,2,3-Trichloropropane
GC/MS	EPA 8260B	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260B	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260B	1,2-Dichlorobenzene
GC/MS	EPA 8260B	1,2-Dichloroethane (EDC)
GC/MS	EPA 8260B	1,2-Dichloropropane
GC/MS	EPA 8260B	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B	1,3-Dichlorobenzene
GC/MS	EPA 8260B	1,3-Dichloropropane
GC/MS	EPA 8260B	1,4-Dichlorobenzene
GC/MS	EPA 8260B	1,4-Dioxane
GC/MS	EPA 8260B	2,2-Dichloropropane
GC/MS	EPA 8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	EPA 8260B	2-Chlorotoluene
GC/MS	EPA 8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	EPA 8260B	4-Chlorotoluene
GC/MS	EPA 8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	EPA 8260B	Acetone
GC/MS	EPA 8260B	Acetonitrile
GC/MS	EPA 8260B	Acrolein
GC/MS	EPA 8260B	Acrylonitrile
GC/MS	EPA 8260B	Allyl chloride

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260B	Benzene
GC/MS	EPA 8260B	Bromobenzene
GC/MS	EPA 8260B	Bromochloromethane
GC/MS	EPA 8260B	Bromodichloromethane
GC/MS	EPA 8260B	Bromoform
GC/MS	EPA 8260B	Bromomethane
GC/MS	EPA 8260B	Carbon Disulfide
GC/MS	EPA 8260B	Carbon Tetrachloride
GC/MS	EPA 8260B	Chlorobenzene
GC/MS	EPA 8260B	Chloroethane
GC/MS	EPA 8260B	Chloroform
GC/MS	EPA 8260B	Chloromethane
GC/MS	EPA 8260B	Chloroprene
GC/MS	EPA 8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	EPA 8260B	cis-1,3-Dichloropropene
GC/MS	EPA 8260B	cis-1,4-Dichloro-2-butene
GC/MS	EPA 8260B	Cyclohexane
GC/MS	EPA 8260B	Dibromochloromethane
GC/MS	EPA 8260B	Dibromomethane
GC/MS	EPA 8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	EPA 8260B	Ethyl methacrylate
GC/MS	EPA 8260B	Ethylbenzene
GC/MS	EPA 8260B	Hexachlorobutadiene
GC/MS	EPA 8260B	Hexane
GC/MS	EPA 8260B	Iodomethane
GC/MS	EPA 8260B	Isobutyl alcohol
GC/MS	EPA 8260B	Isopropylbenzene (Cumene)
GC/MS	EPA 8260B	Methacrylonitrile
GC/MS	EPA 8260B	Methyl Acetate
GC/MS	EPA 8260B	Methyl methacrylate
GC/MS	EPA 8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	EPA 8260B	Methylcyclohexane

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260B	Methylene Chloride, or Dichloromethane
GC/MS	EPA 8260B	Naphthalene
GC/MS	EPA 8260B	n-Butylbenzene
GC/MS	EPA 8260B	n-Propylbenzene
GC/MS	EPA 8260B	p-Isopropyltoluene
GC/MS	EPA 8260B	Propionitrile
GC/MS	EPA 8260B	sec-Butylbenzene
GC/MS	EPA 8260B	Styrene
GC/MS	EPA 8260B	tert-Butylbenzene
GC/MS	EPA 8260B	Tetrachloroethene (PCE; PERC)
GC/MS	EPA 8260B	Toluene
GC/MS	EPA 8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	EPA 8260B	trans-1,3-Dichloropropene
GC/MS	EPA 8260B	trans-1,4-Dichloro-2-butene
GC/MS	EPA 8260B	Trichloroethene (TCE)
GC/MS	EPA 8260B	Trichlorofluoromethane (CFC-11)
GC/MS	EPA 8260B	Vinyl acetate
GC/MS	EPA 8260B	Vinyl Chloride (VC)
GC/MS	EPA 8260B	Xylenes (Total)
GC/MS	EPA 8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	EPA 8270C/D	1,1'-Biphenyl
GC/MS	EPA 8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D	1,2,4-Trichlorobenzene
GC/MS	EPA 8270C/D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/D	1,2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dioxane
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D	2,4,6-Trichlorophenol (TCP)

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	EPA 8270C/D	2,4-Dimethylphenol
GC/MS	EPA 8270C/D	2,4-Dinitrophenol
GC/MS	EPA 8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	EPA 8270C/D	2,6-Dichlorophenol
GC/MS	EPA 8270C/D	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol (o-Cresol)
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 8270C/D	2-Nitrophenol (ONP)
GC/MS	EPA 8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	EPA 8270C/D	3-Methylphenol
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 8270C/D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Methylphenol (p-Cresol)
GC/MS	EPA 8270C/D	4-Nitroaniline (PNA)
GC/MS	EPA 8270C/D	4-Nitrophenol (PNP)
GC/MS	EPA 8270C/D	Acenaphthene
GC/MS	EPA 8270C/D	Acenaphthylene
GC/MS	EPA 8270C/D	Acetaphenone
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 8270C/D	Benzidine
GC/MS	EPA 8270C/D	Benzo(a)anthracene

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)pyrene
GC/MS	EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270C/D	bis(2-Chloroethyl)ether (BCEE)
GC/MS	EPA 8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	EPA 8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole
GC/MS	EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/D	Dibenzofuran (DBF)
GC/MS	EPA 8270C/D	Diethyl phthalate (DEP)
GC/MS	EPA 8270C/D	Dimethyl phthalate (DMP)
GC/MS	EPA 8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	EPA 8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	EPA 8270C/D	Fluoranthene
GC/MS	EPA 8270C/D	Fluorene
GC/MS	EPA 8270C/D	Hexachlorobenzene (HCB)
GC/MS	EPA 8270C/D	Hexachlorobutadiene (HCBD)
GC/MS	EPA 8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	EPA 8270C/D	Hexachloroethane (HCE)
GC/MS	EPA 8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Naphthalene
GC/MS	EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	N-Nitrosodimethylamine
GC/MS	EPA 8270C/D	N-Nitroso-di-n-propylamine (NDPA)

Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	EPA 8270C/D	Pentachlorophenol
GC/MS	EPA 8270C/D	Phenanthrene
GC/MS	EPA 8270C/D	Phenol
GC/MS	EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/ECD	EPA 8081A/B	4,4'-DDD
GC/ECD	EPA 8081A/B	4,4'-DDE
GC/ECD	EPA 8081A/B	4,4'-DDT
GC/ECD	EPA 8081A/B	Aldrin
GC/ECD	EPA 8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	EPA 8081A/B	alpha-Chlordane
GC/ECD	EPA 8081A/B	beta-BHC (beta-HCH)
GC/ECD	EPA 8081A/B	delta-BHC (delta-HCH)
GC/ECD	EPA 8081A/B	Chlordane
GC/ECD	EPA 8081A/B	Dieldrin
GC/ECD	EPA 8081A/B	Endosulfan I
GC/ECD	EPA 8081A/B	Endosulfan II
GC/ECD	EPA 8081A/B	Endosulfan sulfate
GC/ECD	EPA 8081A/B	Endrin
GC/ECD	EPA 8081A/B	Endrin aldehyde
GC/ECD	EPA 8081A/B	Endrin ketone
GC/ECD	EPA 8081A/B	gamma-BHC (Lindane; gamma-HCH)
GC/ECD	EPA 8081A/B	gamma-Chlordane
GC/ECD	EPA 8081A/B	Heptachlor
GC/ECD	EPA 8081A/B	Heptachlor epoxide
GC/ECD	EPA 8081A/B	Methoxychlor
GC/ECD	EPA 8081A/B	Toxaphene
GC/ECD	EPA 8082 /A	Aroclor-1016
GC/ECD	EPA 8082 /A	Aroclor-1221
GC/ECD	EPA 8082 /A	Aroclor-1232
GC/ECD	EPA 8082 /A	Aroclor-1242

Solid and Chemical Materials		
Technology	Method	Analyte
GC/ECD	EPA 8082 /A	Aroclor-1248
GC/ECD	EPA 8082 /A	Aroclor-1254
GC/ECD	EPA 8082 /A	Aroclor-1260
GC/ECD	EPA 8082 /A	Aroclor-1262
GC/ECD	EPA 8082 /A	Aroclor-1268
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4,5-TP (Silvex)
GC/ECD	EPA 8151A	2,4-D
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichlorprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP (Mecoprop)
HPLC/UV	EPA 8330A	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	EPA 8330A	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	EPA 8330A	2,4-Dinitrotoluene (DNT)
HPLC/UV	EPA 8330A	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A	2-Nitrotoluene (ONT)
HPLC/UV	EPA 8330A	3-Nitrotoluene
HPLC/UV	EPA 8330A	3,5-Dinitroaniline
HPLC/UV	EPA 8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A	4-Nitrotoluene (PNT)
HPLC/UV	EPA 8330A	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	EPA 8330A	Nitroglycerin
HPLC/UV	EPA 8330A	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	EPA 8330A	Nitrobenzene
HPLC/UV	EPA 8330A	Nitroguanidine

Solid and Chemical Materials		
Technology	Method	Analyte
HPLC/UV	EPA 8330A	PETN
HPLC/UV	EPA 8330B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330B	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	EPA 8330B	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	EPA 8330B	2,4-Dinitrotoluene (DNT)
HPLC/UV	EPA 8330B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330B	2-Nitrotoluene (ONT)
HPLC/UV	EPA 8330B	3-Nitrotoluene
HPLC/UV	EPA 8330B	3,5-Dinitroaniline
HPLC/UV	EPA 8330B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330B	4-Nitrotoluene (PNT)
HPLC/UV	EPA 8330B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	EPA 8330B	Nitroglycerin
HPLC/UV	EPA 8330B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	EPA 8330B	Nitrobenzene
HPLC/UV	EPA 8330B	Nitroguanidine
HPLC/UV	EPA 8330B	PETN
GC/FID	FLPRO	Petroleum Range Organics
GC/FID	EPA 8015B	TPH DRO
GC/FID	EPA 8015B	TPH GRO
HPLC/MS	EPA 6850	Perchlorate
ICP	EPA 6010B/C	Aluminum
ICP	EPA 6010B/C	Antimony
ICP	EPA 6010B/C	Arsenic
ICP	EPA 6010B/C	Barium
ICP	EPA 6010B/C	Beryllium
ICP	EPA 6010B/C	Boron
ICP	EPA 6010B/C	Cadmium
ICP	EPA 6010B/C	Calcium
ICP	EPA 6010B/C	Chromium, total

Solid and Chemical Materials		
Technology	Method	Analyte
ICP	EPA 6010B/C	Cobalt
ICP	EPA 6010B/C	Copper
ICP	EPA 6010B/C	Iron
ICP	EPA 6010B/C	Lead
ICP	EPA 6010B/C	Magnesium
ICP	EPA 6010B/C	Manganese
CVAA	EPA 7471A/B	Mercury
ICP	EPA 6010B/C	Molybdenum
ICP	EPA 6010B/C	Nickel
ICP	EPA 6010B/C	Potassium
ICP	EPA 6010B/C	Selenium
ICP	EPA 6010B/C	Silver
ICP	EPA 6010B/C	Sodium
ICP	EPA 6010B/C	Strontium
ICP	EPA 6010B/C	Tin
ICP	EPA 6010B/C	Titanium
ICP	EPA 6010B/C	Thallium
ICP	EPA 6010B/C	Vanadium
ICP	EPA 6010B/C	Zinc
UV/Vis	EPA 7196A	Hexavalent Chromium
TOC	Lloyd Kahn	Total Organic Carbon
Colorimetric	EPA 353.2	Nitrocellulose
Colorimetric	EPA 9012A/B	Cyanide
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	EPA 9034	Sulfide
Probe	EPA 9045C/D	pH
Preparation	Method	Type
Preparation	EPA 1311	TCLP
Preparation	EPA 1312	SPLP
Preparation	NJ Modified 3060A	Hexavalent Chromium
Preparation	EPA 3050B	Metals Digestion
Preparation	EPA 3546	Organics Microwave Extraction



Solid and Chemical Materials		
Technology	Method	Analyte
Preparation	EPA 3550B/C	Organics Sonication
Preparation	SM 2540B 20 th /21 st edition	Percent Solids (Percent Moisture)
Preparation	EPA 5035 /A	Purge and Trap Solid

Notes:

- 1) This laboratory offers commercial testing service.



Approved By: _____

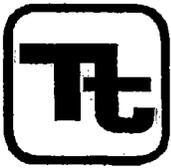
R. Douglas Leonard
Chief Technical Officer

Date: April 8, 2011

Issued: 11/30/09 Revised: 2/9/10 Revised: 3/31/10 Revised: 10/8/10 Revised: 1/25/11 Revised: 4/8/11

APPENDIX C

FIELD TASK MODIFICATION REQUEST FORM



TETRA TECH
FIELD TASK MODIFICATION REQUEST FORM

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

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

Activity Description: _____

Reason for Change: _____

Recommended Disposition: _____

Field Operations Leader (Signature) _____ Date _____

Approved Disposition: _____

Project/Task Order Manager (Signature) _____ Date _____

Distribution:
Program/Project File – _____
Project/Task Order Manager – _____
Field Operations Leader – _____
Other: _____

