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SITE INSPECTION (SI) WORK PLAN
MUNITIONS CONSTITUENTS AT THREE MUNITIONS RESPONSE SITES
NAVAL AIR STATION BRUNSWICK
BRUNSWICK, MAINE

JUNE 2009

Prepared for:

NAVAL FACILITIES ENGINEERING COMMAND MID-ATLANTIC (MIDLANT)
BRAC PMO Northeast
4911 South Broad Street
Philadelphia, PA 19112-1303

Prepared by:

MALCOLM PIRNIE, INC.
104 Corporate Park Drive
White Plains, NY 10602

and

TETRA TECH NUS, INC.
661 Andersen Drive, Foster Plaza #7
Pittsburgh, PA 15220-2745

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MUNITIONS CONSTITUENTS AT THREE MUNITIONS RESPONSE SITES

NAVAL AIR STATION BRUNSWICK (NASB)
BRUNSWICK, MAINE

NAVFAC Contract Number: N62472-02-D-1300 (Malcolm Pirnie)
N62472-03-D-0057 (Tetra Tech), CTO 69

Reviewed and Approved by:

Review Signatures:

Thomas E. Johnston 6/16/09
Tom Johnston
Tetra Tech NUS, Inc.
Quality Assurance Manager

Linda Klink 6/16/09
Linda Klink, P.E.
Tetra Tech NUS, Inc.
Project Manager

Approval Signatures:

Michael Daly
Environmental Protection Agency, Region I
Project Manager

Todd Bober 6-16-09
Todd Bober
Naval Facilities Engineering Command Mid-Atlantic
Remedial Project Manager

Malcolm Pirnie, Inc. prepared this document at the direction of Naval Facilities Engineering Command Atlantic. The draft was submitted by Malcolm Pirnie in February 2007, under the direction of Terri Akbas (QEP, Team Leader) and Jeffrey Bennett (P.E., BCEE, Vice President, Program Officer). Tetra Tech was tasked by the Navy to finalize this document. This document should be used only with the approval of Naval Facilities Engineering Command Atlantic. This report is based, in part, on information provided in other documents and is subject to the limitations and qualifications presented in the referenced documents.

JUNE 2009

As per the State of Maine Department of Professional and Financial Regulations, Title 32, Chapter 73, the undersigned has reviewed the geological content of the attached report entitled: "*Site Inspection (SI) Work Plan for Munitions Constituents at Three Munitions Response Sites, Naval Air Station Brunswick, Brunswick, Maine*", prepared by Malcolm Pirnie, Inc. and finalized by Tetra Tech NUS, Inc.

STATE OF MAINE
CHARLES
D.
RACE
243
CERTIFIED GEOLOGIST

6/16/09

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

Date

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ACRONYMS

Air Force	United States Air Force
APP	Accident Prevention Plan
BRAC	Base Realignment and Closure
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CG	Certified Geologist
CLEAN	Comprehensive Long-Term Environmental Action Navy
CRREL	Cold Regions Research and Engineering Laboratory
CSM	Conceptual Site Model
CTC	Cost to Complete
CTO	Contract Task Order
DMM	Discarded Military Munitions
DoD	Department of Defense
DQO	Data Quality Objectives
EOD	Explosive Ordnance Disposal
ERDC	Engineer Research and Development Center
GIS	Geographical Information System
GPS	Global Positioning System
HASP	Health and Safety Plan
Malcolm Pirnie	Malcolm Pirnie, Inc.
Marines	United States Marine Corp.
MC	Munitions Constituents
MEC	Munitions and Explosives of Concern
MEDEP	Maine Department of Environmental Protection
MRP	Munitions Response Program
MRP sites	Munitions Response Site
MSAD	Maine School Administration District
NASB	Naval Air Station Brunswick
NAVFAC	Naval Facilities Engineering Command
Navy	Department of the Navy
NDWS	National Drinking Water Standard
NFA	No Further Action
NOSSA	Naval Ordnance Safety & Security Activity
ORNL	Oak Ridge National Laboratory
PA	Preliminary Assessment
PAH	Polycyclic Aromatic Hydrocarbons

PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparability
PMO NE	Program Management Office Northeast
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RI	Remedial Investigation
RPM	Remedial Project Manager
SAGE	Semi-Automatic Ground Environment
SAP	Sampling and Analysis Plan
SDZ	Surface Danger Zone
SI	Site Inspection
SOW	Statement of Work
SVOCs	Semivolatile Organic Compounds
TAL	Target Analyte List
TOC	Total Organic Carbon
TtNUS	Tetra Tech NUS, Inc.
U.S.	United States
USEPA	United States Environmental Protection Agency
UXO	Unexploded Ordnance
UXOSS	Unexploded Ordnance Safety Officer
WWII	World War II
XRF	X-Ray Fluorescence

1.0 INTRODUCTION

Malcolm Pirnie, Inc. (Malcolm Pirnie) has prepared the following Work Plan for the Site Inspection (SI) of Three Munitions Response Program (MRP) sites at Naval Air Station Brunswick (NASB), Cumberland County, Maine (ME), under the Department of the Navy (Navy) Naval Facilities Engineering Command (NAVFAC) Atlantic, Contract Number N62472-02-D-1300, Task Order 0002 Modification 02. Tetra Tech NUS, Inc. (TtNUS or Tetra Tech) was tasked to finalize this document under the Comprehensive Long-Term Environmental Action Navy (CLEAN) Contract Number N62472-03-D-0057, Contract Task Order (CTO) 069.

This Final SI Work Plan has been developed to provide a description of the necessary tasks to complete this CTO and to ensure that the project deliverable will be in conformance with the NAVFAC, Mid-Atlantic Project Statement of Work (SOW), delivered 14 September 2006 to Malcolm Pirnie. In addition, this version of the Work Plan incorporates the ideas and resolutions generated during the development and review process for this project, including a site visit/scoping meeting conducted with Tetra Tech and the Stakeholders in October 2007, as well as additional discussions at routine project meetings, conference calls, and comment letters. For safety reasons and to provide a better basis for establishing munitions constituents (MC) sampling locations, the Navy has now decided to conduct MEC clearance before conducting the MC sampling at the munitions and explosives of concern (MEC) sites (Site 12 EOD, Quarry, and Former Munitions Bunker West). As a result, the subject Work Plan for all six sites has been revised to only include the remaining small arms ranges (Skeet Range, Topsham Skeet Range, and Machine Gun Boresight Range). This will allow field work at these small arms ranges to proceed in the meantime. A work plan for the three MEC sites will be issued at a later date, following evaluation of findings from the MEC clearance effort, and associated comments included herein will be addressed at that time.

This Final SI Work Plan will be used with the understanding that unanticipated conditions may dictate a change in the Plan as written. Any necessary deviations from the Work Plan will be brought to the attention of the NAVFAC, Mid-Atlantic Remedial Project Manager (RPM) as soon as possible before the deviation is implemented, and a written request for variance will be submitted to document the decision made. The Maine Department of Environmental Protection (MEDEP) and United States Environmental Protection Agency (USEPA) will be notified by the Navy of any deviations from the Work Plan.

1.1 PROJECT OBJECTIVES

The Department of Defense (DoD) has established policy and guidance for munitions response actions under the Military Munitions Response Program. Key program drivers developed to date conclude that

munitions response actions will be conducted under the process outlined in the National Contingency Plan, as authorized by the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA).

The objective of the SI described in this Final SI Work Plan is to conduct an on-site investigation and gather sufficient data to determine the presence or absence of MC that may remain from activities conducted by the DoD during operation of these sites and that may subsequently pose a threat to human health and/or the environment. Under the MRP, the primary goal of the SI is to collect the appropriate amount of information necessary to make one of the following decisions: 1) whether a Remedial Investigation (RI) is required at a site; 2) whether an immediate response is needed; or 3) whether the site qualifies for no further action (NFA).

The SI at NASB will address MC issues for the three MRP sites. The SI for MEC is being addressed under separate cover, in preparation by Tetra Tech. The secondary goal of the SI is to collect information for the Navy in order to budget MRP projects, including Cost to Complete (CTC) estimates and site prioritization for the MRP sites.

1.2 WP ORGANIZATION

This Work Plan consists of six sections, as outlined below:

Section 1.0: Introduction.
Section 2.0: Navy MRP Site Descriptions provides a detailed description of each MRP site.
Section 3.0: Scope of Work discusses the proposed activities to be conducted by Tetra Tech NUS, Inc. (TtNUS) as part of the SI.
Section 4.0: Health and Safety describes the health and safety procedures for the SI (refers to a Navy internal document).
Section 5.0: Project Management outlines the project schedule and project personnel for the SI.
Section 6.0: Project Deliverables presents a summary of the reporting to be completed for the SI.

The Sampling and Analysis Plan (SAP), which includes a Quality Assurance Project Plan (QAPP), is included in Appendix A. Results from the USEPA results of X-Ray Fluorescence (XRF) soil sampling at the Machine Gun Boresight Range are included in Appendix B. MC field activities at the MRP sites in this SI are limited to MC sampling; MEC is not expected to be present at these sites and a magnetometer assisted clearance of the ground surface for anomaly avoidance purposes will not be conducted; therefore, a Project Quality Control Plan and MEC Management and Contingency Plan are not applicable. Appendix C includes supplemental historical information for each of the sites collected and evaluated following the October 2007 kickoff meeting/site visit with the Stakeholders; also included are the

conceptual site models developed by Malcolm Pirnie from the Preliminary Assessments (PAs). Appendix D consists of Responses to Stakeholder comments.

2.0 MRP SITE DESCRIPTIONS

2.1 OVERVIEW

The NASB main base is located in Cumberland County, approximately 25 miles northeast of Portland, ME. The installation is located just south of U.S. Route 1, approximately two miles east of Brunswick's main business district and five miles inland from the Atlantic Ocean.

The NASB main base sits on approximately 3,070 acres. It is the last active duty DoD airfield remaining in the northeastern U.S. and has been included in the Base Realignment and Closure (BRAC) 2005 list, with an operational closure date of September 2011. The NASB is currently home to three active duty and two Reserve squadrons as well as 29 tenant commands. The installation is one of Maine's largest employers with over 4,800 military and civilian personnel. The NASB also provides support for other Navy units in Maine, including the Navy ships at Bath, the Navy Security Group at Winter Harbor, the U.S. Naval Radio Station at Cutler, the U.S. Naval Survival School at Rangeley, and the Department of Naval Sciences at the Maine Maritime Academy at Castine. Figure 2-1 shows the general layout of NASB main base, as well as the locations of the three MRP sites that are the focus of this Work Plan.

The installation was originally constructed and occupied in March 1943 with the primary mission of training British Naval Command (Royal Canadian Air Force) pilots. The station carried out a secondary mission of anti-submarine warfare during World War II (WWII). In October 1946, the installation was deactivated and the land and buildings were leased jointly to the University of Maine and Bowdoin College as annexes to ease the overcrowded conditions caused by the Government Issue Bill student influx. The University of Maine and Bowdoin College terminated their leases in 1949, and the station was taken over by the Brunswick Flying Service, a private company. Following this period of caretaker status, the station was selected by the Navy as a prime center for development. During the development period, the U.S. Air Force reached an agreement with the Navy authorizing the construction of an Air Force Control and Warning Facility on the station as a part of the continental circumferential radar screen.

The station was recommissioned in March 1951 as a Naval Air Facility with the mission of supporting three land-plane patrol squadrons and one fleet aircraft service squadron with a planned future mission as a master jet base. The station retained the mission of anti-submarine warfare. In December 1950, the Navy requested funds from Congress for the master jet project. This base modification required dual 8,000-foot runways and two outlying fields: one for gunnery and one for carrier practice landings. In addition, the Secretary of Defense submitted a request to Congress for funding in June 1951. This money was to be used for: additional barracks: officers' quarters: enlisted men's clubs: control tower:

storage: and communication buildings; and new galleys and mess facilities. The new buildings and facilities would make the station a permanent installation in Brunswick.

Following the reactivation period, several new permanent facilities were erected to replace the WWII "temporary" buildings. New facilities included a modern operations tower, three-deck barracks, and a large mess hall. In addition to these facilities, a new enlisted men's club, Navy Exchange, and Bachelor Officers' Quarters were constructed.

In December 1951, the Naval Air Facility was officially changed to the designation of Naval Air Station. The Arctic Survival Training School was established on-base September 1956 to train members deploying to the Arctic in north-country survival techniques.

Topsham Air Force Base, currently known as Topsham Annex, was initially developed by the U.S. Air Force (Air Force) in 1956 as an airspace surveillance and communications facility. Prior to 1956, the installation was occupied by a small dairy farm and woodland. The Air Force's mission for Topsham Air Force Base was to perform the mid-coast Maine segment of the Semi-Automatic Ground Environment (SAGE) system. SAGE was a networked radar and communications system that provided airspace surveillance of the U.S. and Canada during the 1960s. Topsham Air Force Base included the typical support infrastructure of a small military base: barracks, engineering, supply, recreation, administration, communication, etc. In the early 1960s, 51 additional buildings comprising 177 housing units were constructed. In the 1970s, Topsham Air Force Base was acquired by the Navy and placed under the operational control of NASB as a housing annex and space for support units; it was then renamed Topsham Annex. Portions of the Topsham Annex have been excessed by the Navy, with four parcels were transferred to the Maine School Administration District (MSAD) No. 75. The MSAD No. 75 property includes seven buildings, ball fields, roadways, and parking lots. The Topsham Annex Skeet Range overlaps Parcel 1 of the MSAD No. 75 property along its northern property boundary, and Parcels 2, 3, and 4 of the MSAD No. 75 property are located south of the skeet range.

2.2 MACHINE GUN BORESIGHT RANGE

The Machine Gun Boresight Range site is located in the eastern portion of the installation. The range site encompasses approximately 0.3 acres from the firing line to the berm with an approximately 1029.4 acre surface danger zone (SDZ) during historical operations.

The former range was used during the 1950s to align and test fire aircraft mounted guns. Naval aircraft of the 1950s would have fired machine gun ammunition including .30-caliber and .50-caliber. At the time the range was active, the area east of the range was undeveloped and wooded. The range is visible on a

1957 aerial photograph of the installation; however, today there are no visible remnants of the Machine Gun Boresight Range or the range berm.

An updated map believed to be from the late 1950s to early 1960s found at the National Archives had the range labeled as a pistol range; however, the exact history or use of the range as a pistol range is unknown. In the early 1960s, the layout of the installation was changed and eventually taxiways connecting the runways to the range area were removed and the range was abandoned.

Building 55 and the associated parking lot appear to sit on the approximate area of the compass rose and part of the former range floor; however the former location of the backstop for the range is not currently covered. Figure 2-2 shows the layout of the former Machine Gun Boresight Range.

The former Machine Gun Boresight Range is not suspected to contain MEC. It is assumed that all MEC onsite were removed when the berm was demolished; however, the ultimate disposition of the berm is unknown.

There is the potential for MC at the former Machine Gun Boresight Range. The primary MC of concern associated with machine gun and small arms ranges is lead. By design, the majority of the bullets fired at the range would have landed and accumulated in the berm. It is unknown if NASB has any procedures in place to remove and dispose of lead, as it accumulated on the range when it was active or when the range was closed. Sampling data was not available to estimate the potential for lead in the soil. Other metals of concern include antimony, arsenic, and copper. Perchlorate is also a contaminant of concern, every fifth round fired from machine guns was a tracer round which would have contained pyrotechnics.

2.3 SKEET RANGE

The Skeet Range site is located in the southeast portion of the installation in an open field approximately 75 meters north of and 100 meters east of Building 55. The former range was located adjacent to Range Road just northeast of the taxiway intersection.

The former Skeet Range was used for the training of military personnel during the 1950s. Navy Programming Guidance from the 1950s defined the SDZ of a skeet range as a 900-foot radius from the shooting field. The configuration of the range has changed over the years giving it an area of 78 acres. On range maps from 1952, the range is shown with the direction of fire to the north; however, in a 1957 aerial photograph, the range is shown with the direction of fire to the east. It appears that the layout of the range and direction of fire were changed to maintain a safe separation distance from the shooting field outlined in the Programming Guide because the area east of the range was undeveloped. Figure 2-3 shows the layout for the former Skeet Range.

Lead shot from expended shot gun ammunition is not considered MEC; therefore, the former Skeet Range is not suspected to contain MEC.

There is the potential for MC to be present at the former Skeet Range. The primary constituent of concern is lead from the shotgun ammunition and polycyclic aromatic hydrocarbons (PAHs) from the clay targets. It is unknown if the Navy performed any lead removal from the range when it was active or after it was abandoned. During the time the range was operational, lead was not considered an environmental or health hazard. Other associated MC, which are less likely to be of concern, may include antimony (increases hardness), arsenic (present in lead), nickel (coating on some shot), and lead azide (associated with gunpowder). Black powder was supplemented by smokeless powder early in the 20th century. Smokeless powder is the propellant associated with the use of shotguns and is so named because the combustion products are mainly gaseous compared to black powder. Propellants (nitrocellulose and possibly nitroglycerine) and plasticizers (dinitrotoluene and dibutyl phthalate) are associated with smokeless powder.

2.4. TOPSHAM ANNEX SKEET RANGE

The Topsham Annex Skeet Range is located in the northern portion of Topsham Annex along the Navy property line and east of the Mt. Ararat Middle School athletic field. The former 29-acre area was used during the Air Force's use of Topsham Annex prior to the 1970s. Navy Programming Guidance from the 1950s defined the SDZ of a skeet range as a 900-foot radius from the shooting field. Based on the visual survey conducted by Malcolm Pirnie and historical layout, it appears that the direction of fire was toward the north. The majority (27 acres) of the SDZ extends off-base onto private property and onto land transferred to the MSAD No. 75. No structures remain at the site, but clay target fragments were identified during the visual survey. The site is currently undeveloped, and future use will likely not change on the Navy owned property prior to transfer. The future use of the off-base portion of the range is unknown. The Navy will begin the process to secure right of entry for the private property so that field activities can be conducted, including soil and groundwater sampling. Figure 2-4 shows the layout for the Topsham Annex Skeet Range.

Lead shot from expended shot gun ammunition is not considered MEC; therefore, the Topsham Annex Skeet Range is not suspected to contain MEC.

There is the potential for MC to be present at the Topsham Annex Skeet Range. The primary constituents of concern are lead from shot and PAHs from the clay targets. As noted in Section 2.3, other MC associated with shotgun ammunition, which are less likely to be of concern, may include: antimony (increases hardness), arsenic (present in lead), nickel (coating on some shot), and lead azide (associated

with gunpowder). Propellants (nitrocellulose and possibly nitroglycerine) and plasticizers (dinitrotoluene and dibutyl phthalate) associated with smokeless powder may also be present.

Data from the July 2004 Investigative Activities indicated that lead is present in the surface soils. However, the soil samples were taken at the firing point and not within the likely shotfall area; therefore, the samples may not be representative of the entire Topsham Annex Skeet Range.

2.5 CONTAMINANTS OF CONCERN

MC are the chemical constituents that originate from UXO, DMM, or any other military munitions including explosives and non-explosive materials, and emissions, degradation, or breakdown elements of such munitions. Generally, a few categories of MC are: explosives and breakdown products (trinitrotoluene, cyclotrimethylenetrinitramine, and cyclotetramethylene-tetranitramine), pyrotechnics, propellants, incendiaries (perchlorate, nitroglycerine, nitrocellulose, nitrogyononium, white phosphorus), and metals [Target Analyte List (TAL) metals]. Table 2-1 summarizes the specific MC of concern at each of the MRP sites at NASB and Table 2-2 summarizes indicator parameters that will be collected to provide information on migration potential. Indicator parameters include total organic carbon (TOC) analysis for all sediment samples as an indicator to aid in toxicity evaluation. For soil, four soil samples each at the NASB Skeet Range and the Topsham Skeet Range will be analyzed for pH, TOC, and CEC.

Table 2-1: Analyses at Three MRP Sites

MRP Site	MC				
	TAL Metals	Explosives (Dinitrotoluene)	Propellants (Nitroglycerin)	Perchlorate	PAHs
Machine Gun Boresight Range	X		X	X	
NASB Skeet Range	X	X	X		X
Topsham Annex Skeet Range	X	X	X		X
MRP Site	**Other Analysis**				
	Nitrates	pH	TOC	CEC	
Machine Gun Boresight Range					
NASB Skeet Range	X	X	X	X	
Topsham Annex Skeet Range	X	X	X	X	

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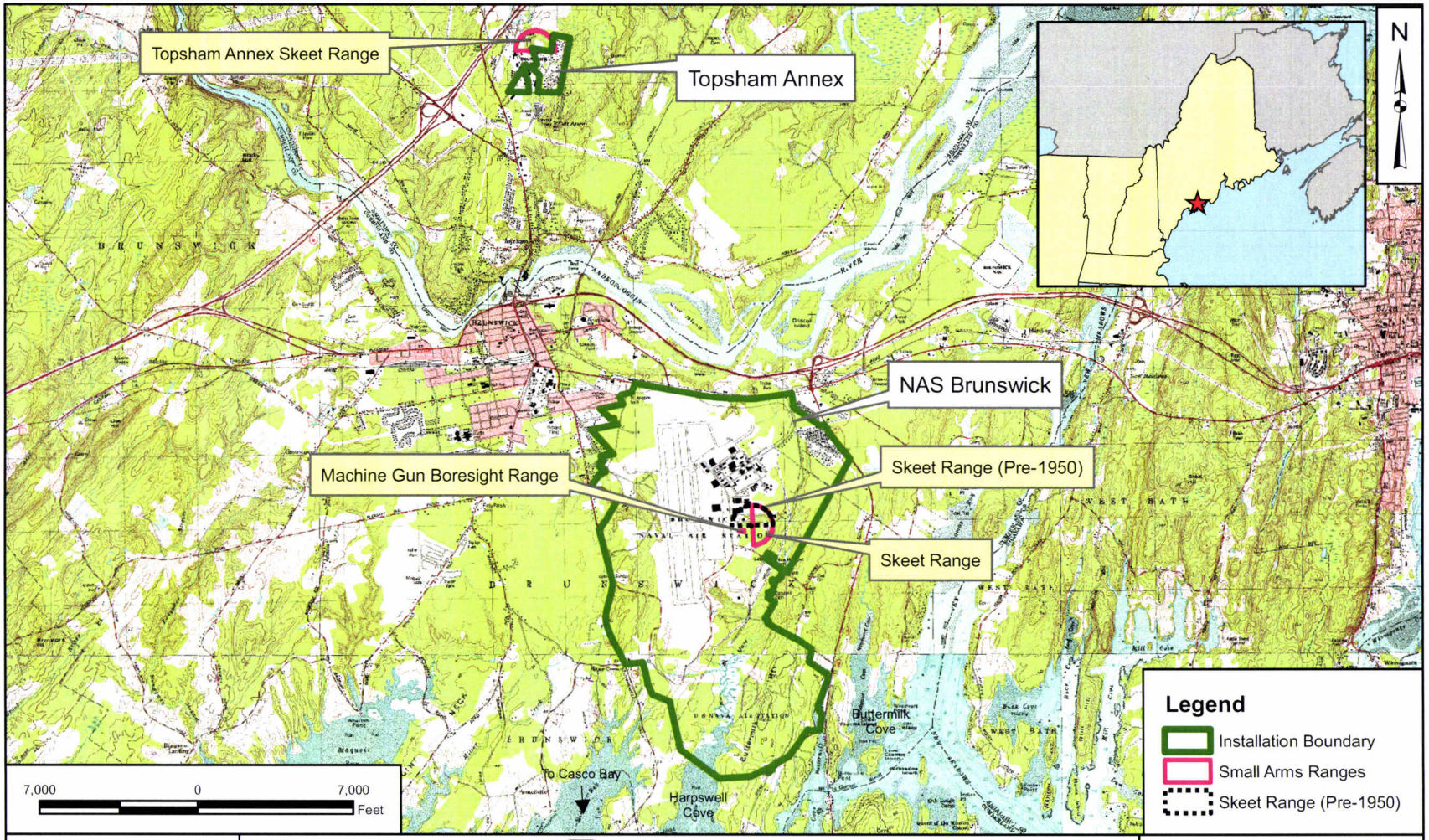

DRAWN BY J. ENGLISH	DATE 06/12/09	Tetra Tech NUS, Inc.	CONTRACT NUMBER N62472-02-D-1300/N62472-03-D-0057
CHECKED BY L. KLINK	DATE 06/12/09	SITE LOCATION MAP NAVAL AIR STATION BRUNSWICK, MAINE	APPROVED BY ___ DATE ___
REVISED BY ___	DATE ___		APPROVED BY ___ DATE ___
SCALE AS NOTED			FIGURE NO. FIGURE 2-1 / REV 0

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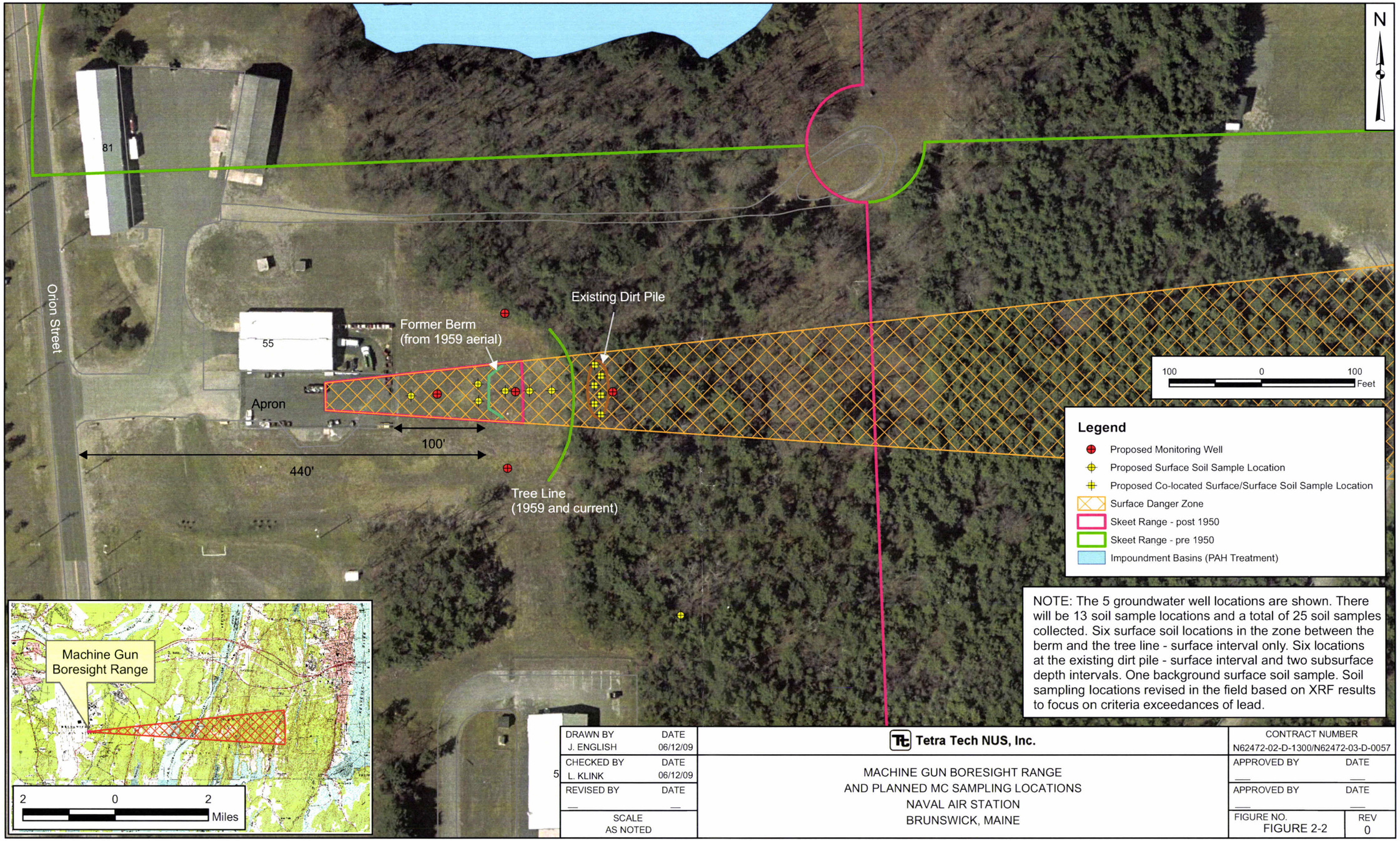
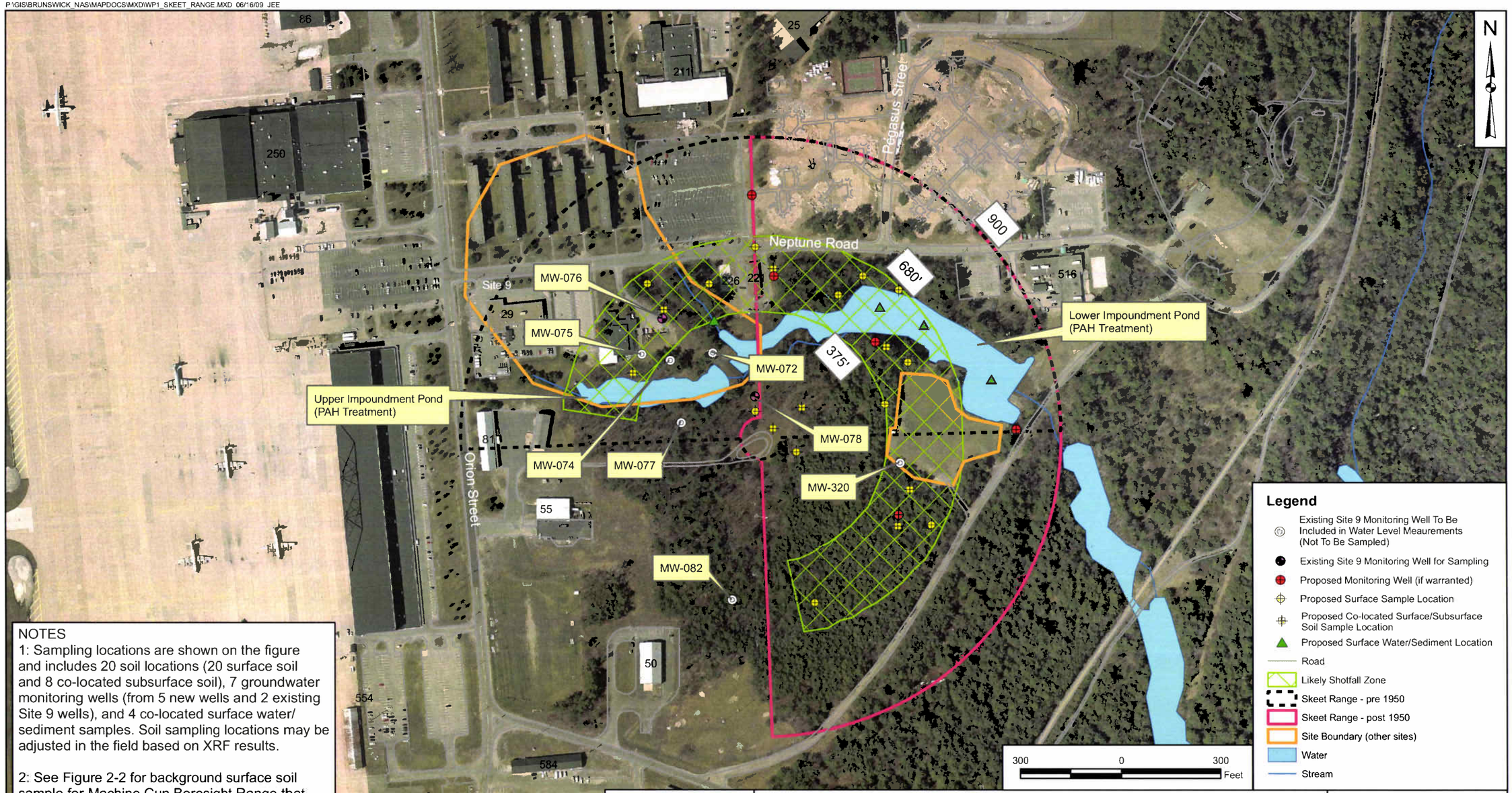


NOTE: The 5 groundwater well locations are shown. There will be 13 soil sample locations and a total of 25 soil samples collected. Six surface soil locations in the zone between the berm and the tree line - surface interval only. Six locations at the existing dirt pile - surface interval and two subsurface depth intervals. One background surface soil sample. Soil sampling locations revised in the field based on XRF results to focus on criteria exceedances of lead.

DRAWN BY J. ENGLISH	DATE 06/12/09	Tetra Tech NUS, Inc.	CONTRACT NUMBER N62472-02-D-1300/N62472-03-D-0057
CHECKED BY L. KLINK	DATE 06/12/09	MACHINE GUN BORESIGHT RANGE AND PLANNED MC SAMPLING LOCATIONS NAVAL AIR STATION BRUNSWICK, MAINE	APPROVED BY — DATE —
REVISED BY —	DATE —		APPROVED BY — DATE —
SCALE AS NOTED			FIGURE NO. FIGURE 2-2 / REV 0

P:\GIS\BRUNSWICK_NAS\MAPDOCS\MXD\WP1_SKEET_RANGE.MXD 06/16/09 JEE



NOTES

1: Sampling locations are shown on the figure and includes 20 soil locations (20 surface soil and 8 co-located subsurface soil), 7 groundwater monitoring wells (from 5 new wells and 2 existing Site 9 wells), and 4 co-located surface water/ sediment samples. Soil sampling locations may be adjusted in the field based on XRF results.

2: See Figure 2-2 for background surface soil sample for Machine Gun Boresight Range that will also serve as background for NASB Skeet Range.

3: The need for monitoring well installation will be discussed with the EPA and MEDEP after evaluating soil XRF results.

DRAWN BY J. ENGLISH	DATE 06/12/09	Tetra Tech NUS, Inc.	CONTRACT NUMBER N62472-02-D-1300/N62472-03-D-0057
CHECKED BY L. KLINK	DATE 06/16/09	SKEET RANGE AND PLANNED MC SAMPLING LOCATIONS NAVAL AIR STATION BRUNSWICK, MAINE	APPROVED BY — DATE —
REVISED BY —	DATE —		APPROVED BY — DATE —
SCALE AS NOTED			FIGURE NO. FIGURE 2-3 / REV 0

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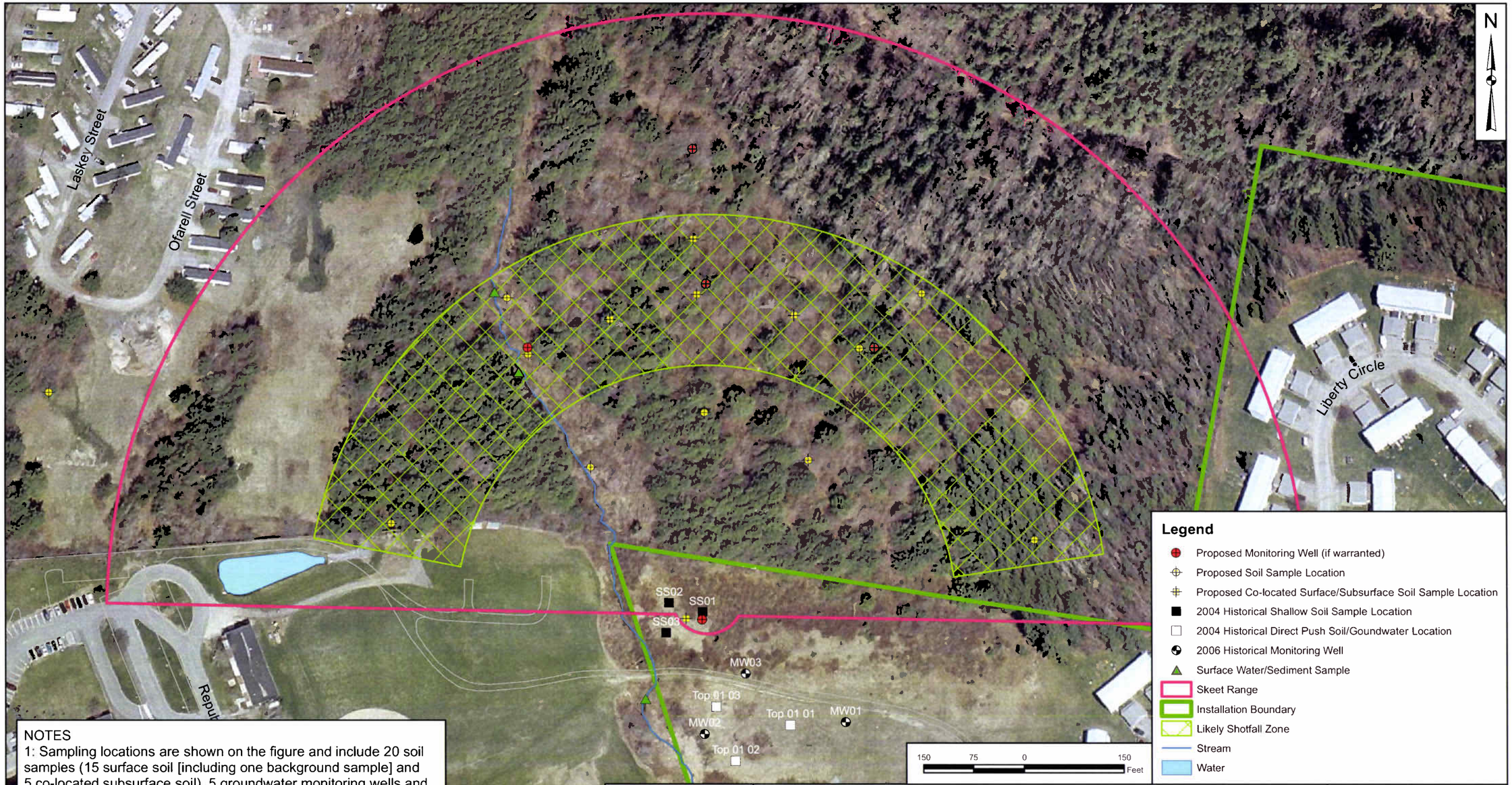

NOTES
1: Sampling locations are shown on the figure and include 20 soil samples (15 surface soil [including one background sample] and 5 co-located subsurface soil), 5 groundwater monitoring wells and 3 surface water/sediment samples, including one location upstream. Soil sampling locations may be revised in the field based on XRF results.

2: The need for monitoring well installation will be discussed with the EPA and MEDEP after evaluating soil XRF results.

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CHECKED BY L. KLINK	DATE 06/15/09	TOPSHAM ANNEX SKEET RANGE AND PLANNED MC SAMPLING LOCATIONS NAVAL AIR STATION BRUNSWICK, MAINE	APPROVED BY — DATE —
REVISED BY —	DATE —		APPROVED BY — DATE —
SCALE AS NOTED			FIGURE NO. FIGURE 2-4 / REV 0

3.0 SCOPE OF WORK

The scope of the SI will be based on DoD guidance for performing response actions on military ranges and *EPA [Environmental Protection Agency] Guidance for Performing Site Inspections Under Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)*; Interim Final, September 1992, as well as U.S. Army Corps of Engineers guidance on ordnance and explosive response actions under the Defense Environmental Restoration Program for Formerly Used Defense Sites. The MRP SI includes the following inter-related tasks:

Preliminary Assessment (PA) – This task consists of identifying data gaps from the range inventory, obtaining and reviewing historical records, and conducting a site visit to assess the current conditions of the MRP sites.

SI Work Plan – This task consists of preparing and submitting a site-specific SI Work Plan (including a SAP). The SI Work Plan will identify project objectives and the data collection program designed to meet these objectives.

Stakeholder Meetings – A meeting with Navy and regulatory stakeholders was held in October 2007 prior to the SI fieldwork to discuss the historical site information review findings, the proposed data collection/evaluation objectives and approach presented in the SI Work Plan, and the criteria for NFA or additional action determinations. Subsequent revisions resulted from additional discussions at routine project meetings, conference calls, and comment letters. As discussed in Section 1.0, the subject Work Plan for all six sites has been revised to only include the remaining small arms ranges (Skeet Range, Topsham Skeet Range, and Machine Gun Boresight Range).

SI Fieldwork – This consists of performing investigative activities and preparing reports of findings.

SI Report – This task consists of preparing and submitting an SI Report summarizing the results of the fieldwork, including an updated Conceptual Site Model (CSM) developed for each site with an appendix containing all information necessary to complete the MRP Site Prioritization Protocol.

3.1 PA

PAs were conducted at the three MRP sites to support the SI. All PAs provided information pertinent to identifying, verifying, and establishing the physical limits and potential for MEC and MC at each site. Historical records, aerial photographs, existing site maps, and existing environmental restoration documents were reviewed, and interviews with installation personnel were conducted. Available existing

installation-specific background studies were reviewed, and a site visit was conducted to gather information regarding current site conditions, including the possible presence of MEC and/or MC at each site.

The Final PA Report for the Topsham Annex was submitted in September 2005. The Final PA Report for the Machine Gun Boresight Range and the Skeet Range (which also included the Former Munitions Bunker West Area) was submitted in September 2006. Comments from the BRAC Program Management Office Northeast (PMO NE) and other stakeholders were incorporated into the Final PA Reports.

3.2 STAKEHOLDER MEETINGS

A Stakeholder Meeting was held in October 2007 and involved the Navy and regulatory stakeholders, and provided an opportunity to discuss project objectives, activities, and findings. The Stakeholder meeting was held prior to the SI field work. The meeting also provided an opportunity for stakeholders to discuss questions and make decisions regarding the MRP sites. The Stakeholder Meeting provides an integrated approach to planning and documenting that will produce the type and quality of data needed for site-specific decision making. The decisions made during the Stakeholder Meeting confirmed and refined the scope of MC field activities for the three MRP sites (Machine Gun Boresight Range, Skeet Range, and Topsham Annex Skeet Range). Subsequent revisions resulted from additional discussions at routine project meetings, conference calls, and comment letters. As discussed in Section 1.0, the subject Work Plan for all six sites has been revised to only include the remaining small arms ranges (Skeet Range, Topsham Skeet Range, and Machine Gun Boresight Range).

3.3 SI FIELDWORK

Based on information obtained during the PA, MEC investigations will not be performed at these MRP sites. The Machine Gun Boresight Range, the NASB Skeet Range, and the Topsham Annex Skeet Range are not suspected to contain MEC.

Field sampling will be conducted at all three sites and will include the collection of sufficient information to show whether MC are present at these MRP sites at NASB. The MRP site locations are provided on Map 2-1 through Map 2-4.

3.3.1 MC Field and Data Evaluation Activities

Table 3-1 provides a summary of decisions made to address MC.

Table 3-1: Summary of MC SI Activities		
MRP sites	**MC SI Activities**	
	Activity	**Purpose**
Machine Gun Boresight Range	Conduct XRF investigation (USEPA) to aid in establishing the former berm location (see Appendix B) and, in particular, if the nearby dirt pile is remnants of the former berm, and thereby establish locations for MC sampling. Map dirt pile via GPS. Collect 13 discrete surface soil samples (includes 1 background location to be analyzed for TAL metals and PAHs) and analyze for TAL metals and propellants (nitroglycerine). Collect 12 discrete subsurface soil samples and analyze for TAL metals and propellants (nitroglycerine). Collect 5 groundwater samples and analyze for TAL metals, propellants (nitroglycerine), and perchlorate.	CTC and site prioritization. Support MC NFA or RI determination. NFA determination is possible if sample analytical data for site are below analyte-specific screening criteria: Soil: Oak Ridge National Laboratory (ORNL) Regional residential soil screening levels (September 2008) Groundwater: The more stringent of ORNL Regional screening levels for tap water and Maine MEGs
NASB Skeet Range	Conduct XRF investigation (TtNUS), discrete samples, to aid in establishing locations for MC sampling. Collect 20 discrete surface soil samples and analyze for TAL metals and PAHs, analyze the 4 samples collected in and around the firing point for explosives (dinitrotoluene) and propellants (nitroglycerine), and 4 of the samples for pH, TOC, and CEC. Collect 8 discrete subsurface soil samples and analyze for TAL metals and PAHs, analyze the 2 samples collected around the firing point for explosives (dinitrotoluene) and propellants (nitroglycerine). Collect 4 sediment samples (1 discrete from the tributary and 3 composite from the impoundment ponds) and analyze for TAL metals and TOC (no PAHs). If decided upon, collect 7 groundwater samples (5 new locations plus 2 existing SWMU 9 wells) and analyze for TAL metals, PAHs, and nitrates. The groundwater sample collected from MW-NASB-078 will be analyzed for explosives (dinitrotoluene) and propellants (nitroglycerine). Collect 4 surface water samples (1 discrete from the tributary and 3 composite from the impoundment ponds) and analyze for TAL metals (no PAHs).	CTC and site prioritization. Support MC NFA or RI determination. NFA determination is possible if sample analytical data for site are below analyte-specific screening criteria: Soil: ORNL Regional screening levels Groundwater: The more stringent of ORNL Regional screening levels for tap water and Maine MEGs Surface Water/Sediment: Criteria established previously for other NAS Brunswick IR sites (see QAPP, which is Attachment 1 of SAP).

Table 3-1: Summary of MC SI Activities		
MRP sites	**MC SI Activities**	
	Activity	**Purpose**
Topsham Annex Skeet Range	Conduct XRF investigation (TtNUS), discrete samples, to aid in establishing the site boundaries and locations for MC sampling. Map any historic site features. Collect 15 discrete surface soil samples (includes 1 background location to be analyzed for TAL metals and PAHs only) and analyze for TAL metals and PAHs, analyze the 4 samples collected in and around the firing point for explosives (dinitrotoluene) and propellants (nitroglycerine), and 4 of the samples for pH, TOC, and CEC. Collect 5 discrete subsurface soil samples and analyze for TAL metals and PAHs, analyze the sample collected around the firing point for explosives (dinitrotoluene) and propellants (nitroglycerine). Collect 3 discrete sediment samples (includes 1 upstream background sample) and analyze for TAL metals, PAHs, and TOC. If decided upon, collect 5 groundwater samples and analyze for TAL metals, PAHs, and nitrates. One groundwater sample will be analyzed for explosives (dinitrotoluene) and propellants (nitroglycerin). Collect 3 discrete surface water samples (includes 1 upstream background sample) and analyze for TAL metals and PAHs.	CTC and site prioritization. Support MC NFA or RI determination. NFA determination is possible if sample analytical data for site are below analyte-specific screening criteria: Soil: ORNL Regional screening levels Groundwater: The more stringent of ORNL Regional screening levels for tap water and Maine MEGs Surface Water/Sediment: Criteria established previously for other NAS Brunswick IR sites (see QAPP, which is Attachment 1 of SAP).

Collection of discrete grab samples is proposed for surface and subsurface soil samples at these sites. Sediment and surface water samples will be obtained as discrete grab samples.

The goal of the field sampling activities for MC is to determine if the site has been impacted by MC. Analytical data exceeding background levels and appropriate regulatory limits will be used as justification to move the site into the RI phase. The SI field sampling activities are not intended to determine the nature and extent of all contaminants. All fieldwork will be of sufficient quality to meet the DQOs for the project as dictated in the QAPP. The details of the planned MC field sampling activities are provided in the SAP. Investigative activities will be conducted based on information obtained during the PA process and data review. Complete sampling and analysis of relevant media will be conducted to assess the risk/hazard posed by any MC found at the site.

3.3.1.1 Soil/Sediment Sampling

Soil samples will be collected at all of the MRP sites. Up to 25 soil discrete samples (12 surface and 12 subsurface soil samples plus 1 background surface soil sample) will be collected from the Machine Gun Boresight Range (analyzed for TAL metals and propellants); 20 discrete soil samples (14 surface and 5 subsurface soil samples plus 1 background surface soil sample) will be collected from the Topsham Annex Skeet Range (analyzed for TAL metals and PAHs) the 4 samples collected from in and around the firing point will also be analyzed for explosives (dinitrotoluene), and propellants (nitroglycerine); and 28 discrete soil samples (20 surface and 8 subsurface soil samples; note the common 1 background surface soil sample is included in the count for Machine Gun Boresight Range) will be collected from the NASB Skeet Range (analyzed for TAL metals and PAHs) and the 4 samples collected from in and around the firing point will also be analyzed for explosives (dinitrotoluene) and propellants (nitroglycerine). Four soil samples each from both the Topsham Annex Skeet Range and the NASB Skeet Range will also be analyzed for pH, TOC, and CEC. The tabulation of soil samples at the Machine Gun Boresight Range includes 1 surface soil sample collected off site for comparison purposes with background conditions. The background soil samples will be collected at locations shown on the figures; however, if there is evidence of fill or other historic soil disturbance in the area, the background samples will be relocated.

Two discrete sediment samples will be collected from the Topsham Annex Skeet Range brook within the shotfall zone, and an additional discrete background sample will be collected upstream; all samples will be analyzed for PAHs and TAL metals. Four sediment samples will be collected associated with the PAH detention basins at the other Skeet Range and analyzed for TAL metals (no PAHs). The three samples from within the PAH detention basin will be composite samples and the sample from the tributary near the detention basin will be a discrete sample. All sediment samples will be analyzed for total organic carbon (TOC). Sediment samples will not be collected at the Machine Gun Boresight Range because no surface water bodies or ditches are present.

3.3.1.2 Surface Water/Groundwater Sampling and Water-Level Measurements

Provided that surface water is present during the sampling event, three discrete surface water samples (two within the shotfall zone and one background upstream) will be collected at the Topsham Annex Skeet Range brook and will be analyzed for TAL metals and PAHs. At the other Skeet Range, the three samples from within the PAH detention basin will be composite samples and the sample from the tributary near the detention basin will be a discrete sample. The surface water samples will be analyzed for TAL metals. Surface water samples will not be collected at the Machine Gun Boresight Range where no surface water bodies or ditches are present.

Temporary monitoring wells may be installed at Topsham Skeet Range. The Navy will discuss potential groundwater sampling requirements and locations with the EPA and MEDEP after conducting site reconnaissance and soil sampling activities. Should temporary monitoring well be installed, one groundwater sample will be collected from each of the 5 temporary wells.. These groundwater samples will be analyzed for TAL metals, PAHs, and nitrates. One groundwater sample at the firing point will be analyzed for explosives (dinitrotoluene) and propellants (nitroglycerin). If monitoring wells are installed, it is proposed that one well will be installed at the firing point, 3 will be installed within the likely shotfall zone, and 1 will be installed surrounding the target area outside of the shotfall zone. The well outside of the shotfall zone will aid in determining the extent of contamination, if any, beyond the shotfall zone.

Temporary monitoring wells may be installed at the NASB Skeet Range. The Navy will discuss potential groundwater sampling requirements and locations with the EPA and MEDEP after conducting site reconnaissance and soil sampling activities. Should temporary monitoring wells be installed, one groundwater sample will be collected from each of the five temporary wells. In addition, groundwater from two existing wells installed for the Site 9 investigation will be sampled because they are properly placed to aid in the Skeet Range investigation - one groundwater sample will be collected from the existing well MW-NASB-078, serving for the firing point well, and the other groundwater sample will be collected from existing well MW-NASB-076, located within the shotfall zone. These groundwater samples will be analyzed for TAL metals, PAHs, and nitrates. The groundwater sample collected from MW-NASB-078 will also be analyzed from explosives (dinitrotoluene) and propellants (nitroglycerin).

One groundwater sample will be collected from each of the five temporary groundwater wells that will be installed at the Machine Gun Boresight Range during field activities, for a total of five groundwater samples. These samples will be analyzed for TAL metals, propellants, and perchlorate. The five temporary groundwater wells will be installed at the Machine Gun Boresight Range at the following locations; one at the firing point and the other 4 positioned one in each direction surrounding the berm.

Synoptic water-level data will be collected from each well sampled to aid in establishing groundwater flow direction for each site. If monitoring well installation is decided upon at the Skeet Range, Site 9 monitoring wells MW-NASN-072, -074, -075, -076 (to be sampled also), -077, -078 (to be sampled also), -082 and -320 will be included in the water level data collection effort. If monitoring well installation is decided upon at Topsham Skeet Range, three existing nearby wells (MW1, MW2, and MW3) are present from a 2006 investigation of an adjacent area and will be included in the water-level data collection effort.

3.3.1.3 Laboratory Analysis

The total number of samples that will be collected and the selected laboratory analyses are presented in Table 3-2. The analytical methods were selected on the basis of the munitions items known to have been

used at the site and include a standard suite of range-related analytical parameters to account for unknown items. The standard analytical methods include TAL Metals (USEPA Method 6010B), Explosives (SW-846 Method 8330A for dinitrotoluene), Perchlorate (SW-846 Method 6850 [with filtration at the laboratory]) PAHs (SW-846 Method SIM 8270C), TOC (Lloyd Kahn Method), pH (SW-846 Method 9045D), CEC (SW-846 Method 9081), nitrates (SW-846 Method 9056 or EPA 300) and propellants (nitroglycerin by SW-846 Method 8332). For the skeet ranges, nitrocellulose and dibutyl phthalate are not included because they are secondary contaminants with low toxicity and are not expected at significant concentrations; however, if propellants and explosives are found at levels of concern during the MC SI, dibutyl phthalate will be included in the analytical suite in the future during an RI and/or remediation.

Table 3-2: Sample Summary Table

MRP Site	Number of Samples / Media					
	TAL Metals	Explosives (Dinitrotoluene)	Propellants (Nitroglycerin)	Perchlorate	PAHs	Nitrates
Machine Gun Boresight Range	13 surface soil (includes 1 background sample) 12 subsurface soil 5 groundwater		13 surface soil 12 subsurface soil 5 groundwater	5 groundwater	1 surface soil (background sample)	
NASB Skeet Range	20 surface soil* 8 subsurface soil 7 groundwater (5 from new wells and 2 from existing wells) 4 surface water 4 sediment*	4 surface soil 2 subsurface soil 1 groundwater (MW-NASB-078)	4 surface soil 2 subsurface soil 1 groundwater (MW-NASB-078)		20 surface soil 8 subsurface soil 7 groundwater**	7 groundwater
Topsham Annex Skeet Range	15 surface soil* (includes 1 background sample) 5 subsurface soil 3 sediment * 3 surface water (includes 1 upstream collocated sample) 5 groundwater	4 surface soil 1 subsurface soil 1 groundwater	4 surface soil 1 subsurface soil 1 groundwater		15 surface soil 5 subsurface soil 3 sediment 3 surface water 5 groundwater** 1 surface water co-located sediment (background sample)	5 groundwater

Total Organic Carbon (TOC) analysis will be conducted for all sediment samples as an indicator parameter to aid in toxicity evaluation of TAL metals. Four soil samples each from NASB Skeet Range and Topsham Annex Skeet Range will be analyzed for pH, TOC, and CEC.

** Temporary monitoring may be installed. The Navy will discuss potential groundwater sampling requirements and locations with the EPA and MEDEP after conducting site reconnaissance and soil sampling activities.

Note: Sample quantities presented do not include QC samples. For details on QC samples, refer to Table 4-2 through 4-4 of the SAP.

3.3.1.4 Chemistry Analysis

DQOs for sampling, analysis, and QA/QC will be reached by collecting the proper quantities and types of samples, using the correct analytical methodologies, implementing field and laboratory QA/QC procedures, and using various data validation and evaluation processes. The DQOs for each analytical method are provided in the QAPP. Laboratory requirements for the analytical methods being used for this project are provided in the SAP and QAPP. These procedures include requirements for sample preparation, sampling containers, preservation methods, and holding times.

The QAPP has been developed to support the sampling, analysis, and evaluation activities associated with this project. The QAPP consists of policies, procedures, specifications, standards, and documentation sufficient to produce data of quality adequate to meet the DQOs for the project.

The QAPP has been prepared to ensure that this responsibility is met throughout the duration of this project. It addresses procedures to assure the PARCC of field and laboratory data generated during the course of this project. It also provides a framework for evaluating existing data that may be used in this project. The QAPP defines the first stage of the QA requirements for sample and data acquisition, handling, and assessment.

QA procedures such as tracking, reviewing, and auditing are implemented as necessary to ensure that all project work is performed in accordance with professional standards, USEPA and Navy regulations and guidelines, and the specific goals and requirements stated in this Work Plan.

QC of sample collection, analysis, and assessment will be performed by technical project personnel. Laboratory equipment will be maintained and calibrated, and records of these activities will be kept in accordance with established procedures. This will include laboratory oversight by project personnel, as well as laboratory data and document review.

Per the USEPA criteria for data quality for risk-based projects, 10 percent of the analytical data are required to meet Tier III data validation level of QA/QC related to sample collection, laboratory analysis, and data validation techniques. However, 100% data validation is planned so that data can be used to

support an NFA and/or RI decision. Data validation reports for all parameters will be generated according to the requirements described in attachment B of the USEPA – New England Data Validation Guidelines for Evaluating Environmental Guidelines. Following the processes identified in the QAPP, final data usability will be determined by the Navy Project Chemist in coordination with the Project Manager, Project Chemist, and independent Project Data Validator. Overall QA review of documentation, field sampling, and laboratory QC will allow determination of the acceptability of these data for use in this project.

Sample chemical analyses are discussed in greater detail in the QAPP.

3.3.1.5 Data Analysis

The project-specific DQOs for data collection, analysis, and quality assurance (QA) /quality control (QC) objectives will be met by implementing field QA/QC procedures and using various data evaluation processes. The DQOs for the data collection methods are provided in the SAP and QAPP, along with requirements for data collection and instrument calibration methods being used in the SI.

The QAPP has been developed to support the analysis and evaluation activities associated with this project. The QAPP consists of policies, procedures, specifications, standards, and documentation sufficient to produce data of quality adequate to meet the DQOs for the project. The QAPP, which includes a series of worksheets, has been written in conformance with the USEPA *Uniform Federal Policy for QAPPs*, Final Version 1, March 2005.

The QAPP addresses procedures to assure the precision, accuracy, representativeness, completeness, and comparability (PARCC) of field data generated during the course of this project, when applicable. It also provides a framework for evaluating existing data that may be used in this project. The QAPP defines the first stage of the QA requirements for data acquisition and assessment.

The QA procedures, such as tracking, reviewing, and auditing, are implemented as necessary to ensure that all project work is performed in accordance with professional standards, USEPA and Navy regulations and guidelines, and the specific goals and requirements stated in this SI Work Plan.

The QC of data collection, analysis, and assessment will be performed by technical project personnel. Equipment and instruments will be maintained and calibrated, and records of these activities will be kept in accordance with established procedures. This will include oversight by project personnel, as well as data and document review.

Data analysis will include field and QA/QC data tabulation, data analysis (comparison with project DQOs), and data validation. A summary report that will include text and tables summarizing the data validation

findings will be included in an appendix of the SI Report. The QA/QC review will allow determination of acceptability of data for use for this project.

3.3.1.6 XRF Investigations and Site Mapping

X-Ray Fluorescence (XRF) spectrometry investigations and additional site mapping will be conducted at all three sites – Machine Gun Boresight Range, NASB Skeet Range, and Topsham Skeet Range. Discrepancies and or lack of information on these sites have led to uncertainty on the site boundaries. Therefore, XRF investigations may aid in establishing the locations for MC sampling; in particular, by obtaining a greater number of real time samples resulting in a higher level of confidence in identifying area(s) of maximum contamination for fixed-based laboratory analysis. At each of the sites, a grid will be established over the site area and samples will be analyzed in the field for lead, the primary site contaminant, by XRF in the field, beginning with the grid squares expected to have the maximum concentrations and working outward. The USEPA will conduct the XRF investigation at the Machine Gun Boresight Range. TtNUS will conduct the XRF investigation at the NASB Skeet Range and Topsham Skeet Range.

At the Machine Gun Boresight Range, the disposition of the former berm is unknown and a dirt pile on site may or may not be the remnants of the former berm. The existing dirt pile boundary will be mapped in the field using a hand-held surveying GPS device. The distance to the dirt pile has been measured but the entire outline of the dirt pile should be mapped. Additionally, any areas with concrete debris should be mapped as they may represent the historical flared concrete end walls of the backstop berm. Maximum concentrations are expected at the existing dirt pile and/or the former berm location based on historical aerial photographs. Up to 90 soil samples are planned for collection via XRF, with a minimum of 30 soil samples. The minimum 30 samples would be sufficient if it can be established that maximum lead concentrations present exceed the residential standard of 400 mg/kg and the source area locations (i.e., former berm location, existing dirt pile, and area in between) are consistent with those of the current-day conceptual site model, because the decision to move forward to an RI would be evident at this point and the data collected could be built upon during the RI. Otherwise, up to 90 samples will be collected via XRF to meet the objective. During field analysis, the XRF lead data will be reviewed on a ½ day basis (approximately every 15 samples) to support the continuing effort of determining sample locations.

At the NASB Skeet Range, aerial photographs are available showing the site location. However, the site has been disturbed during construction of the impoundment ponds and the disturbance may have extended beyond the edges of the impoundment ponds. The site is further complicated in that the orientation of the skeet range was changed. Maximum concentrations are expected within the shotfall zone(s), working radially outward from the center. Up to 90 surface soil samples are planned for collection via XRF, with a minimum of 30 surface soil samples. The minimum 30 samples would be

sufficient if it can be established that maximum lead concentrations present exceed the residential standard of 400 mg/kg and the source area location (i.e., shotfall zone) is consistent with that of the current-day conceptual site model, because the decision to move forward to an RI would be evident at this point and the data collected could be built upon during the RI. Otherwise, up to 90 samples will be collected via XRF to meet the objective. Considering the changed orientation of the NASB Skeet Range over the years, the number of XRF samples is expected to be on the upper end (90 samples) with concentration on the overlap area of the two historic shotfall fans. The XRF samples will be discrete in order to "match" the discrete sampling methodology for the samples to be sent to the fixed-base analytical laboratory. During field analysis, the XRF lead data will be reviewed on a ½ day basis (approximately every 15 samples) to support the continuing effort of determining sample locations.

At Topsham Skeet Range, very little information is available on the exact location of the range and aerial photographs do not resolve this issue. Also, additional effort is warranted because most of the site lies outside of the NAS Brunswick boundary and so the Navy will not be able to place land use controls on the property. Any remaining historical features, such as remaining wooden posts that are reportedly the launching points of the skeet targets, etc., will be mapped via a hand-held surveying GPS device. Maximum concentrations are expected within the shotfall zone, working radially outward from the center. Similar to the Machine Gun Boresight Range, up to 90 surface soil samples are planned for collection via XRF, with a minimum of 30 surface soil samples. The minimum 30 samples would be sufficient if it can be established that maximum lead concentrations present exceed the residential standard of 400 mg/kg and the source area location (i.e., shotfall zone) is consistent with that of the current-day conceptual site model, because the decision to move forward to an RI would be evident at this point and the data collected could be built upon during the RI. Otherwise, up to 90 samples will be collected via XRF to meet the objective. The XRF samples will be discrete in order to "match" the discrete sampling methodology for the samples to be sent to the fixed-base analytical laboratory. During field analysis, the XRF lead data will be reviewed on a ½ day basis (approximately every 15 samples) to support the continuing effort of determining sample locations.

The XRF results will aid in locating the samples for MC fixed-based laboratory analysis (soil) and in determining the location and necessity of installing temporary monitoring wells at these sites. The Navy will discuss potential groundwater sampling requirements and locations with the EPA and MEDEP after conducting site reconnaissance and soil sampling activities at the two skeet ranges. Samples will be collected at the locations of highest concentrations established by the XRF investigation. The XRF results will be used for screening purposes only and so no confirmation sampling for the XRF samples are planned prior to collecting the MC soil samples for fixed-based laboratory analysis. Planned soil sampling locations shown on the figures may be adjusted based on XRF results. Soil samples will be collected as per the sampling scheme, relative to the skeet range location shown on the figures; the

sample locations shown provide full site coverage of the shotfall zone and firing point. However, it may become apparent during XRF sampling that the actual skeet range location is not as depicted on the figures and so, based on XRF results, the soil sampling locations may be shifted as necessary while still maintaining the same coverage. Additional details on XRF Sampling are provided in Appendix A SAP, Section 4.6.4, XRF Sampling of Lead in Soil.

3.4 SI REPORT

Information obtained from fieldwork will be used to refine the existing CSM and MRP site boundaries. The SI Report will include photographs taken during field activities, a description of field observations, GPS data for relevant points, analytical data, and a discussion of the sample data. The SI Report will include a discussion of the findings of the fieldwork and will recommend future site response, if necessary. The SI Report will also identify what additional data must be collected, if any, in order to make decisions regarding future response actions. Survey data coordinates (both GPS and land surveyor based) will be provided in the report, as well as an electronic version.

4.0 HEALTH AND SAFETY

The procedures for ensuring health and safety during the MRP SI are presented in a Health and Safety Plan (HASP) prepared by TtNUS as an internal Navy requirement, and has been reviewed and approved by the Navy separate from the subject Work Plan.

5.0 PROJECT MANAGEMENT

The Navy contractor will provide all of the documents and will participate in all of the meetings and conference calls in accordance with the protocols stated in the NAVFAC Mid-Atlantic project SOW.

5.1 PROJECT SCHEDULE

The project schedule, provided in Table 5-1, has been established in accordance with the Malcolm Pirnie SOW dated 14 September 2006 and subsequent modifications:

Task 1 – Meeting Support
Task 2 – SI Work Plan

The project schedule and task status is provided in Table 5-1.

Table 5-1: Project Schedule

Task Status	Task	Completion Date
Complete	Project Schedule (Malcolm Pirnie)	November 2006
Complete	Meeting Support (Malcolm Pirnie)	October 2006
Complete	Draft SI MC Work Plan (Malcolm Pirnie)	January 2007
Complete	Navy Review Comments	February 2007
Complete	Draft Final SI MC Work Plan (Malcolm Pirnie)	February 2007
Complete	Stakeholder Review Comments	March 2007
Complete	Stakeholder Meeting Support	October 2007
Not Complete	Revised Draft Final SI MC Work Plan (Tetra Tech)	February 2008
Not Complete	Stakeholder Review Comments	March 2008
Not Complete	Final MC Work Plan	June 2009
Not Complete	SI MC Fieldwork	July/August 2009
Not Complete	Draft SI MC Report	January 2010
Not Complete	Navy Review Comments	January 2010
Not Complete	Draft Final SI MC Report	February 2010
Not Complete	Stakeholder Review Comments	April 2010
Not Complete	Final SI MC Report	May 2010

Note: TBD = to be determined

5.2 PROJECT PERSONNEL

The following sub-sections identify the Navy, regulatory, and project personnel that comprise the project team.

5.2.1 Navy and Regulatory Project Personnel

Table 5-2 lists the Navy and Regulatory personnel involved with this project and their associated agencies/organizations.

Table 5-2: Navy and Regulatory Personnel

Name	Org Code	Title	Work Phone
NAVY BRAC PMO NE			
Mr. Todd Bober	NAVFAC MidAtlantic	RPM	(215) 897-4911
Mr. Paul Burgio	NAVY BRAC PMO NE	BRAC Environmental Coordinator	(215) 897-4915
Mr. David Barclift	NAVFAC MidAtlantic	BRAC Technical Support	(215) 897-4913
NAVFAC Atlantic			
Ms. Jennifer Wright Ms. Amy Van Dercook Mike Green	NAVFAC LANTDIV	Technical Support	(757) 322-8428 (757) 322-4764 (757) 322-8108
NASB			
Ms. Lisa Joy	NASB	NASB Environmental Director	(207) 921-1720
Mr. Michael Fagan		IR Coordinator for MRP Sites	(207) 921-1717
USEPA, New England Region I			
Mr. Michael Daly	EPA	RPM Federal Facilities Superfund Section	(617) 918-1384
Maine Department of Environmental Protection			
Ms. Claudia Sait	MEDEP	RPM Federal Facilities Section	(207) 287-7713

5.2.2 Project Personnel

Project personnel and their responsibilities are listed in Table 5-3.

Table 5-3: Project Personnel

Name	Title	Work Phone
Linda Klink	Project Manager	412-921-8650
Charles Race	Maine Certified Geologist	978-474-8437
Ralph Basinski	MRP Scientist	412-921-8308
TBD	Field Operations Leader	TBD

6.0 PROJECT DELIVERABLES

The project deliverables are expected to include:

- Summary of historical activities resulting in deposition of MC on each site
- Description of MC detected above method detection limits on each MRP site, including sample analytical data
- Description of migration/exposure pathways for MC and human and environmental receptors as part of the revised CSM
- A recommendation on the path forward (e.g., NFA, proceed to RI, or interim measure) warranted at each MRP site
- Data that must be collected, if any, in order to make decisions regarding future response actions

Additional project deliverables include the following:

- Meeting agendas and minutes
- Project schedule (updated monthly)
- Monthly delivery order reviews
- Draft, Draft Final, and Final SI Work Plans with appendices
- Draft, Draft Final and Final SI Reports
- All Geographical Information System (GIS) and map files
- Copies of relevant source material requested by the RPM and/or required by the SOW
- Electronic Data Deliverable (EDD) format, Version 5, submitted to MEDEP electronically

APPENDIX A

SAMPLING AND ANALYSIS PLAN (SAP)

SAMPLING AND ANALYSIS PLAN
NAVAL AIR STATION BRUNSWICK, MAINE

JUNE 2009

Prepared for:

NAVAL FACILITIES ENGINEERING COMMAND MID-ATLANTIC (MIDLANT)
BRAC PMO Northeast
4911 South Broad Street
Philadelphia, PA 19112-1303

Prepared by:

MALCOLM PIRNIE, INC.
104 Corporate Park Drive
White Plains, NY 10602

and

TETRA TECH NUS, INC.
661 Andersen Drive, Foster Plaza #7
Pittsburgh, PA 15220-2745

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ACRONYMS

bgs	Below Ground Surface
BIP	Blow-In-Place
BRAC PMO NE	Base Realignment and Closure Program Management Office Northeast
CG	Certified Geologist
COC	Chain-of-Custody
CRREL	Cold Regions Research and Engineering Laboratory
CSM	Conceptual Site Model
DPT	Direct-Push Technology
DQCR	Daily Quality Control Report
DQO	Data Quality Objectives
EOD	Explosive Ordnance Disposal
ERDC	Engineer Research and Development Center
FOL	Field Operations Leader
GPS	Global Positioning System
HASP	Health and Safety Plan
IDW	Investigative-Derived Waste
MC	Munitions Constituents
MEC	Munitions and Explosives of Concern
MEDEP	Maine Department of Environmental Protection
MRP	Munitions Response Program
Malcolm Pirnie	Malcolm Pirnie, Inc.
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NASB	Naval Air Station Brunswick
Navy	Department of the Navy
NFA	No Further Action
NOSSA	Navy Ordnance Safety and Security Activity
NTU	Nephelometric Turbidity Units
ORP	Oxidation/Reduction Potential
PA	Preliminary Assessment
PAH	Polycyclic Aromatic Hydrocarbons
PCB	Polychlorinated Biphenyl
PM	Project Manager
POC	Point of Contact
PPE	Personal Protective Equipment

PVC	Polyvinyl Chloride
QA	Quality Assurance
QAPP	Quality Assurance Program Plan
QC	Quality Control
RAB	Restoration Advisory Board
RI	Remedial Investigation
SAP	Sample and Analysis Plan
SI	Site Inspection
SOP	Standard Operating Procedure
SVOC	Semivolatile Organic Compound
TAL	Target Analyte List
TCL	Target Compound List
TOC	Total Organic Carbon
TtNUS	Tetra Tech NUS, Inc. or Tetra Tech
U.S.	United States
USCS	Unified Soil Classification System
USEPA	United States Environmental Protection Agency
UXO	Unexploded Ordnance
VOC	Volatile Organic Compound
XRF	X-Ray Fluorescence
^{o}C	Degrees Celsius

1.0 INTRODUCTION

Under Contract Number N62472-02-D-1300, Task Order 0002, Malcolm Pirnie, Inc. (Malcolm Pirnie) has been tasked by the Department of the Navy (Navy) Naval Facilities Engineering Command Atlantic to author a Sampling and Analysis Plan (SAP) as part of the Site Inspection (SI) Work Plan for three munitions Response Program (MRP) sites at Naval Air Station Brunswick (NASB), Cumberland County, Maine (ME). Tetra Tech NUS, Inc. (TtNUS or Tetra Tech) was tasked to finalize this document under the Comprehensive Long-Term Environmental Action Navy (CLEAN) Contract Number N62472-03-D-0057, Contract Task Order (CTO) 069. TtNUS will be performing the SI field work and will be responsible for following the SI Work Plan and this SAP. TtNUS is responsible for creating the Quality Assurance Project Plan (QAPP), which is included in Attachment 1 of this SAP. Attachments to the QAPP include UFP-QAPP Worksheets (1-A), Field SOPs (1-B), Field Forms (1-C), Analytical Laboratory SOPs (1-D), and MRP Site Visit Documentation (1-E).

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

Project personnel and their responsibilities are listed in Section 5.2 of the SI Work Plan.

3.0 PROJECT SCOPE AND OBJECTIVES

The goal of this project is to determine the presence or absence of munitions constituents (MC) that may remain at the three MRP sites (Machine Gun Boresight Range, Skeet Range, and the Topsham Annex Skeet Range) from activities conducted by the Department of Defense during operation of these sites and that may potentially pose a threat to human health and/or the environment.

The presence of MEC is not anticipated at these three MRP sites; therefore, qualified team members [Unexploded Ordnance (UXO) Technicians] will not be on-site, and no munitions and explosives of concern (MEC) for avoidance activities are necessary at these sites. Samples will be collected to selectively analyze for Target Analyte List (TAL) metals, propellants (nitroglycerin), explosives (dinitrotoluene), perchlorate, pH, total organic carbon (TOC), cation exchange capacity (CEC), nitrates, and Target Compound List (TCL) polycyclic aromatic hydrocarbons (PAHs) as dictated by historical MC site activities for a given site. Moreover, all sediment samples will be collected for TOC analyses to aid in evaluating toxicity of metals.

Surface soil, subsurface soil, sediment, surface water, and groundwater samples will be collected for analytical laboratory testing. The analytical methods were selected on the basis of the types of munitions (and other types of contamination) known to have been used at the sites, and include the standard suite of range-related analytical parameters to account for unknown items. The standard analytical methods include TAL Metals (SW-846 Method 6010B), Explosives (dinitrotoluene only by SW-846 Method 8330A), Perchlorate [SW-846 Method 6850 (with filtration at the laboratory)], TOC (Lloyd Kahn Method), pH (SW-846 Method 9045D), CEC (SW-846 Method 9081), propellants (nitroglycerin by SW-846 Method 8332), nitrates (SW-846 Method 9056 or EPA 300) and TCL PAHs (USEPA Method 8270C SIM).

Field and laboratory work will be of the quality to support screening comparisons with USEPA Soil Screening Levels and Ecological Screening Values (where applicable). Metal concentrations will be compared to USEPA and ME Department of Environmental Protection (MEDEP) residential soil criteria. An installation-wide background study is currently underway at NASB. Background data should be available at the time of the SI Report, and analytical results will be compared to the background data.

Table 3-1 provides the site location, quantity, media type, and target constituents.

Table 3-1: Sample Summary Table

MRP Site	Number of Samples / Media					
	TAL Metals	Explosives (Dinitrotoluene)	Propellants (Nitroglycerin)	Perchlorate	PAHs	Nitrates
Machine Gun Boresight Range	13 surface soil (includes 1 background sample) 12 subsurface soil 5 groundwater		13 surface soil 12 subsurface soil 5 groundwater	5 groundwater	1 surface soil (background sample)	
NASB Skeet Range	20 surface soil* 8 subsurface soil 7 groundwater (5 from new wells and 2 from existing wells) 4 surface water 4 sediment*	4 surface soil 2 subsurface soil 1 groundwater (MW-NASB-078)	4 surface soil 2 subsurface soil 1 groundwater (MW-NASB-078)		20 surface soil 8 subsurface soil 7 groundwater**	7 groundwater
Topsham Annex Skeet Range	15 surface soil* (includes 1 background sample) 5 subsurface soil 3 sediment * 3 surface water (includes 1 upstream collocated sample) 5 groundwater	4 surface soil 1 subsurface soil 1 groundwater	4 surface soil 1 subsurface soil 1 groundwater		15 surface soil 5 subsurface soil 3 sediment 3 surface water 5 groundwater** 1 surface water co-located sediment (background sample)	5 groundwater

Total Organic Carbon (TOC) analysis will be conducted for all sediment samples as an indicator parameter to aid in toxicity evaluation of TAL metals. Four soil samples each from NASB Skeet Range and Topsham Annex Skeet Range will be analyzed for pH, TOC, and CEC.
** Temporary monitoring wells to be installed if warranted. The Navy will discuss potential groundwater sampling requirements and locations with the EPA and MEDEP after conducting site reconnaissance and soil sampling activities.

Note: Sample quantities presented do not include QC samples. For details on QC samples, refer to Tables 4-2 through 4-4.

3.1 DATA QUALITY OBJECTIVES AND CRITERIA

The following sections outline the Data Quality Objectives (DQOs) and project quality assurance (QA) objectives for the MEC visual survey and MC soil sampling at the three MRP sites at NASB.

3.1.1 Data Quality Objectives

The seven-step USEPA DQO process was used to develop the rationale for this field investigation. This process is further described in the *DQO Process for Hazardous Waste Site Investigations* (USEPA, 2000a) and *Guidance for the DQOs Process* (USEPA, 2000b).

The DQO process is a strategic planning approach to optimize data collection activities and define the criteria that the data collection design should satisfy. The results generate a scientific and resource-effective data-collection design. The DQOs selected are qualitative and quantitative statements that:

- clarify the study objectives
- define the appropriate types and amount of data to collect
- determine the appropriate conditions (e.g. location and time) for data collection
- specify the tolerable limits on decision errors

Using the DQO process to plan field activities assures that the type, quantity, and quality of data used in decision making is appropriate for its intended use. The DQO process comprises the following seven steps:

- state the problem
- identify the decisions
- identify the inputs to the decision
- define the boundaries of the study
- develop the decision rules
- specify the tolerable limits on decision errors
- optimize the design for obtaining data

These steps are further described in the following subsections.

3.1.1.1 State the Problem

There are three MRP sites to be addressed as part of this SI, all of which are described in detail in Section 2.0 of the SI Work Plan. The Navy and members of the planning team want to determine if a no

further action (NFA) recommendation is appropriate for these MRP sites or if a recommendation to proceed to a Remedial Investigation (RI) is warranted.

Based on historical records and previous visual inspections summarized in the Final Preliminary Assessments (PAs) (Malcolm Pirnie, 2005 and 2006) and additional information gathered following the October 2007 kickoff meeting/site visits with the Stakeholders, the following is known. Note that PA and Work Plan figures used 2003 GIS aerial photographs as the background.

- Machine Gun Boresight Range (see Appendix C-1)

  Based on evaluation of the PA Conceptual Site Model and supplemental information, MC investigation should encompass the original berm location, the existing dirt pile beyond the site, and the area in between, considering that the berm could have been graded during bulldozing. Considering the uncertainty of the berm disposition, additional investigative techniques are warranted (XRF sampling for lead) to maximize the value of the data collected. Groundwater contamination (primarily perchlorate) is possible, although not considered during the PA.

  PA Conceptual Site Model

  - The former range was used during the 1950s to align and test fire aircraft mounted guns.
  - The former range is not suspected to contain MEC.
  - Potential exists for MC to be present primarily in the surface soil and potentially in the shallow subsurface soil, considering that the berm has been disturbed.

  Supplemental Information

  - Mapping was revised based on historical aerial photographs provided by MEDEP, particularly the 1959 aerial (Appendix C-1). There is no evidence of the berm at its original location. However, there is an existing dirt pile further out from the site that may or may not be remnants of the historical berm. Of note, the Site 1 and 3 landfills were not in operation in 1978 when the berm was bulldozed and so the berm material would not have been disposed of at these landfills.

  - Numerous other wells and other historical samples are available (Appendix C-1). Based on data from Site 9 located less than ¼ mile to the north, groundwater flow direction is presumed to be generally to the north toward the impoundment ponds while locally controlled, then probably shifting to the northeast toward the west arm of Picnic Pond; further to the south, groundwater flow direction is expected to shift to the southeast toward Merriconeag Stream.

- Skeet Range (see Appendix C-2)

  Considering the site changes (shift in orientation) and site disturbances (impoundment pond construction) over the years, additional investigative techniques are warranted (XRF sampling for lead) to maximize the value of the data collected. Groundwater contamination is not anticipated unless lead concentrations in soil are significantly elevated and a low pH suggests that lead migration is plausible. The Navy will discuss potential groundwater sampling requirements and locations with the EPA and MEDEP after conducting site reconnaissance and soil sampling activities. Based on evaluation of the PA Conceptual Site Model and supplemental information, two existing Site 9 wells can be employed for groundwater sampling at the Skeet Range (if groundwater investigation is decided upon) to save on unnecessary new well installation. Numerous historical samples are available from previous investigations at Site 9 and Site 6 and the Skeet Range SI Report can compare SI results and contamination patterns to aid in evaluating contaminant source and extent of contamination. Also, considering that PAH treatment impoundments are located on site and PAHs are a suspected Skeet Range contaminant, PAH surface water/sediment data collected from the impoundments would not have much value in assessing Skeet Range contamination.

  <u>PA Conceptual Site Model</u>

  - The former range was used during the 1950s to train military personnel.
  - The former range is not suspected to contain MEC.
  - Potential exists for MC to be present primarily in the surface soil.

  <u>Supplemental Information</u>

  - The mapping from the PA was confirmed, based on historical aerial photographs provided by MEDEP, particularly the 1959 aerial (Appendix C-2).

  - Historical data is available from adjacent Site 9 and Site 6. Two wells from Site 9 are properly located for groundwater sampling to support the Skeet Range groundwater investigation, if groundwater investigation is decided upon. Numerous other wells and other historical samples are available (Appendix C-2). Based on adjacent Site 9 data, groundwater flow direction is presumed to be generally to the north toward the impoundment ponds while locally controlled, then probably shifting to the northeast toward the west arm of Picnic Pond; further to the south, groundwater flow direction is expected to shift to the southeast toward Merriconeag Stream. Pertinent text, analytical results, and field forms are included in Appendix C-2. The impoundments on site are PAH treatment areas for runoff from the runways

- Topsham Annex Skeet Range (see Appendix C-3)

  Based on evaluation of the PA Conceptual Site Model and supplemental information, the exact location of the Topsham Skeet Range is uncertain. Most of the site is located outside of the NAS Brunswick boundary; therefore, the Navy will not be able to place land use controls on the property and so more extensive investigation is warranted. Considering the uncertainty of the range location, additional investigative techniques are warranted (sampling for lead) to maximize the value of the data collected. Groundwater contamination is not anticipated unless lead concentrations in soil are significantly elevated and a low pH suggests that lead migration is plausible. If groundwater investigation is warranted based on XRF results, The Navy will discuss potential groundwater sampling requirements and locations with the EPA and MEDEP after conducting site reconnaissance and soil sampling activities. If groundwater investigation is decided upon, groundwater flow direction will also be established considering nearby potential source areas other than the Skeet Range; collection of supplemental water levels from the three existing nearby wells from the 2006 investigation at TOP-01 may be useful for this evaluation.

  PA Conceptual Site Model

  - The former range was used by the U.S. Air Force for training prior to the 1970s.
  - The former range is not suspected to contain MEC.
  - Potential exists for MC to be present primarily in the surface soil, and potentially sediment and surface water.

  Supplemental Information

  - Most of the site is located outside of the NAS Brunswick boundary; therefore, the Navy will not be able to place land use controls on the property. Updated aerial photographs (Spring of 2006) provided by the Town of Topsham are included in Appendix C-3.

  - Historical data is available from 2004 and 2006 investigations of the Skeet Range and nearby areas at Topsham Annex. Of note, reference figures in Appendix C-3 associated with the 2004 Investigation Report (EA/ECC, February 2006) show inferred groundwater flow direction to the west based on regional groundwater flow. However, the subsequent 2006 Investigation Report (Tetra Tech, draft) text indicates that groundwater flow direction, determined from water levels during monitoring well construction and survey data is to the southeast. This information will support site groundwater investigation, if groundwater investigation is decided upon. Pertinent text, analytical results, and field forms are included in Appendix C-3, and a brief summary is provided as follows:

Skeet Range: For the 2004 investigation, 3 composite surface soil samples (SS-01, SS-02, and SS-03) were collected from an area believed to be the shotfall area of the Skeet Range where skeet pigeon chips were found. The samples were collected from depth intervals of 0 to 6 inches and analyzed for lead, with resulting concentrations of 202, 82.9, and 49.9 mg/kg, respectively, all less than the Maine RAGs criteria of 375 mg/kg.

TOP-01 (Filled Area): TOP-01 was a nearby reported area where fill material (i.e., construction rubble, extra soil) was dumped and buried. For the 2004 investigation, three direct-push soil borings (TOP-01-01, TOP-01-02, and TOP-01-03) were advanced and two subsurface soil samples and one shallow groundwater sample were collected from each boring; each sample was analyzed for VOCs, SVOCs, TPH-DROs, TPH-GROs, and RCRA 8 metals and the soil samples were additionally analyzed for PCBs. Relative to Skeet Range suspected contaminants, lead was detected at TOP-01-02 at a concentration of 22.9 µg/L, exceeding both the Maine MEG of 10 µg/L and the Federal MCL of 15 µg/L. For the 2006 investigation, five direct-push soil borings (TOP-01-04 through TOP-01-09) surrounding the 2004 data points were advanced and two subsurface soil samples were collected from each boring. Additionally three shallow monitoring wells (TOP-01-MW-01, TOP-01-MW-02, and TOP-01-MW-03) were installed; groundwater samples were collected from each well. Samples were analyzed for VOCs, SVOCs, TPH-DRO, and RCRA 8 metals. Lead was only detected at monitoring well TOP-01-MW-02, at a concentration (7.2 µg/L) not exceeding criteria.

TOP-02 (Former Oil/Water Separator): TOP-02 was a nearby reported area where an oil/water separator was formerly located. For the 2004 investigation, three direct-push soil borings (TOP-02-01, TOP-02-02, and TOP-02-03) were advanced and one subsurface soil sample and one shallow groundwater sample were collected from each boring; each sample was analyzed for TPH-DROs, TPH-GROs, and RCRA 8 metals. Lead, a primary suspected Skeet Range contaminant, was not detected in any of the three wells.

Although groundwater contamination is not expected at any of the MRP sites, it will be important to verify this assumption at the Machine Gun Boresight Range. For the two skeet ranges, groundwater contamination is not anticipated unless lead concentrations in soil are significantly elevated and a low pH suggests that lead migration is plausible.

3.1.1.2 Identify the Decisions

Groundwater (as warranted), surface water, sediment, and surface/subsurface soil sampling and subsequent analyses will be used to help determine the answer to the following question:

Is an NFA recommendation appropriate for MC at any of the three MRP sites, or is it warranted to proceed to an RI?

NFA for MC will be recommended for these MRP sites if analytical results are below project action levels [Worksheet #15 (Appendix A SAP, Attachment 1-A UFP-QAPP Worksheets) Reference Limits and Evaluation Table] and there is a high level of certainty that the site source areas were located accurately for sampling. Additional investigation for MC will be recommended if MC analytical results are greater than project action levels and are determined to represent an unacceptable risk to human or ecological receptors and if the perceived site source areas were not accurately mapped (i.e., not verified as source areas during the SI). An immediate remedial action will be recommended if analytical results indicate an immediate threat to human health or the environment.

3.1.1.3 Identify the Inputs to the Decision

The following inputs to the decision for the investigation at the three MRP sites have been identified:

- Presence or absence of MC
- If present, determine the concentration values for MC detected
- The spatial boundary of MC, if MC is detected
- If MC is present in soil, determine if contamination has migrated to surface water (where present), sediment (where present), or groundwater (as warranted)

3.1.1.4 Define the Boundaries of the Study

3.1.1.4.1 Machine Gun Boresight Range

The Machine Gun Boresight Range is roughly bounded by Building 52, Building 55, and a containment pond to the north: Building 50 to the south: open field and forested area to the east: and a parking lot and Orion Street to the west.

The vertical boundary for MC is anticipated at two feet bgs.

3.1.1.4.2 Skeet Range

The Skeet Range is roughly bound by Building 211 to the north, forested area to the south and east, and Orion Street to the west.

The vertical boundary for MC is zero to 12 inches bgs.

3.1.1.4.3 Topsham Annex Skeet Range

The Topsham Annex Skeet Range is roughly bound by undeveloped land to the north, recreational fields and undeveloped land to the south, undeveloped land and residential property to the east, and residential property to the west.

The vertical boundary for MC is zero to 12 inches bgs.

3.1.1.5 Develop the Decision Rules

The objective of this investigation is to evaluate the potential presence of MC on the surface and in the subsurface of the MRP sites. To support this objective, the following decision factors were developed:

- NFA for MC will be recommended for the MRP sites if MC is not present in concentrations greater than project action levels [Worksheet #15 (Appendix A SAP, Attachment 1-A UFP-QAPP Worksheets) Reference Limits and Evaluation Table] and there is a high level of certainty that the site source areas were located accurately for sampling.

- Additional investigation for MC will be recommended if MC analytical results are greater than project action levels and/or are determined to represent an unacceptable risk to human or ecological receptors and if the perceived site source areas were not accurately mapped (i.e., not verified as source areas during the SI).

- An interim action on an expedited basis will be required if MC concentrations indicate an immediate threat to human health or the environment.

In addition to the decision factors, the Conceptual Site Models (CSMs) from the Final PAs (Malcolm Pirnie, 2005 and 2006) will be refined based on the visual survey and site investigation findings. The CSMs will present hypotheses regarding MEC and MC presence, potential routes of migration, and their potential impact on sensitive receptors. The CSMs will also provide a tool to evaluate and refine decisions regarding the MRP sites.

3.1.1.6 Specify Tolerable Limits on Decision Errors

The following major limits on decision errors were identified for the SI field activities regarding MC sampling:

- The two potential decision errors for this project are: identifying MRP site media as MC-impacted when it is really not (Type I error) and identifying MRP site media as not MC-impacted when it really is (Type II error). The consequence of a Type I decision error will be unnecessarily incurred project costs associated with a remediation action. The consequence for a Type II decision error will be continued liability and potential risk to human health and the environment. Precision and accuracy requirements for the data are presented in the QAPP. These requirements will be adequate for minimizing project decision errors.

Further discussion of data usability assessment is provided in the QAPP (Appendix A, Attachment 1) Section 4.2.3.

3.1.1.7 Optimize the Design for Obtaining Data

The overall goal of the DQO process is to design a SAP that will meet the needs of the data user (i.e., the decision-maker).

Activities associated with supporting this objective include:

- Selection of the most resource-effective sampling design that satisfies the DQOs
- Generation of sampling and analysis design alternatives based upon cost consideration, time constraints, sample selection constraints, access limitations due to safety, and other considerations

3.2 DATA REVIEW, VERIFICATION AND VALIDATION

This section describes the procedures that are planned to review, verify, and validate field and laboratory data. This section also discusses procedures for verifying that the data are sufficient to meet DQOs for the project.

3.2.1 Field Data Verification

Project team personnel will review field data to identify inconsistencies or anomalous values. Any inconsistencies will be resolved as soon as possible by the field personnel responsible for data collection.

All field personnel will be responsible for following the sampling and documentation procedures described in this SAP so that defensible and justifiable data are obtained.

Outliers that result from errors found during data verification will be identified and corrected, while outliers that cannot be attributed to errors in sampling, measurement, transcription, or calculation will be clearly identified in project reports.

3.2.2 Laboratory Data Verification

The laboratory will review data prior to its release from the laboratory. The analytical method performance will be determined by an examination of precision, accuracy, and completeness using the following QA/QC samples:

- Method Blanks: Measure of laboratory contamination and accuracy
- Laboratory Duplicates: Measure of laboratory precision
- Field Duplicates: Measure of field sampling and laboratory precision.
- Matrix Spikes: Measure of laboratory accuracy and any sample matrix effects
- Surrogate Spike Recoveries: Measure of laboratory accuracy
- Laboratory Control Samples: Measure of laboratory accuracy

The laboratory is required to evaluate their ability to meet these objectives. Outlying data will be flagged in accordance with laboratory standard operating procedures (SOPs) and corrective action shall be taken to rectify the problem. The laboratory case narratives will describe how the data did or did not meet the method criteria, and must describe the overall quality of the data and whether or not the data are valid and usable.

In order to ensure the analytical data generated by the laboratory are accurate, the Analytical Coordinator will review the electronic data deliverable from the laboratory to ensure that the data submitted electronically correspond to the hard copy results in the laboratory data deliverable.

3.2.3 Laboratory Data Validation

Data validation is an analyte- and sample-specific process that extends the data evaluation beyond methodical, procedural, or contractual compliance (i.e., data verification) to determine the analytical quality of a specific data set. Data validation criteria are based upon the quality objectives developed in this SAP/QAPP or presented in the sampling or analytical method. When possible, data validation will include a determination of the reasons for any failure to meet methodical, procedural, or contractual

requirements, and an evaluation of the impact of such failure on the overall data set. Data validation applies to activities in the analytical laboratory, as well as in the field.

For a Level III data validation, the data values for routine and QC samples are assumed to be correctly reported by the laboratory. Data quality will be assessed by comparing data collection, QC, and reporting parameters to the appropriate criteria (or limits) as specified in the project SAP, by Contract Laboratory Program requirements, or by method-specific requirements. If calculations for quantification are verified, it will be done on a limited basis and may require raw data in addition to the standard data forms normally present in a data package.

3.2.4 Data Reporting Requirements

The laboratory will prepare Level III data packages, as prescribed in the Work Plan, that will consist of a case narrative, copies of all chain-of-custody (COC) records, sample results, and QA/QC summaries. The case narrative will provide the following information:

- Laboratory name, project name, project order number, analytical batch information, and a table that cross-references client and laboratory sample IDs
- Detailed documentation of all sample shipping and receiving, preparation, analytical, and quality deficiencies
- Thorough explanation of all instances of manual integration
- Copies of all associated nonconformance and corrective action forms that will describe the nature of the deficiency and the corrective action taken
- Copies of all associated sample receipt notices

Electronic Data Deliverables (EDDs) are required for all analytical results. An automated laboratory information management system (LIMS) must be used to produce the EDD. The laboratory will verify EDDs internally before they are issued, and EDDs will be delivered in a format compatible with DEEMS.

4.0 FIELD ACTIVITIES

4.1 RATIONALE/DESIGN

The sampling rationale for this study is to collect sufficient data to confirm the presence/absence of MC within the MRP sites. The following items have been incorporated into the sampling program design.

4.2 STAKEHOLDER MEETING

The Stakeholder Meeting is used to gain a consensus regarding the sampling program approach. Based on the discussions at the Stakeholder Meeting, a strategy can be implemented for MC activities at each of the three MRP sites at NASB. Site visits and scoping discussions were conducted at a Stakeholder Meeting on October 23 through October 24, 2007 and additions/clarifications have been addressed herein (see Attachment 1-E).

4.3 MUNITIONS CONSTITUENTS INVESTIGATIVE ACTIVITIES

4.3.1 Machine Gun Boresight Range

Twenty-five biased discrete soil samples will be collected at this site based on visual observations and locations of the former range features and based on results of the USEPA's X-Ray Fluorescence (XRF) investigation. Six surface soil locations will be sampled in the zone between the firing point and the berm. Six locations at the berm will be sampled from the surface plus up to two discrete subsurface intervals since the berm was disturbed and turned over, for a total of 18 samples. The locations shown on the sampling figure are approximate and will be refined based on the results of the XRF investigation. Also, one discrete surface soil sample will be collected for comparison purposes with background conditions. One groundwater sample will be collected from each of 5 temporary wells that will be installed during the field work effort. The location of the wells will be one at the firing point and the other 4 positioned one in each direction surrounding the berm. The soil and groundwater samples will be analyzed for TAL metals and propellants (nitroglycerin) using USEPA Method 6010B and8330A, respectively; groundwater samples will additionally be analyzed for perchlorate using SW846 Method 6850 (with filtration at the laboratory).

4.3.2 Skeet Range

Twenty biased discrete surface soil and 8 discrete subsurface soil samples will be collected at this site based on visual observations and the results of TtNUS's XRF investigation. The locations shown on the sampling figure are approximate and will be refined based on the results of the XRF investigation (see

Section 4.6.4 XRF Sampling of Lead in Soil for additional details). Soil samples will be collected as per the sampling scheme, relative to the skeet range location shown on the figures; the sample locations shown provide full site coverage of the shotfall zone and firing point. However, it may become apparent during XRF sampling that the actual skeet range location is not as depicted on the figures and so, based on XRF results, the soil sampling locations may be shifted as necessary while still maintaining the same coverage. Surface soil samples will be collected from the 0 to 3-inch bgs interval. Subsurface soil samples will be collected from the 3- to 12-inch bgs interval; if at a given location, XRF results for a subsurface soil sample exceeds 375 mg/kg for lead, an additional subsurface soil sample from the 12- to 18-inch interval will additionally be collected. Seven groundwater (if warranted) four co-located surface water/sediment samples (three composite samples from within the impoundments and one discrete sample from the tributary) will be collected at this site based on visual observations and locations of the former range features, which includes an overlap of the pre-1950 and post-1950 footprints. If groundwater investigation is decided upon, seven groundwater samples will be collected. One groundwater sample will be collected from each of the five new temporary wells that will be installed at the Skeet Range during field activities; four of the new wells will be located within the likely shotfall zones and one of the new wells will be located outside of the shotfall zone. In addition, one groundwater sample will be collected from the existing well MW-NASB-078, which will serve as the firing point well, and one groundwater sample will be collected from existing well MW-NASB-076 (if intact) within the shotfall zone.

All soil and groundwater (if warranted) samples will be analyzed for TAL metals using SW-846 Method 6010B and for PAHs using SW-846 Method 8270C SIM. The four surface and two subsurface soil samples at the firing point will also be analyzed for explosives (dinitrotoluene) using SW-846 Method 8330A and propellants (nitroglycerin) using SW-846 Method 8332. Four random soil samples will be analyzed for pH using SW-846 Method 9045D, TOC using the Lloyd Kahn Method, and CEC using SW-846 Method 9081. If groundwater sampling is conducted, all groundwater samples will be analyzed for nitrates using SW-846 Method 9056 or EPA 300 and one groundwater sample will be analyzed from explosives (dinitrotoluene) and propellants (nitroglycerin) using SW-846 Method 8330A. The surface water and sediment samples will be analyzed for TAL metals. Additionally, all sediment samples will be analyzed for TOC using Method SW-846 9060.

4.3.3 Topsham Annex Skeet Range

Fifteen biased discrete surface soil, which includes one surface soil sample for comparison purposes with background conditions, and 5 discrete subsurface soil samples will be collected at this site based on visual observations and the results of TtNUS's XRF investigation. The locations shown on the sampling figure are approximate and will be refined based on the results of the XRF investigation (see Section 4.6.4 XRF Sampling of Lead in Soil for additional details). Soil samples will be collected as per the sampling scheme, relative to the skeet range location shown on the figures; the sample locations shown

provide full site coverage of the shotfall zone and firing point. However, it may become apparent during XRF sampling that the actual skeet range location is not as depicted on the figures and so, based on XRF results, the soil sampling locations may be shifted as necessary while still maintaining the same coverage. Surface soil samples will be collected from the 0 to 3-inch bgs interval. Subsurface soil samples will be collected from the 3- to 12-inch bgs interval; if at a given location, XRF results for a subsurface soil sample exceeds 375 mg/kg for lead, an additional subsurface soil sample from the 12- to 18-inch interval will additionally be collected. Three co-located discrete surface water/sediment samples, which includes one upstream sample for comparison purposes and 5 groundwater samples (if warranted) will also be collected at this site. If groundwater investigation is decided upon, groundwater samples will be collected from five new wells. One well will be installed at the firing point, three will be installed within the likely shotfall zone, and one will be installed surrounding the target area outside of the shotfall zone. The surface water/sediment samples will be collected from the brook running through the site with both samples collected within the shotfall zone. In addition, one upstream surface water/sediment sample will also be collected for comparison purposes. All samples will be analyzed for TAL metals using SW-846 Method 6010B and for PAHs using SW-846 Method 8270C SIM. Four surface and one subsurface soil sample will also be analyzed for explosives (dinitrotoluene) and propellants (nitroglycerin) using SW-846 Method 8330A, and four random soil samples will be analyzed for TOC using the Lloyd Kahn Methods, pH using SW-846 Method 9045D and CEC using SW-846 Method 9081. All groundwater (if warranted) samples will be analyzed for nitrates using SW-846 Method 9056 or EPA 300 and one groundwater sample (from the firing point) will be analyzed from explosives (dinitrotoluene) and propellants (nitroglycerin) using SW-846 Method 8330A. Additionally, all sediment samples and four random soil samples will be analyzed for TOC using the Lloyd Kahn Methods..

4.4 SAMPLING OVERVIEW

Collection of discrete grab samples is proposed for surface and subsurface soil samples at these sites.

Sediment and surface water samples will be obtained as discrete grab samples for the Topsham Skeet Range while for the NASB Skeet Range three of the samples will be composite (within impoundment ponds) and one will be discrete (from the tributary). Groundwater samples (at the Machine Gun Boresight Range and if warranted at the skeet ranges) will be discrete grab samples collected using USEPA Region 1 New Monitoring Well Sampling Procedures.

Surface soil/sediment samples will be collected with a polyethylene disposable scoop or similar equipment while wearing nitrile gloves. New scoops and gloves will be used at each sampling location. The analytical samples will be collected and placed directly into the appropriate sample containers, labeled, and placed in an ice chest chilled to a maximum temperature of 4 degrees Celsius (°C). A portion of the sample will be set aside and used to log a description of the soil characteristics on a soil

sampling log form using the Unified Soil Classification System (USCS). An example of this soil sampling log form is Attachment 1-C.

Surface water samples will be collected using disposable bailers and polyethylene gloves. New bailers and gloves will be used at each sampling location. The analytical samples will be collected and placed directly into the appropriate sample containers, labeled, and placed in an ice chest chilled to a maximum temperature of 4°C. A portion of the sample will be set aside and used to log a description of soil characteristics on a soil sampling log form using the USCS.

Groundwater samples will be collected using a peristaltic pump, disposable tubing, and polyethylene gloves. New tubing and gloves will be used at each location. Before taking a sample, each well will be purged and groundwater sampled using USEPA Region 1 low-flow sampling protocol. The analytical samples will be collected and placed directly into the appropriate sample containers, labeled, and placed in an ice chest chilled to a maximum temperature of 4°C.

After a sample is put into the ice chest, the COC form will be filled out. Reusable sampling equipment (if used) will be decontaminated before moving to the next sampling location. Surface sample locations will be recorded using a hand-held GPS unit.

MEC visual surveys will not be conducted at these three MRP sites as MEC is not suspected to be present.

4.5 SITE UTILITY CLEARANCE AND DIGGING PERMIT

Before any intrusive drilling or subsurface sampling activities commence at the site, utility maps of the facility will be obtained from the Navy and thoroughly reviewed. The Navy will be responsible for providing clearance for utilities. TtNUS will be responsible for pre-marking proposed sampling locations or indicating areas for sampling. No intrusive activities (i.e., drilling or hand augering) will occur until the proposed areas intended for drilling have been completely cleared in accordance with TtNUS SOP S1 (HS-1.0).

Predefined coordinates based on the sample location maps will be loaded into a high-resolution GPS unit, and the GPS will be used to navigate to each of the sampling locations. Locations will be moved as necessary to provide proper utility clearance but may also be moved to address health and safety issues, site access issues (proper and safe maneuvering of the drill rig, orientation of the drill rig with respect to current site activities, etc.), and to avoid unmovable surface obstructions. If any location needs to be moved by a distance of 10 feet or more, TtNUS personnel will contact the TtNUS Project Manager and

Navy prior to field activities to determine whether the point should be eliminated or whether an acceptable alternative location can be found.

4.6 INVESTIGATION EQUIPMENT AND PROCEDURES

Soil borings and temporary wells will be completed by direct-push methods. A Geoprobe® or other similar direct-push technology (DPT) equipment will be used to collect subsurface soil samples and to install temporary groundwater monitoring wells. Handaugering will also be employed for surface and shallow subsurface soil sampling, as warranted by site conditions.

A summary list of SOPs follows. These SOPs are included in the QAPP, Attachment 1-B.

S1	HS-1.0	UTILITY LOCATING AND EXCAVATION CLEARANCE
S2	SA-2.5	DIRECT PUSH TECHNOLOGY
S3	SA-1.3	SOIL SAMPLING
S4	GH-1.5	BOREHOLE AND SAMPLE LOGGING
S5	SA-1-1	GROUNDWATER SAMPLE ACQUISITION AND ON-SITE WATER QUALITY TESTING
S6	GH-1.2	EVALUATION OF EXISTING MONITORING WELLS AND WATER-LEVEL MEASUREMENTS
S7	GH-2.9	WELL ABANDONMENT
S8	SA-7.1	DECONTAMINATION OF FIELD EQUIPMENT AND WASTE HANDLING
S9	SA-6.3	FIELD DOCUMENTATION
S10	USEPA REGION 1 LOW-FLOW PURGING AND SAMPLING METHODS	
S11	USEPA REGION 1 DRAFT CALIBRATION OF FIELD INSTRUMENTS	
S12	SA-1.2	SURFACE WATER AND SEDIMENT SAMPLING
S13	GH-2.8	GROUNDWATER MONITORING WELL INSTALLATION
S14	GH-1.3	SOIL AND ROCK DRILLING METHODS
S15	XRF SOP	STANDARD OPERATING PROCEDURE FOR FIELD PORTABLE X-RAY FLUORESCENCE

4.6.1 Direct-Push Methods for Surface and Subsurface Soil

The procedure for soil sampling using DPT is included in TtNUS SOP S2 (SA-2.5). Hand-augering and/or use of a trowel will be employed where locations are not accessible for a drill rig and/or where only shallow subsurface samples are planned (as is the case for the Skeet Range and Topsham Skeet Range). Hand augering will be conducted in accordance with SOP S14 (GH-1.3).

All surface soil samples at these three non-MEC sites, the Skeet Range, Topsham Skeet Range, and Machine Gun Boresight Range, will be discrete samples. Subsurface soil samples will also be collected as discrete samples.

A new acetate liner will be advanced at each boring location using a four foot Macro-Core® or similar sampler and in accordance with procedures outlined in TtNUS SOP S3 (SA-1.3). The soil core will be visually inspected and logged by the field geologist, noting soil texture, grain size (sand, silt or clay), color (and any unusual discoloration), qualitative moisture content, and classification according to the Unified Soil Classification System (USCS). The field geologist will identify the soil depositional environment. Soil samples will be collected and logged in accordance with procedures outlined in TtNUS SOP S3 (SA-1.3) and S4 (GH-1.5).

All borings not converted into temporary well points will be backfilled with bentonite chips and patched with cement, cold-patch asphalt, or soil at grade to match the surrounding area. Care will be taken to return each location to its original condition after sampling. Each boring location will be revisited after backfilling is completed to re-apply soil, cement grout, or cold-patch, if necessary. After a boring has been sampled and backfilled, it will be identified by a wooden stake, driven into the center of the boring. If two or more drive attempts are made to collect adequate sample volume, the stake will be driven in the center of the area represented by those borings. The locations and distances between the borings will be clearly described in the sample collection logs. The stake will have brightly colored flagging attached to increase visibility, and will be labeled using a waterproof marker with the soil boring number.

All direct-push equipment will be steam-cleaned prior to the start of the field investigation, decontaminated between each borehole to prevent cross-borehole contamination, and decontaminated prior to leaving the site.

4.6.2 Temporary Monitoring Point Installation

Borings for installation of temporary monitoring wells will be advanced using DPT and in accordance with TtNUS SOP S2 (SA-2.5) and the following methodology. A GeoProbe® or similar DPT will be used to advance two-inch outside diameter Macro-Core® or similar sampler of two-foot length to several feet below the local water table. If cobbles or thick gravel layers are encountered and prohibit advancement of the sampler using DPT, or if open boreholes can not be maintained that allow installation of temporary monitoring wells, a Geoprobe ® DT-325 dual-tube sampler (3.25-inch outside diameter) will be used to advance the soil boring.

The Macro-Core® or DT-325 dual tube sampler will be removed from the borehole, and a temporary polyvinyl chloride (PVC) well will be installed. A Geoprobe® pre-pack PVC well screen will have an inside

diameter of approximately 1.0 inches, a slot size of approximately 0.01 inches (slot size openings), and a pre-pack well screen length of approximately 10 feet (outside diameter of 2.5 inches). The well screen will be installed in the upper surficial aquifer and will intersect the estimated depth of the groundwater table, based on the presence of water observed during soil lithologic sampling. The well screen will be capped at the bottom. PVC riser pipe (Schedule 40) will be attached to the screen to extend the well above grade. The screen and riser pipe will be flush jointed. Sand filter pack will be placed above the pre-pack well screen to a height of approximately one foot, followed by a bentonite seal (one foot minimum thickness) installed above the sand pack. A pre-manufactured sand pack is recommended because the boring size is expected to be too small for the annulus between the well screen and the boring to allow free flow of sand. Only 100 percent, certified pure, sodium bentonite will be used for well construction. The annulus above the bentonite seal will be backfilled with bentonite rock chips to approximately 1 feet bgs, followed by a 0.5-foot thick drainage layer, and capped with native materials.

Each temporary well will be developed by pumping and surging to remove sediments from inside the casing and within the sand pack which surrounds the well screen. Each new well will be developed after waiting approximately 12 hours (minimum) after installation is complete. Each of the wells will be developed for approximately one-half hour.

The water levels in the temporary wells will be allowed to stabilize for 12 hours or more after development, depending on the permeability of the formation, before sampling. The temporary wells will be protected with a lockable expansion cap and locked with keyed-alike locks when left unattended to minimize the introduction of particles or contaminants into the well from the surface.

Each temporary monitoring well will be developed by pumping and surging. Field parameters (e.g., pH, temperature, specific conductance, ORP, turbidity) and groundwater levels will be monitored during well development. Well development will continue up to 2 hours per well until the discharge water meets 10 Nephelometric Turbidity Units (NTUs).

After TtNUS evaluation of water level data and analytical results and subsequent concurrence with the Navy and regulatory agencies, some of the temporary wells may be abandoned. For these wells to be abandoned, the well riser and screen will be pulled from the borehole, and the borehole will be backfilled with bentonite/cement grout from the bottom of the borehole to the ground surface. If possible, the bottom of the well will be knocked out and used as a tremie pipe for emplacement of the grout to avoid collapse of the borehole. The grout will be emplaced from the bottom of the hole to the surface and consist of a cement-bentonite slurry. The well riser and screen will be steam cleaned at the decontamination pad before disposal. The surface at each temporary well location will be restored to its original condition (vegetated, asphalt, or concrete).

Boring logs will be prepared for each temporary well. The boring logs will record installation methods, depths of installation, abandonment details, and other pertinent information. A map of the temporary monitoring well locations and appropriate data from the boring logs will also be recorded into the field logbook notes.

4.6.3 Surface Water and Sediment Sampling

Surface water and sediment samples will be co-located and collected in accordance with TtNUS SOP S12 (SA-1.2). Sediment samples will be collected from zero to 6-inch depth.

Most of the sediment samples will be discrete. However, at NASB Skeet Range, three of the four surface water samples (from the impoundment ponds) will be composite samples. The composite surface water samples will be collected in the middle of the water body and will be collected from the mid to deep portion of the water column (two to three depths) where there is direct interaction with contaminants in the sediment. Sediment samples collected at this site will be a composite of two samples to help improve characterization. The samples will be collected by field personnel using waders or, if to deep, a small watercraft to access the middle of the water body will be used.

Surface water quality parameters [pH, specific conductance, temperature, oxidation/reduction potential (ORP), salinity, and dissolved oxygen] will be measured using a YSI Model 650 MDS water quality meter (or equivalent) and flow-through cell. Turbidity will be measured using a Lamotte 2020 (or equivalent) and the turbidity measurement will be performed from a sample collected from a “T” connector before the flow-through cell.

Calibration and standards checks will be conducted on the water quality meter in accordance with the manufacturer’s recommendations and SOP S11 (USEPA Region I methods). The calibration procedure for all parameters except ORP will use, at a minimum, two standards to bracket the measurement range. The ORP measurement uses one standard (Zobell standard). The flow-through cell will be decontaminated before initiation of well purging at each well. Precautions will be taken to prevent air entrapment and/or air leaks in the purging system so that potential problems with stabilizing dissolved oxygen are minimized. Precautions will include: (1) taking care to fill the entire flow-through cell with water, while minimizing air entrapment, before initiating purging and (2) maintaining a full cell of water by pinching the discharge line shut and elevating the discharge at the end of the tubing from the pump, above the cell.

4.6.4 XRF Sampling of Lead in Soil

X-Ray Fluorescence (XRF) spectrometry investigations will be conducted at two sites – NASB Skeet Range and Topsham Skeet Range. Discrepancies and/or lack of information and/or site disturbance on these two sites have led to uncertainty on the site boundaries. Therefore, XRF investigations may aid in establishing the locations for MC sampling; in particular, by obtaining a greater number of real time samples resulting in a higher level of confidence in identifying area(s) of maximum contamination for fixed-based laboratory analysis. At each of the two sites, a grid will be established over the site area and samples will be analyzed in the field for lead by XRF in the field, beginning with the grid squares expected to have the maximum concentrations and working outward. TtNUS will conduct the XRF investigation at the Topsham Skeet Range and NASB Skeet Range.

An XRF investigation was conducted by the USEPA at the Machine Gun Boresight Range on May 1, 2008 and results are provided in Appendix B. At the Machine Gun Boresight Range, the disposition of the former berm is unknown and a dirt pile on site may or may not be the remnants of the former berm. The existing dirt pile boundary will be mapped in the field using a hand-held surveying Global Positioning System (GPS) device. The distance to the dirt pile has been measured but the entire outline of the dirt pile should be mapped. Maximum concentrations are expected at the existing dirt pile and/or the former berm location based on historical aerial photographs. Between 30 and 90 soil samples were planned for collection and screening via XRF. The minimum 30 samples may be sufficient if it can be established that maximum lead concentrations present exceed MEDEP RAGS residential standard of 375 mg/kg (USEPA action level of 400 mg/kg) and the source area locations (i.e., former berm location, existing dirt pile, and area in between) are consistent with those of the current-day conceptual site model. Otherwise, up to 90 samples were to be collected via XRF to meet the objective. During field analysis, the XRF lead data were reviewed on a ½ day basis (approximately every 15 samples) to support the continuing effort of determining sample locations. As shown in Appendix B, of the 50 XRF samples collected (which excludes 6 duplicate XRF samples and 6 fixed-base laboratory confirmation samples), only 3 had lead concentrations exceeding the MEDEP RAGS 375 mg/kg residential standard. These XRF samples included two samples at location MBR-SB-01 (surface soil at 1640 mg/kg and 12-16” interval at 610 mg/kg) and location MBR-SB-22 at 401 mg/kg. These results will be further assessed in combination with the planned soil sampling results in evaluating site contamination.

At Topsham Skeet Range, very little information is available on the actual operations of the range and aerial photographs do not resolve this issue. Any remaining historical features, such as remaining wooden posts, etc., will be mapped via a hand-held surveying GPS device. Maximum concentrations are expected within the shotfall zone, working radially outward from the center. For both the Topsham Skeet Range and the NASB Skeet Range, all field visual observations including physical observation of lead shot, bullets, and pieces of clay targets will be recorded on sample log sheets. Prior to collection of the

XRF samples, a site "walk-over" will be conducted by the FOL to identify any obvious areas of fragment/lead shot accumulation. Additional discretionary surface soil samples may be collected based on visual observation of accumulated fragments or shot in the area to be sampled. Emphasis will be placed on collecting samples required to delineate contaminated areas. Additional "step-out" samples will also be collected at the boundaries of the area to be sampled if lead concentrations greater than the field action level of 300 mg/kg are detected based on the results of the XRF field screening analysis of the initial surface soil samples. An XRF field action level of 300 mg/kg will be used as the discriminator between contaminated and uncontaminated locations, which corresponds to the MEDEP action level of 375 mg/kg reduced by a 20 percent margin of error to minimize the potential that contaminated areas go undetected. The margin of error was selected based on recent experiences with field XRF analyses at other small arms ranges.

For the skeet ranges, the initial horizontal study boundary encompasses the area that, based on historical information, is most likely to have been impacted by site activities as shown on the sample location maps. Lateral expansion of this horizontal study boundary may be necessary if the XRF concentration in the samples collected along this boundary exceed 300 mg/kg. The study area has the following three sub-zones: the firing point, an area downrange of the firing line where clay targets (i.e., missed targets) and clay target fragments actually hit by shotguns would have accumulated (spent lead shot would also be present in this area), and the shotfall zone that is the downrange area where the greatest concentrations of lead shot would accumulate although target fragments are unlikely to be present. The sampler will try to ensure good spatial coverage of the targeted contamination areas when selecting XRF sampling locations to validate the sampling design; areas outside of those expected to be contaminated will also be sampled and if the sampling design is correct, it is anticipated that the additional locations will exhibit lesser, and perhaps even non-detectable, concentrations of lead than are found in the targeted contamination area.

Between 30 and 90 surface soil samples are planned for collection and screening via XRF at each of the two skeet ranges. Surface soil samples will be collected from the 0 to 3-inch bgs interval. The XRF samples will be discrete in order to "match" the discrete sampling methodology for the samples to be sent to the fixed-base analytical laboratory. The minimum 30 samples may be sufficient if it can be established that maximum lead concentrations present exceed the MEDEP level of 375 mg/kg and the source area location (i.e., shotfall zone) is consistent with that of the current-day conceptual site model, because the decision to move forward to an RI would be evident at this point and the data collected could be built upon during the RI. Otherwise, up to 90 samples will be collected via XRF to meet the objective. Considering the changed orientation of the NASB Skeet Range over the years, the number of XRF samples is expected to be on the upper end (90 samples) with concentration on the overlap area of the

two historic shotfall fans. During field analysis, the XRF lead data will be reviewed on a ½ day basis (approximately every 15 samples) to support the continuing effort of determining sample locations.

The XRF results will aid in locating the samples for MC fixed-based laboratory analysis, which will be collected at the locations of highest concentrations established by the XRF investigation. The XRF results will be used for screening purposes only and so no confirmation sampling for the XRF samples are planned prior to collecting the MC soil samples for fixed-based laboratory analysis. However, all XRF sample data will be later correlated with fixed-base laboratory data, when received. Performance criteria (correlation coefficients) for correlation of lead XRF data to fixed-base laboratory data for lead is based on a correlation coefficient greater than or equal to 0.65 and less than or equal to 1.00. These correlations will be used to translate field XRF valued into equivalent laboratory lead concentrations. Correlation analyses are not anticipated to be generated for the other metals.

Planned soil sampling locations shown on the figures may be adjusted based on XRF results. After collection of all XRF samples and confirmation or adjustment of the shotfall zone, the actual samples for fixed-base laboratory analyses will be determined in accordance with the sampling design shown on the figure. Soil sampling methodology is discussed in Section 4.6.1. All sample locations will be marked with a wooden stake or brightly colored pin flag indicating the sample location. Coordinates will be determined by GPS at each individual sample location which will allow for future repeatable investigations or guide in any remedial action. All sample location markers will be removed prior to demobilization from the site.

Each soil sample collected for XRF analysis will be collected with a hand-held XRF unit. Each sample will be removed and placed in either an unused, labeled, large zip-lock bag (gallon sized) or a decontaminated stainless steel mixing bowl. The soil will be thoroughly mixed using a kneading motion if in a zip-lock bag or with a stainless steel spoon (or equivalent) if in a bowl. Any gravel, rocks, or other large debris items will be removed during the mixing procedure. Any bullets, bullet fragments, or lead shot observed in a sample will be recorded in a logbook and then removed so as not to bias lead results. Mixing will be performed for approximately 2 minutes or until the sample material appears to be well mixed. After the soil sample is thoroughly mixed, the soil will be transferred to an unused, labeled large zip-lock bag (if not already) and stored in a cooler that is always maintained under custody procedures. A representative aliquot of the homogenized soil (approximately 1.5 cubic inches) will be collected and placed in an unused smaller zip-lock bag. The large zip-lock bag will be returned to the cooler. The representative aliquot will be dried in a toaster oven at a temperature not to exceed 150 degrees Celsius. After the sample is thoroughly mixed and dried, the sample will be analyzed with XRF in accordance with the TtNUS XRF SOP (S-15). Three measurements per aliquot are planned and the average of the three recorded values will be calculated and reported along with the three measurements on a daily sample

analysis form. After each day of XRF analysis, the data file generated by the XRF spectrometer will be downloaded for review by the FOL and the Project Manager.

Upon validation, laboratory lead concentrations that are co-located with field XRF lead concentrations will be compared through a correlation plot. The field data will be plotted against the laboratory data, and a linear least-squares fit will be applied to generate an estimate of the "best" line through the data. If the data set exhibits more than one region where there appears to be a linear fit to the data but the regions have different slopes, the data may be subdivided into separate regions for fitting. No more than three separate regions of the correlation will be fit in this manner, and as few regions as possible will be used to adequately represent a linear fit. If a correlation coefficient (commonly denoted as "r") of 0.65 or greater is obtained for a region, the fit will be considered adequate to additionally translate and use field XRF results to the equivalent laboratory lead concentrations with confidence. A correlation coefficient less than 0.65 will be considered unacceptable for making this translation and reliance on the laboratory data only will be used. An ideal correlation coefficient would be 1.0. The use of subdivisions of the overall concentrations range to represent a best linear fit will be most important for concentrations in the range of about 300 to 450 mg/kg because the 375mg/kg risk screening value for lead is within this range. XRF data collected for this project is considered supplemental data and will be added to the soil data set only if there is good correlation; otherwise, the fixed-base laboratory data will stand on its own.

4.7 GROUNDWATER FIELD ACTIVITIES

4.7.1 Groundwater Purging

Low-flow purging and sampling is being implemented because this method will provide the least disturbance to the surrounding formation (i.e., less turbulence in sampling and hence less turbidity), allowing for a more representative groundwater sample to be collected. Low-flow purging and sampling activities will not begin until at least 12 hours after the temporary monitoring wells have been developed. An adjustable-rate peristaltic pump will be used for purging and sampling purposes. New silicone pump tubing and polyethylene tubing will be attached to the pump and inserted near the mid-point of the well screen before each well is purged, so as not to disturb any sediment that may be located near the bottom of the well. Low-flow purging and sampling techniques will be utilized in accordance with TtNUS SOP S5 (SA-1.1) and general accordance with TtNUS SOP S10 (USEPA Region I methods).

During well purging, the water level in each well will be monitored every five to 10 minutes as purging occurs. A drawdown of less than 0.3 feet will be targeted. Initially, the pumping rate will be set at approximately 0.3 liters per minute, or lower if needed. The pumping rate will be reduced if turbidity is greater than 10 NTUs after all other field parameters have stabilized. The measured pumping rates and groundwater level data will be recorded on the well purging log sheets located in Attachment 1-C.

Water quality parameters [pH, specific conductance, temperature, oxidation/reduction potential (ORP), salinity, and dissolved oxygen] will be measured during purging using a YSI Model 650 MDS water quality meter (or equivalent) and flow-through cell. Turbidity will be measured using a Lamotte 2020 (or equivalent) and the turbidity measurement will be performed from a sample collected from a "T" connector before the flow-through cell. Water quality measurements will be recorded on the well purging log sheet every five to 10 minutes.

Calibration and standards checks will be conducted on the water quality meter in accordance with the manufacturer's recommendations and SOP S11 (USEPA Region I methods). The calibration procedure for all parameters except ORP will use, at a minimum, two standards to bracket the measurement range. The ORP measurement uses one standard (Zobell standard). The flow-through cell will be decontaminated before initiation of well purging at each well. Precautions will be taken to prevent air entrapment and/or air leaks in the purging system so that potential problems with stabilizing dissolved oxygen are minimized. Precautions will include: (1) taking care to fill the entire flow-through cell with water, while minimizing air entrapment, before initiating purging and (2) maintaining a full cell of water by pinching the discharge line shut and elevating the discharge at the end of the tubing from the pump, above the cell.

Purging will continue until all of the parameters have stabilized to the criteria listed below and the minimum purge volume (one well volume) has been removed from a well. Stabilization criteria for the water quality parameters are as follows:

- pH $\pm$ 0.1 standard units
- turbidity $\pm$ 10 percent for values greater than 5 NTU
- specific conductance $\pm$ 3 percent
- temperature $\pm$ 3 percent
- ORP $\pm$ 10 millivolts
- dissolved oxygen $\pm$ 10 percent

If the parameters have not stabilized within two hours, then sampling will be initiated. The final values of water quality parameters measured just before sampling commences will be recorded on the groundwater sample collection log sheet located in Attachment 1-C.

4.7.2 Temporary Monitoring Well Sampling

After purging is completed, the flow-through cell will be disconnected from the pump discharge tube and samples will be collected directly from the discharge tube of the pump. Sample containers will be filled [following the method detailed in TtNUS SOP S2 (SA-2.5)] by allowing the groundwater to flow gently down the inside of the container with minimal turbulence. Except for perchlorate samples, the bottles should be filled completely with no head space remaining in the bottle then capped. For perchlorate samples only, the sample bottles will be left about one third empty; filtration of the sample will be conducted at the analytical laboratory and not in the field. Leaving the sample bottle unfilled will prevent the establishment of anaerobic conditions in the sample bottle that are conducive to perchlorate decomposition. No preservatives are necessary for perchlorated bottles. Perchlorate samples will be shipped within 12 hours of sample collection and the laboratory will filter immediately upon receipt, which is targeted to occur within 24 hours of samples collection.

As applicable, all sample bottles will have the appropriate type and amount of preservative already added to the containers, prior to arrival at the site (i.e., preservatives are added to the bottles by the analytical laboratory before shipping to the site). TtNUS SOPs S5 (SA-1.1) and S10 (USEPA Region I methods) will be followed for purging and sampling activities.

4.7.3 Water-Level Measurements

One round of water levels will be manually measured in all monitoring wells where groundwater samples were collected for a given site. If groundwater investigation is warranted for the Skeet Range, water-level collection will include the two existing monitoring wells selected for sampling (MW-NASB-076 and MW-NASB-078). If groundwater investigation is warranted for the Topsham Skeet Range, three nearby monitoring wells (MW1, MW2, and MW3) will be included for water-level collection, even though groundwater samples are not to be collected from these wells. These measurements will be made using an electric water-level indicator capable of 0.01-foot accuracy in accordance with TtNUS SOP S6 (GH-1.2). Water levels will be recorded on Groundwater-Level Measurement log sheets (Attachment 1-C).

4.7.4 Temporary Well Abandonment

Select temporary wells will be properly abandoned after evaluation of water-level data and analytical results, in concurrence with the Navy and regulatory agencies, but only after evaluating the SI analytical and water-level results to ensure future use of a given well is not warranted. Abandonment will comply with TtNUS SOP S7 (GH-2.9) and Maine Chapter 405 Section 5 (H). For abandonment purposes, the temporary well riser and screen will be pulled from the borehole, and the borehole will be backfilled with

bentonite/cement grout from the bottom of the borehole to the ground surface. If possible, the bottom of the well will be knocked out and used as a tremie pipe for emplacement of the grout to avoid collapse of the borehole. Each backfilled boring will be checked the following day for subsidence of the grout and finished smooth to existing grade. The grout sealant will consist of sodium bentonite.

The well riser and screen will be steam cleaned at the decontamination pad before disposal.

4.7.5 Temporary Well Completion to Permanent Well

Select temporary wells may be converted to permanent monitoring wells after evaluation of water-level data and analytical results in concurrence with the Navy and regulatory agencies. Well completion will be conducted in accordance with TtNUS SOP S13 (GH-2.8), and will include a protective casing with lock, a concrete pad, and bollards where stick-up wells are completed in the vicinity of roadways.

4.8 SAMPLE HANDLING AND MANAGEMENT

The following sections outline the procedures that will be used by field personnel to document project activities and sample collection procedures. Detailed and accurate documentation is necessary in order to ensure data integrity.

4.8.1 Sample Custody

Documentation of field observations will be recorded in a field logbook and/or field log sheets including sample collection logs, boring logs, and monitoring well construction logs found in Attachment 1-C. Field logbooks utilized on this project will consist of a bound, water-resistant logbook. All pages of the logbook will be numbered sequentially and observations will be recorded with indelible ink. Field logbooks will be maintained according to SOP S9 (SA-6.3). Field sample log sheets will be used to document sample collection details, and other observations and activities will be recorded in the field logbook. Instrument calibration logs (Attachment 1-C) will be used to record the daily instrument calibration.

For sampling and field activities, the following types of information will be recorded as appropriate:

- Site name and location
- Date and time of logbook entries
- Personnel and their affiliations
- Weather conditions
- Activities involved with the sampling
- Subcontractor activity summary

- Site observations including site entry and exit times
- Site sketches made onsite
- Visitor names, affiliations, arrival and departure times
- Health and Safety issues including personal protective equipment and clothing (PPE)

The following sections outline the information that will be documented in the field according to the media to be sampled and the activities to be performed.

Sample collection information for all media (soil, surface water, sediment, and groundwater) will be recorded on TtNUS sampling logs. The field logbooks and sample log sheets will remain on site for the duration of the investigation. After the investigation, the field sampling log sheets will be organized by date and media and placed in the project file. The field logbooks for this project will be used only for this site, and will also be categorized and maintained in the project file 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 given titles based on date and activity.

Sample custody procedures are designed to provide documentation of preparation, handling, storage, and shipping of all samples collected. Field chain-of-custody procedures are described in TtNUS SOP S9 (SA-6.3). Laboratory chain-of-custody procedures are described in the laboratory SOPs.

Integrity of the samples collected during the site investigation will be the responsibility of identified persons from the time the samples are collected until incorporated into the final report. Stringent chain-of-custody procedures will be followed to document sample possession.

4.8.2 Field Custody

The TtNUS field operations leader (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.

Sample logs or other records will be signed and dated by the persons making the entries.

COC forms will be completed to the fullest extent possible before sample shipment. They will include the following information: project name, sample identification, date and time samples collected, analyses to be conducted, matrix, type of sample, grab or composite designation, preservative (if any), sample depths, and name of sampler. Sample salinity will also be noted on COC forms to aid the analytical laboratory in establishing dilution requirements for metals analyses where salinity could present an interference concern.

These forms will be filled out in a legible manner, using waterproof ink, and will be signed by the sampler. Similar information will be provided on the sample label, which will be securely attached to the sample bottle. The label will also include the general analyses to be conducted. In addition, sampling forms will be used to document collection, filtration, and preparation procedures. Copies of all forms used during field activities are provided in the SOPs.

4.8.3 Transfer of Custody

The following procedures will be used when transferring custody of samples.

Sample coolers will be custody-sealed for security according SOP S9 (SA-6.3) and accompanied by a COC form. When transferring samples, the individuals relinquishing and receiving them will sign, date, and note the time on the COC form. This record documents the sample custody transfer from the sampler to the laboratory, often through another person or agency (common carrier). Upon arrival at the laboratory, internal sample custody procedures will be followed as defined in the laboratory SOPs.

Prior to shipment to the laboratory for analysis, samples will be properly packaged. Individual custody records will accompany each shipment. Shipping containers will then be sealed for shipment to the laboratory. The methods of shipment, courier name, and other pertinent information will be entered in the "remarks" section of the custody record.

All shipments will be accompanied by the COC form identifying the contents. The original record will accompany the shipment and a copy will be retained by the field sampler.

Proper documentation (i.e. airbills) will be maintained for shipments by common carrier.

4.8.4 Sample Shipment Procedures

The following procedures will be followed when shipping samples for laboratory analysis.

Samples requiring cooling to 4°C will be promptly chilled with ice or Blue Ice and will be packaged in an insulated cooler for transport to the laboratory. A temperature blank will be included in each cooler to be used as a temperature indicator. Each temperature blank will be clearly identified by the field sampling team. Ice will be sealed in containers to prevent leakage of water. Samples will not be frozen.

Only shipping containers that meet all applicable state and federal standards for safe shipment will be used.

The field COC form will be placed inside the shipping container in a sealed, plastic envelope. Shipping containers will be sealed with nylon strapping tape, and custody seals will be signed, dated, and affixed, in a manner that will allow the receiver to quickly identify any tampering that may have occurred during transport to the laboratory.

Samples to be delivered to the laboratory will be made by a public courier. After samples have been collected, they will be sent to the laboratory within 72 hours. Under no circumstances will sample holding times be exceeded.

4.8.5 Laboratory Sample Custody

To ensure the integrity of a sample from collection through analysis, it is necessary to have an accurate, written record that traces the possession and handling of the sample. This documentation is referred to as the COC form.

A sample is under custody if:

- The sample is in the physical possession of an authorized person
- The sample is in view of an authorized person after being in his/her possession
- The sample is placed in a secure area by an authorized person after being in his/her possession
- The sample is in a secure area, restricted to authorized personnel only

When samples are received, the COC is signed and dated to acknowledge sample receipt. The sample custodian must examine the shipping containers and verify that the correct number of containers was received. The shipping containers are then opened and the enclosed sample paperwork is removed. Samples are removed from the shipping containers and the bottle condition, temperature blank, and sample salinity must be noted. The information on the COC, the airbill, the containers, and the laboratory request is reviewed to note any discrepancies.

The laboratory will be required to fax the COC forms and sample log-in information to the TtNUS Project Manager (PM) after every shipment.

All samples received by the laboratory must be stored at 4°C until analysis. Laboratory holding times are specified by the contract and presented in the QAPP.

4.9 EQUIPMENT DECONTAMINATION

TtNUS will obtain pre-cleaned sample containers for laboratory analyses. These containers will meet the requirements of the USEPA Specification and Guidance for Contaminant-Free Sample Containers (OSWER Directive No. 9240.0-05A).

Equipment decontamination will follow the procedures described in the TtNUS SOP S8 (SA-7.1). Non-disposable equipment items that come in contact with the media to be sampled and will require decontamination and include:

- stainless steel mixing bowls and spoons (in contact with soils)
- DPT tooling (in contact with soils)
- water level indicators (in contact with groundwater)

If the equipment is new, the initial cleaning will consist only of a soapy water wash followed by a tap water and distilled water rinse. Disposable sampling materials (e.g., tubing for peristaltic pumps) that are individually packaged from the factory will not require decontamination before sampling.

To ensure that sampling equipment has been decontaminated properly, equipment rinsate blanks will be collected and analyzed.

4.10 INVESTIGATIVE-DERIVED WASTE MANAGEMENT

Five types of investigative-derived waste (IDW) will be generated during this investigation that could be potentially contaminated: soil cuttings, well development and purge water, DPT decontamination wastewater, sampling equipment decontamination wastewater, and PPE. Based on the historical site activities and types of contaminants present, none of these IDW materials is expected to present a significant risk to human health or the environment if properly managed.

Soil cuttings and excess soil core material resulting from the DPT drilling activity will be placed in 55-gallon labeled, sealable steel drums. The soils from each site will be segregated, so that if the soil analyses show that soils from one or more of the sites are clean, these soil materials can later be retrieved and disposed of as clean soil. The drums of soil IDW will be labeled, sealed, and transported to a central location designated by the Navy. Proper disposal of these wastes will be performed by the Navy once the analytical results of the soil samples are received from the laboratory and reviewed.

Well development waters and purge waters will be pumped into 55-gallon sealable drums, which will be properly labeled and transported to a central location to be designated by the Navy. Proper disposal of

these wastes will be performed by the Navy once the analytical results are received from the laboratory and reviewed.

4.11 FIELD ANALYTICAL METHOD REQUIREMENTS

4.11.1 Water Quality Meters

4.11.1.1 Field Analytical Method Requirements – Water Quality Meters

Field measurements of pH, dissolved oxygen, specific conductance, salinity, temperature, turbidity, and ORP will be collected during low-flow purging for well sampling. Specific conductance, pH, temperature, and turbidity measurements will be collected during well development as described in TtNUS SOP S5 (SA-1.1). These measurements will be made using a YSI Model 6820 (or equivalent) for all parameters except turbidity. A Lamotte 2020 (or equivalent) will be used for the turbidity measurement. Each of these measurements will be performed using an electrode or sensor designed specifically for the parameter being measured. The calibration and use of the meter will be performed per TtNUS SOP S5 (SA-1.1) and SOP S11 (USEPA Region I method).

4.11.1.2 Field Analytical Instrument Calibration – Water Quality Meters

The multiprobe water quality meters will be calibrated per TtNUS SOP S5 (SA-1.1) and SOP S11 (USEPA Region I method). The meter will be calibrated at the beginning of each field day. The calibration will be checked against standard solutions at the end of each field day or more frequently if fluctuations in readings appear to be abnormal. Records of instrument calibration will be maintained on a field instrument calibration log sheet, provided in SOP S5 (SA-1.1). The field geologist will maintain instrument manuals on site. If the meter does not maintain an acceptable level of calibration during a single day's use, then the meter will be calibrated more frequently or it will be replaced.

4.11.2 XRF Spectrometer

4.11.2.1 Field Analytical Method Requirements – XRF Spectometer

Field measurements of lead (in parts per million or ppm) will be performed as described in the TtNUS XRF SOP (S-15) on select soil samples at the three sites. The measurements will be performed using a field-portable XRF unit operated per the manufacturer's specifications. The XRF operator will ensure that the sample identifier, numerical result, and measurement units are appropriately recorded for every sample analyzed. Also required are the name of the person performing the analysis, the date and time of analysis, and a reference to the calibration/standardization on which the measured values are based.

4.11.2.2 Field Analytical Instrument Calibration – XRF Spectometer

The XRF spectrometer will be standardized or calibrated in accordance with the TtNUS XRF SOP (S-15) at the beginning of each field day with a source standard(s) of known concentration(s). This standard(s) will represent anticipated site conditions to ensure appropriate standardization/calibration to anticipated lead levels that will be encountered at the site. If calibration is conducted, records of instrument calibration will be maintained on a field instrument calibration log sheet provided in Attachment 1-C. Standardization records will be recorded in the same manner as the sample analysis results. The field geologist will maintain instrument manuals on site. If the meter does not maintain an acceptable level of standardization/calibration during a single day's use, the meter will be standardized/calibrated more frequently or it will be replaced.

4.12 FIELD EQUIPMENT AND MAINTENANCE

Equipment, instruments, gauges, and other items requiring preventive maintenance will be serviced, when appropriate, by the equipment supplier, the FOL (or designee), and/or drilling subcontractor in accordance with the manufacturer's recommendations. Manufacturer's procedures identify the schedule for servicing critical items in order to minimize the downtime of the measurement system. It will be the responsibility of the FOL to adhere to this maintenance schedule and to arrange any necessary and prompt service required. Service of the equipment, instruments, tools, gauges, etc. shall be performed to the extent possible by the FOL (or designee). If the service requires a more qualified person, then the supplier or the manufacturer will be contacted for assistance. Field logs shall be established by the FOL (or designee) to record maintenance, service procedures, and schedules. Maintenance records will be documented and traceable to the specific equipment, instruments, and gauges.

It will be the responsibility of the FOL (or designee) to inspect all supplies to be used as part of the field program during mobilization and use. Supplies to be inspected include sampling equipment, field meters, and sample containers.

If the FOL encounters any problem with the supplies, he or she will inform the TtNUS PM and the laboratory supplying the containers. The TtNUS PM, in consultation with the Navy PM and QA/QC officer, will instruct the FOL on any corrective actions that should be implemented.

4.13 SURVEYING

Any historical site features encountered during the SI will be mapped using a hand-held surveying GPS device. In particular, additional site mapping will be conducted at two sites – Machine Gun Boresight Range and Topsham Skeet Range. At the Machine Gun Boresight Range, the disposition of the former

berm is unknown and a dirt pile on site may or may not be the remnants of the former berm. The existing dirt pile boundary will be mapped in the field using a hand-held surveying Global Positioning System (GPS) device. The distance to the dirt pile has been measured but the entire outline of the dirt pile should be mapped. Additionally, any areas with concrete debris will be mapped as they may represent the historical flared concrete end walls of the backstop berm. At Topsham Skeet Range, very little information is available on the exact location of the range and aerial photographs do not resolve this issue. Any remaining historical features, such as remaining wooden posts that are reportedly the launching points of the skeet targets, etc., will be mapped via a hand-held surveying GPS device.

The vertical elevations and horizontal locations of all temporary monitoring wells, soil sampling locations, and important site features will be surveyed by a Maine-licensed surveyor at the completion of the field investigation. Existing benchmarks will be used. All horizontal locations will surveyed to the nearest 0.1 foot. All elevations will be measured to the nearest 0.01 foot. The surveyor will determine elevations for the ground surface at all locations. Additionally, for the temporary monitoring wells, the elevation of the reference point (notch at the top of the riser pipe) will be surveyed for each of the temporary monitoring wells. The TtNUS FOL will provide the correct sample location name to the surveyor so that database nomenclature is consistent with location names in the QAPP and the database. Upon receiving the survey data from the surveyor, the TtNUS PM will check the survey data for consistency with the location names in the QAPP and the database.

For remote soil boring locations and/or surface water/sediment locations where land surveying is more difficult, a GPS unit may be used to collect coordinate data. However, all temporary wells will need to be surveyed by a land surveyor because of the required accuracy needed to interpret water-level data.

The GPS unit proposed for use is a Trimble GeoExplorer CE, Geo XT handheld unit. Pathfinder Office software is used to download and post-process the data to achieve submeter horizontal accuracy. Field conditions, such as the number of satellites available at the reading time and density of the tree canopy, dictate the amount of time needed to acquire a reading. Coordinates will be established for each sample location to an accuracy of one meter.

Survey data coordinates (both GPS and land surveyor based) will be provided in the SI report, as well as an electronic version.

4.14 FIELD DOCUMENTATION AND DOCUMENTATION RESPONSIBILITIES

It will be the responsibility of the FOL to secure all documents produced in the field (e.g., sampling logs, calibration forms) at the end of each work day. Copies of all forms used during field activities are

included in the SOPs. Sample logs and COC records will be included as an appendix to the report that will be prepared based on results of this investigation.

At the completion of field activities, the FOL will send the TtNUS PM all field records, data, field notebooks, logbooks, COC forms, sample log sheets, daily logs, etc. The PM will ensure that these materials are entered into the TtNUS document control system in accordance with appropriate administrative guidelines.

Changes in project operating procedures may be necessary as a result of changed field conditions or unanticipated events. A summary of the sequence of events associated with field changes is as follows:

- The FOL will notify the TtNUS PM and TtNUS ME Certified Geologist (CG) of the need for the change.

- If necessary, the PM will discuss the change with the pertinent individuals (e.g., ME CG, Navy PM, TtNUS QA Manager) and will provide a verbal approval or denial to the FOL for the proposed change. The USEPA and MEDEP will be consulted by the Navy of any major scope changes that may occur while fieldwork is ongoing. Communications and correspondences to the Restoration Advisory Board (RAB) will be handled through inclusion on the distribution list or written correspondence and updates at RAB meetings.

- The FOL will document the change on a Task Modification Request form and forward the form to the TtNUS PM at the earliest convenient time.

- The TtNUS PM will sign the form and distribute copies to the Navy PM, TtNUS QA Manager, FOL, ME CG, and project file.

- A copy of the completed Task Modification Request form will be attached to the field copy of the affected document.

The possession of all records will be documented; however, only the project FOL or designee may remove field data from the site for reduction and evaluation.

4.15 FIELD EQUIPMENT

A variety of equipment will be used to perform the field activities for this project. Table 4-1 lists the field equipment and supplies that will be used:

Table 4-1: Field Equipment	
Category	**Equipment**
Soil / Sediment Sampling	Disposable scoops (or similar) and plastic sheeting Hand auger
Groundwater Sampling	Peristaltic pump and disposable tubing
Health and Safety Equipment	Hard hats, safety boots, safety glasses, first aid kit, fire extinguisher, protective clothing, and nitrile gloves
Shipping	Packaging tape, labels, seals, COC forms, ice, zip-top bags, coolers, bubble wrap, and packaging material
Documentation	Daily Quality Control Report (DQCR) forms, field notebook, soil sampling logs, and applicable health and safety forms
Sample Containers	See Table 4-1 of QAPP (Attachment 1)
Decontamination Supplies	Liquinox or Alconox detergent, methanol, potable water, deionized water, scrub brushes, and decontamination tubs/buckets

4.16 LABORATORY ANALYSIS

The analytical methods were selected on the basis of the munitions items known to have been used at the site and include the standard suite of range related analytical parameters to account for unknown items. The standard analytical methods include TAL Metals (SW-846 Method 6010B), explosives (dinitrotoluene) and propellants by SW-846 Method 8330A), perchlorate in groundwater (USEPA Method 6850 with filtration at the laboratory), TOC (Lloyd Kahn Method), nitrates in groundwater (SW-846 9056 or EPA 300), pH (SW-846 9046D), CEC (SW-846 9081), and PAHs (USEPA Method 8270C SIM). Tables 4-2 through 4-4 detail the quantities of analyses to be conducted. The method detection limits for these methods are included in the QAPP (Attachment 1). Laboratory SOPs are provided in the QAPP, Attachment 1-D.

4.17 QUALITY ASSURANCE (QA) / QUALITY CONTROL (QC) SAMPLES

The QA/QC procedures are documented in the QAPP. QA/QC samples are those samples analyzed for the purpose of assessing the quality of the sampling effort and of the analytical data and are further discussed in the following sub-sections.

4.17.1 <u>QC Samples</u>

The QC for analytical samples will be provided in the field through the use of the following QC samples:

- Field duplicates (1 in 10)
- Matrix Spikes (MS)/Matrix Spike Duplicates (MSDs) (1 in 20)

- Upgradient samples
- Field blanks
- Equipment blanks

The QC samples will be handled as regular samples. In order for distinctions to be determined between study areas, the different types of samples will be submitted in separate batches for laboratory analysis. Calibrations and associated QC samples will not be mixed among sample types.

4.17.2 QA Samples

The QA for the analytical samples will be provided in the field through the use of duplicate samples. The QA samples are used to evaluate the contractor's laboratory performance. Duplicate samples are collected as a single sample, which is divided into two equal parts. As shown in Tables 4-2, 4-3, and 4-4, samples will be collected at a rate of at least 10 percent of the field samples collected. Sample collection and preservation requirements are outlined in the QAPP.

Table 4-2: Quantities of Analyses for the Machine Gun Boresight Range

Analysis	Medium	Baseline Samples					
		Field Samples	Field Duplicate	Spikes [a]	Equipment Blanks	Field Blanks	Total
TAL Metals, Propellants	Soil	25	3	2	2	-	32
PAHs	Soil	1	-	-	1	-	2
TAL Metals, Propellants, Perchlorate	Groundwater	5	1	1	2	1	9

[a] One sample indicates one MS/MSD pair (i.e., one laboratory QA/QC sample).

Table 4-3: Quantities of Analyses for the Skeet Range

Analysis	Medium	Baseline Samples					
		Field Samples	Field Duplicate	Spikes [a]	Equipment Blanks	Field Blanks	Total
TAL Metals, PAHs	Soil	28	3	2	3	-	36
Explosives, Propellants	Soil	6	1	1	1	-	9
pH, TOC, CEC	Soil	4	-	-	-	-	4

Table 4-3: Quantities of Analyses for the Skeet Range

Analysis	Medium	Baseline Samples					
		Field Samples	Field Duplicate	Spikes [a]	Equipment Blanks	Field Blanks	Total
TAL Metals, PAHs, Nitrates	Groundwater	7	1	1	1	(b)	10
Explosives, Propellants	Groundwater	1	1	1	-	-	3
TAL Metals and TOC	Sediment	4	1	1	-	-	6
TAL Metals	Surface Water	4	1	1	-	-	6

[a] One sample indicates one MS/MSD pair (i.e., one laboratory QA/QC sample).
[b] Addressed by field blanks at Machine Gun Boresight Range.

Table 4-4: Quantities of Analyses for the Topsham Annex Skeet Range

Analysis	Medium	Baseline Samples					
		Field Samples	Field Duplicate	Spikes [a]	Equipment Blanks	Field Blanks	Total
TAL Metals, PAHs	Soil	20	2	1	2	-	25
Explosives, Propellants	Soil	5	1	1	1	-	8
pH, TOC, CEC	Soil	4	-	-	-	-	4
TAL Metals, PAHs, TOC	Sediment	3	1	1		-	5
TAL Metals, PAHs	Surface Water	3	1	1		-	5
TAL Metals, PAHs, Nitrates	Groundwater	5	1	1	1	(b)	8

Table 4-4: Quantities of Analyses for the Topsham Annex Skeet Range

Analysis	Medium	Baseline Samples					
		Field Samples	Field Duplicate	Spikes [a]	Equipment Blanks	Field Blanks	Total
Explosives, Propellants	Groundwater	1	1	1	-	-	3

[a] One sample indicates one MS/MSD pair (i.e., one laboratory QA/QC sample).
[b] Addressed by field blanks at Machine Gun Boresight Range.

4.18 SAMPLING EQUIPMENT DECONTAMINATION

In an effort to achieve the highest level of QC, one-time-use and disposable sampling equipment will be used whenever feasible. This type of equipment includes sampling gloves, scoops, and pre-cleaned sample jars. Other equipment will be decontaminated as necessary, if used.

5.0 FIELD OPERATIONS DOCUMENTATION

Field documentation of the samples taken is of the utmost importance in assuring QC. Field documentation will include Data Quality Control Reports (DQCRs), field notebooks, soil sampling log forms, sample labels, and COC forms. All field documentation will be completed in indelible ink. Corrections will be made by drawing a single line through the text and legibly writing the correction.

5.1 DQCR

As described in the QAPP, the DQCR will be prepared by the FOL each day that fieldwork is performed, commencing with the first day work is performed on-site. All workdays will be documented in this report throughout the duration of the fieldwork. TtNUS will provide DQCRs to the Base Realignment and Closure Program Management Office Northeast (BRAC PMO NE) PM in the SI Report. A sample DQCR form is included in the QAPP.

5.2 FIELD NOTES

Field notes regarding all sampling and field activities will be kept in a bound notebook with prenumbered pages. Indelible ink will be used for all entries. The field notes will be filled out while the fieldwork is taking place and will include all of the information that is reported on the DQCR forms. In addition, soil sampling log forms will be completed for each sample collected. An example soil sampling log form is included in Attachment 1-C to this SAP.

5.3 SAMPLE NUMBERING SCHEME

All samples taken will employ the following numbering system. This system assures that QC checks originating from the field are blind to the laboratory and that a uniform and consistent numbering system is employed in the field.

NASB – [Site Location designation] – [Sample medium and Sample number] – [Sample date (month)(date)(year)]

The following designations will be used for each medium:

SS = surface soil sample
SB = subsurface soil sample
SD = sediment sample

GW = groundwater sample
SW = surface water sample

The following designations will be used for each site name:

SKT = Skeet Range
TASKT = Topsham Annex Skeet Range
MGBR = Machine Gun Boresight Range

For example:

NASB-SKT-SS03-072509 reflects a surface soil sample collected at location SS03 at the Skeet Range on July 25, 2009.
NASB-MGBR-SB04-03-072109 reflects a subsurface soil sample collected at soil boring location SB04 starting at 3 feet below ground surface at the Machine Gun Boresight Range on July 21, 2009.
NASB-MGBR-GW02-071709 reflects a groundwater sample collected at temporary well MW02 at the Munitions Bunker West on July 17, 2009.

All duplicate samples collected will utilize the following standard designation format:

NASB - [Site Location designation] – [DUP][Sample number] – [Sample date (month)(date)(year)]

For example:

NASB-SKT-DUP01-072509 would reflect the first duplicate sample collected at the Skeet Range on July 25, 2009.

All MS/MSD samples collected will utilize the following standard designation format:

NASB – [Site Location designation] – [Sample medium and Sample number/MS or MSD] – [Sample date (month)(date)(year)]

For example, NASB-SKT-SS02/MSD-052509

All QA/QC samples (trip blanks, equipment blanks, field blanks collected will utilize the following standard labeling format:

NASB – [Site Location designation] - [Sample medium][Sample number/Type of QA Sample] – [Sample date (month)(date)(year)] where EB, TB, and FB will be used for equipment blank, trip blank, and field blank, respectively. Similarly IDW will be used to designate IDW samples.

For example, NASB-SKT-SS02/EB-072509 reflects an equipment blank collected at the Skeet Range on July 25, 2009.

5.4 SAMPLE LABELS

Correct sample labeling and the corresponding notation of the sample identification numbers in the field notebook and on the DQCR COC forms will be utilized to prevent misidentification of samples and their eventual results. All sample labels will be generated electronically or completed legibly with indelible ink. The labels will be affixed to the sample bottle and covered with clear tape.

The sample labels will include the following, at a minimum:

- Project name
- Company name
- Name/initials of the collector
- Date and time of collection
- Sample location and depth
- Analysis required
- Preservatives added if any

5.5 COC

The COC procedures will be in accordance with Navy and USEPA procedures. COC procedures are used to document and track samples from collection through reporting of analytical results and to serve as permanent records of sample handling and shipment. Strict COC protocol will be maintained for all samples collected during this project.

The COC forms will be filled out with indelible ink by the FPM, and any mistakes made will be crossed out with a single line, initialed, and dated.

The information on the COC form will include the following:

- Sample identification numbers
- Date and time of sample collection

- Project name and number
- Number of sample containers
- Analyses required
- Turn around time required
- Preservatives used, if any
- Signatures of all parties who had possession of the samples

The COC forms will be complete for every cooler and will be sealed in a plastic resealable bag and taped to the inside of the lid of the cooler. The FPM will keep one copy of the COC form. The laboratory will then sign the COC form upon accepting the samples for analysis. Copies of the COC forms will be included in the SI Report as an appendix and given to the BRAC PMO NE PM upon completion of the field sampling effort.

6.0 SAMPLE PACKAGING AND SHIPPING REQUIREMENTS

Custody of samples must be maintained throughout the shipment of samples to the selected laboratory.

The following procedures will be used to send samples to the laboratory:

1. Use waterproof high-strength plastic ice chests or coolers only.

2. After filling out the pertinent information on the sample label and tag, place the sample in the container and screw on the lid. Secure the bottle lid with strapping tape.

3. Tape cooler drain shut.

4. Place about 3 inches of inert cushioning material, such as plastic bubble wrap in the bottom of the cooler.

5. Enclose the containers in clear plastic bags through which sample labels are visible, and seal the bag. Place containers upright in the cooler in such a way that they do not touch and will not touch during shipment.

6. Put in additional inert packing material to partially cover sample containers (more than halfway). Place bags of ice or ice-gel packs around, among, and on top of the sample containers.

7. Fill the remaining space in the cooler with cushioning material.

8. If sending the samples by common carrier, sign the COC form under “Relinquished by,” enter the carrier name and air bill number, retain a copy for field records, put the COC form in a waterproof plastic Ziploc bag and tape it to the inside lid of the cooler.

9. If sending the samples by courier or field team shipper, follow the above procedures, but also have the receiving carrier sign under “Received by.”

10. Apply custody seals to the front and back of the cooler, across the lid.

11. Secure lid by taping. Wrap the cooler completely with strapping tape at a minimum of two locations. Do not cover any labels. Attach completed shipping label to top of the cooler. The shipping label will have a return address.

12. Ship the cooler by overnight express or courier to the respective laboratory.

The primary laboratory address and point of contact (POC) are noted below:

Empirical Laboratory, LLC
621 Mainstream Drive, Suite 270
Nashville, TN 37228

ATTN: Janice Shilling
(615) 345-1115

7.0 INVESTIGATIVE-DERIVED WASTE

The production of investigative-derived waste (IDW) will be concentrated on the installation of temporary wells. One sample from drill cuttings along with one decontamination/purge/development water sample will be collected per site to be analyzed for Toxicity Characteristic Leaching Potential (TCLP) to determine disposal requirements. Decontamination fluids from steam cleaning of the drill rig are also expected although other decontamination fluids are not expected since dedicated/disposable field sampling equipment will be used. Used gloves, sampling scoops, and any other disposable sampling equipment or PPE will be double-bagged and disposed of off-site by the Navy as non-hazardous waste.

ATTACHMENT 1: QUALITY ASSUANCE PROJECT PLAN

Quality Assurance Project Plan
for
Site Inspection of Munitions Constituents

Three Munitions Response Sites

Naval Air Station Brunswick
Brunswick, Maine

Naval Facilities Engineering Command Mid-Atlantic
Contract Number N62472-03-D-0057
Contract Task Order 69

June 2009

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TABLE

NUMBER

2-1 Laboratory Data Package Elements

ACRONYMS

%R	Percent Recovery
BACSE	Brunswick Area Citizens for a Safe Environment
BFB	Bromofluorobenzene
BRAC	Base Realignment and Closure
CA	Corrective Action
CCC	Continuing Calibration
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-Term Environmental Action Navy
CLP	Contract Laboratory Program
CRQL	Contract Required Quantitation Limit
CTO	Contract Task Order
DFTPP	Decafluorotriphenylphosphine
DOD	Department of Defense
DOD QSM	Department of Defense Quality Systems Manual
DOE	Department of Energy
DPT	Direct push technology
DVM	Data Validation Manager
EDD	Electronic Data Deliverable
ESI	Electrospray
FOL	Field Operations Leader
FSAP	Final Sampling and Analysis Plan
GC/ECD	Gas Chromatography/Electron Capture Detector
GC/MS	Gas Chromatography/Mass Spectrometery
GIS	Geographical Information System
GPS	Global Positioning System
HASP	Health ad Safety Plan
HPLC	High Performance Liquid Chromatography
HSM	Health and Safety Manager
ICP	Inductively Coupled Plasma
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LQAP	Laboratory Quality Assurance Plan
MC	Munitions Constituents
MDL	Method Detection Limit

MEC	Munitions and Explosives of Concern
MEG	Maximum Exposure Guidelines
MEDEP	Maine Department of Environmental Protection
MMRP	Military Munitions Reposnse Program
MPC	Measurement Performance Criteria
MSD	Matrix Spike Duplicate
MS	Matrix Spike
NAVFAC	Naval Facilities Engineering Command
NAS	Naval Air Station
NE	Northeast
NFESC	Naval Facilities Engineering Service Center
NIRIS	Naval Installation Restoration Information Solution
OSHA	Occupational Safety and Health Association
PAHs	Polycyclic Aromatic Hydrocarbons
PARCCS	Precision, Accuracy, Representativeness, Comparability, Completeness, Sensitivity
PDF	Portable Document Format
PDS	Post-Digestion Spikes
PM	Project Manager
PMO	Program Management Office
PPE	Personal Protective Equipment
PQO	Project Quality Objectives
QA	Quality Assurance
QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limits
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RSD	Relative Standard Deviation
SDG	Sample Delivery Group
SI	Site Inspection
SOP	Standard Operating Procedure
SVOC	Semivolatile Organic Compounds
TAL	Target Analyte List
TCLP	Toxicity Characteristics Leaching Procedure
TOC	Total Organic Carbon

TtNUS	Tetra Tech NUS, Inc.
UFP	Uniform Federal Policy
USEPA	United States Environmental Protection Agency
U.S. Navy	United States Navy
UXO	Unexploded Ordnance
WP	Work Plan
XRF	X-Ray Fluorescence

1.0 PROJECT MANAGEMENT AND OBJECTIVES

This Quality Assurance Project Plan (QAPP) is a companion document to the Site Inspection (SI) Work Plan, originally submitted by Malcolm Pirnie in February 2007 and finalized by Tetra Tech NUS (Tetra Tech or TtNUS). This QAPP is provided as an attachment to the Sampling and Analysis Plan (SAP), also originally submitted by Malcolm Pirnie in February 2007 and finalized by TtNUS. These items constitute the planning documents for the sampling and analysis activities. This QAPP addresses specific protocols for sample collection, sample handling and storage, chain-of-custody, laboratory and field analyses, data validation, and data reporting in relationship to munitions constituents (MC) investigation under the Military Munitions Response Program (MMRP).

This QAPP has been prepared by TtNUS on behalf of the Base Realignment and Closure (BRAC) Program Management Office (PMO) Northeast (NE) under the Comprehensive Long-Term Environmental Action Navy (CLEAN) Contract Number N62472-03-D-0057, Contract Task Order (CTO) 69. This QAPP was generated for and complies with applicable U.S. Navy, Maine Department of Environmental Protection (MEDEP), and United States Environmental Protection Agency (USEPA) Region I requirements, regulations, guidance and technical standards, especially USEPA (1999) and Department of Defense and Department of Energy (DOD/DOE)/USEPA (2005). To comply with DOD/DOE/USEPA (2005) the requisite worksheets have been included as Attachment 1-A of this QAPP.

1.1 TITLE AND APPROVAL PAGE

The title page, which serves as the cover page of this QAPP, identifies the project to which this QAPP applies. The signature page of the Work Plan also serves as the approval page for this QAPP. This information is also included as Uniform Federal Policy (UFP) QAPP Worksheet 1.

1.2 DOCUMENT FORMAT AND TABLE OF CONTENTS

1.2.1 Document Control Format

Document control procedures are used to identify the most current version of the QAPP and to help ensure that only the most current version of the QAPP is used by all project participants. To meet this goal, text, tables, and figures in this QAPP include a header indicating the revision number and date. The footer indicates the page number within the section. Revision 0 with the month and year is used as part of the header and would then be updated with any revisions versions. Any revisions made after submittal of the initial final version will be indicated with appropriate sequential revision number and revision date. Individual pages may be updated. In those cases only the updated pages will reflect a change in revision number and date.

1.2.2 Document Control Numbering System

A document control numbering system will not be used for this QAPP because this project has a distinct document distribution list. QAPP dates and revision numbers will identify which revision is received by each recipient. The QAPP and any revisions, addenda, or amendments will be provided in accordance with the QAPP distribution list (see Section 1.2.1).

1.2.3 QAPP Identifying Information

The QAPP identifying information (found on the QAPP cover and SI Work Plan signature page) identifies the key project participants, and the program for which the current project is being performed. The QAPP identifying information is provided on Worksheet 2. Additional information such as a summary of previous site work, the applicable regulatory program, and scoping session dates may be found on UFP QAPP Worksheet 2.

1.3 DISTRIBUTION LIST AND PROJECT PERSONNEL SIGN-OFF SHEET

1.3.1 Distribution List

The QAPP distribution list includes: Navy personnel, MEDEP, USEPA Region I, Brunswick Area Citizens for a Safe Environment (BACSE), NAS Brunswick personnel, and the subcontracted laboratories [Empirical Laboratories and Columbia Analytical Services]. The distribution list for this QAPP is summarized in UFP QAPP Worksheet 3. Each person listed in UFP QAPP Worksheet 3 will receive copies of the QAPP and subsequent revisions. Revisions may be in the form of a complete document reissue or individual pages that have been changed.

1.3.2 Project Personnel Sign-Off Sheet

UFP QAPP Worksheet 4 provides the project personnel sign-off sheet that will be signed by all personnel working on the project. A signature on this form indicates the person has read this QAPP and is familiar with the tasks they are to perform. The completed sign-off sheet will be maintained in the TtNUS project file under the control of the Project Manager.

1.4 PROJECT ORGANIZATION

1.4.1 Project Organization Chart

A project organization chart depicting the agencies and contracting personnel involved with the review and implementation of the QAPP is shown on UFP QAPP Worksheet 5. The Navy, who is the lead agency for this site, and TtNUS (Navy contractor) will implement this QAPP.

1.4.2 Communication Pathways

Pathways have been established for the exchange of project-related information and for making alterations to project methods, schedules, requirements, and command hierarchy that may be required because of unforeseen or changing circumstances. Communication pathways are depicted in UFP QAPP Worksheet 6.

1.4.3 Personnel Responsibilities and Qualifications

Project personnel responsibilities and qualifications are displayed in UFP QAPP Worksheet 7. Resumes of the TtNUS personnel are available on request through the TtNUS Project Manager.

1.4.4 Special Training Requirements and Certifications

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). The unexploded ordnance (UXO) specialist will be certified in accordance with TtNUS Standard Operating Procedure (SOP) HS-2.0, and UXO safety requirements are addressed in greater detail in the TtNUS Health and Safety Plan (HASP) (separate document reviewed and approved by the Navy).

Field personnel experienced with XRF analysis will conduct the analyses. The field personnel responsible for operation of the XRF may require brief instruction on the use of the specific field equipment that would be conducted prior to the start of sampling and analysis and would be noted in the field log book, as appropriate.

The selected analytical laboratories (Empirical Laboratories and Columbia Analytical Services) have successfully completed the laboratory evaluation process required as part of the Naval Facilities Engineering Service Center (NFESC) Quality Assurance Program and described in "Department of

Defense Quality Systems Manual (DOD QSM)" January 2006. Empirical Laboratories is conducting all except the perchlorate analysis, which will be conducted by Columbia Analytical Services.

1.5 PROJECT PLANNING AND PROBLEM DEFINITION

This section summarizes information on the project planning conducted to develop the problem definition. This section also includes site history and background information.

1.5.1 Project Planning

Project planning meetings were originally conducted by another Navy contractor, Malcolm Pirnie, who was tasked with the original preparation of the Work Plan and SAP, submitted as draft final to the Stakeholders in February 2007. Subsequently, TtNUS assumed ownership of the Work Plan and SAP, following the October 2007 kickoff meeting/site visit where numerous additions were agreed upon that were beyond the scope of the Malcolm Pirnie contract with the Navy.

1.5.2 Problem Definition, Site History, and Background

Site history and background are provided in Section 2 of the SI WP.

This SI MC QAPP is focused solely on sample collection activities directed at MC in soil, groundwater, surface water, and sediment, as applicable, at all three MRP sites.

1.6 PROJECT QUALITY OBJECTIVES AND MEASUREMENT PERFORMANCE CRITIERIA

Problem definition and the entire data quality objective process are described in detail in Section 3.1 of the SAP.

1.6.1 Development of Project Quality Objectives Using the Systematic Planning Process

The project quality objectives (PQOs) are outlined in UFP QAPP Worksheet 11 in terms of type, quantity, and quality of data determined using the systematic planning process.

1.6.2 Measurement Performance Criteria

Measurement performance criteria (MPC) are the Precision, Accuracy, Representativeness, Comparability, Completeness, Sensitivity (PARCCS) parameters (i.e., precision, accuracy, representativeness, comparability, completeness, and sensitivity). The PARCCS parameters are qualitative and quantitative statements regarding the quality characteristics of the data used to support

project objectives and ultimately, environmental decisions. Each of these parameters is described below and is displayed for each matrix, analytical group, and concentration level in UFP QAPP Worksheet 12.

1.6.2.1 Precision

Precision is a measure of the degree to which two or more measurements are in agreement and describes the reproducibility of measurements of the same parameter for samples analyzed under similar conditions.

Unless otherwise specified, two samples collected as close as possible in space and time by repeating the sample collection process are referred to as *co-located* field duplicates. Two samples generated by removing representative portions from a single mass or volume of material are called *subsampling* field duplicates. If two portions or a single sample received by the laboratory are removed from that sample and analyzed, the two portions thus analyzed are called laboratory duplicates. If a single portion of sample is prepared and then the prepared sample is analyzed in duplicate, these repeat analyses are called analytical duplicates. In all cases, if more than two samples (portions) are generated or analyzed, the first portion is called the original sample and the additional portions are referred to as replicates. Duplicates, themselves, are the first replicates.

Field precision is assessed by collecting and measuring field duplicates at a rate of 1 duplicate per 10 environmental samples. Acceptance limits for field duplicate precision are provided in UFP QAPP Worksheet 12. This precision estimate encompasses the combined uncertainty associated with sample collection, homogenization, splitting, handling, laboratory and field storage (as applicable), preparation for analysis, and analysis. In contrast, precision estimates obtained from analyzing duplicate laboratory samples incorporate only homogenization, subsampling, preparation for analysis, laboratory storage (if applicable), and analysis uncertainties. Consequently, the *field precision* estimates [i.e., relative percent difference (RPD) values] should equal or exceed the laboratory precision estimates, on average, for each analyte. If field duplicate precision is significantly different from laboratory duplicate precision, the underlying cause will be investigated to determine whether the observed difference could be artifacts of sampling and analysis.

Laboratory precision Quality Control (QC) samples [i.e., laboratory duplicates for inorganic chemicals and matrix spike duplicates (MSDs) for organic chemicals] will be analyzed with a minimum frequency of 5 percent (i.e., 1 QC sample per 20 environmental samples). Laboratory precision is measured by comparing RPD values to precision control limits specified in UFP QAPP Worksheet 12.

Field duplicate sample results, laboratory duplicate results, sampling procedures, sample transport problems (if any), sample matrix problems (if any), and sample heterogeneity will be considered, as

appropriate, to evaluate the overall data precision. Field duplicate precision, expressed as RPD for soil and sediment samples *(solid matrices)*, must be less than 50% and must be less than 30% for surface water or groundwater *(aqueous matrices)*. For example, field duplicate precision will be compared to laboratory precision. The RPD between a sample or Matrix Spike (MS) (Sample 1) and its duplicate or MSD (Sample 2) is calculated for chemical analyses using the following formula:

$$RPD = \frac{|\text{Amount in Sample 1} - \text{Amount in Sample 2}|}{0.5\,(\text{Amount in Sample 1} + \text{Amount in Sample 2})} \text{ X } 100\,\%$$

1.6.2.2 Accuracy/Bias

Accuracy is the degree of agreement between an observed value and an accepted reference value. This parameter is assessed by measuring spiked samples (e.g., surrogate spikes or MSs) or well-characterized samples of certified analyte concentrations [e.g., laboratory control samples (LCSs)] and by measuring laboratory and field blanks. Accuracy measurements are designed to detect biases resulting from sample handling and analysis. The data validation process during which these evaluations are made is described in Section 4.2. Calculation of accuracy is described below.

Accuracy requirements for field measurements are ensured through control over the sample collection and handling and through routine instrument calibration. Accuracy is also monitored through the use of blanks to detect cross-contamination and by monitoring adherence to procedures that prevent sample contamination or degradation. Equipment rinsate blanks shall be collected for reusable equipment at a frequency of 1 per 20 samples per matrix for this investigation to assess cross-contamination via sample collection equipment. Source water blanks will be collected to monitor the purity of water used to decontaminate sampling equipment. Trip blanks, used to gauge whether cross-contamination is occurring during sample storage and transport, will be placed into each cooler containing samples to be analyzed for volatile organic compounds (VOCs). Accuracy shall also be assured qualitatively through adherence to all sample handling, preservation, and holding time requirements.

Accuracy in the laboratory is measured through the comparison spiked sample, or LCS results to known or calculated values and is expressed as a percent recovery (%R). It is also assessed by monitoring the analytical recovery of select surrogate compounds added to samples that are analyzed by select chromatographic methods. MS and surrogate compound analyses measure the combined accuracy effects of the sample matrix, sample preparation, and sample measurement. LCSs are used to assess the accuracy of laboratory operations with minimal sample matrix effects. Post-digestion spikes (PDSs) are used to assess the accuracy of the analytical measurement on the sample extract or digestate and are only used when initial inorganic MS results are suspect. Each spike sample shall be spiked with representative project target analytes for the analysis being performed to ensure that accuracy measures

are obtained for each target analyte. Spiking concentrations shall equal or approximate the default concentrations detailed in the applicable sample preparation or analysis SOPs. LCS and MS analyses are performed at a frequency no less than 1 per 20 associated samples of like matrix.

The %R for a spiked sample, (including surrogate compounds) is calculated by using the following formula:

$$\%R = \frac{\text{Amount in Spiked Sample} - \text{Amount in Sample}}{\text{Known Amount Added}} \text{ X } 100\,\%$$

LCSs are also analyzed to assess accuracy. The %R calculation for LCS spikes is as follows:

$$\%R = \frac{\text{Experimental Concentration}}{\text{Certified or Known Concentration}} \text{ X } 100\,\%$$

Control charts are plotted by the laboratory for each target analyte and are kept on a matrix- and analyte-specific basis. These control charts are used to calculate the upper and lower quality control limits for evaluating precision and bias.

During data validation, any data not meeting accuracy specifications will be identified to the data user through the use of data qualifiers. The field and laboratory blanks provide indications of the potential for having contaminated samples before or during analysis, respectively. Each type of blank will be evaluated for its impact on the sampling or the analytical processes, as appropriate. Laboratory control standards and check standards indicate whether analyte quantitation is accurate and whether the analytical system was capable of generating results within the project accuracy specifications. MS recoveries indicate and will be evaluated to assess the impact of specific sample matrices on the accuracy of project data.

Nominal limits will be set on a project-specific basis (see Worksheet #28).

1.6.2.3 Sensitivity and Quantitation Limits

Sensitivity is the ability of the method or instrument to detect the target analytes at the level of interest. The quantitation limit will be the low level calibration standard. The Method Detection Limit (MDL) is the minimum concentration of an analyte that can be reported with 99% confidence that the analyte concentration is greater than zero.

Analytical methods were chosen for their ability to achieve the appropriate sensitivity for a specific matrix at this site. Analyte MDLs and quantitation limits were compared to project action limits (e.g., Region IX

Preliminary Remediation Goals) to determine whether the chosen analytical method is sensitive enough to meet the project action limits. Project action limits are the minimum screening number among various available standards and criteria available for a given matrix. UFP QAPP Worksheet 15 displays the comparison of typically achievable MDLs and quantitation limits to project action limits by method and matrix.

A low-level (at or near the quantitation limit) laboratory control sample must be processed and analyzed each day samples are processes and analyzed. These low level laboratory control samples should meet nominal acceptance limits (i.e., Accuracy/Precision Limits).

1.6.2.4 Representativeness

Representativeness is a qualitative term that describes the extent to which a sampling design, collected samples, and associated data reflect the environmental conditions of a site. Sampling and analysis methods and procedures were selected during project planning to provide data representing environmental media at locations selected. Adherence to the standardized sample collection, handling, preparation, analysis, and data reporting procedures ensures that the final data accurately represent the desired populations. To evaluate the representativeness, the actual samples collected will be compared to the samples that were intended to be collected. Furthermore, the data verifications and validations will be reviewed to ensure that data have met project specifications for precision and accuracy. The degree to which project specifications have been met will provide a qualitative assessment of the representativeness.

1.6.2.5 Comparability

Comparability is defined as the confidence with which one data set can be compared to another (e.g., between sampling points and between sampling events). Comparability is achieved by using standardized sampling and analysis methods and data reporting formats (including use of consistent units of measure), and by ensuring that quantitation limits and method detection limits are sufficiently low to satisfy project action limits for the duration of the project. The quantitation limits and method detection limits anticipated for this project are presented in UFP QAPP Worksheet 15. Additionally, consideration was given to seasonal conditions and other environmental variations that could exist to influence analytical results, but no such influences appear to exist for this investigation that would indicate a need to collect samples at times other than those planned for this investigation.

1.6.2.6 Completeness

Completeness will be computed in accordance with the following equation:

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

Completeness is a measure of the amount of valid data obtained from the measurement program, compared to the total amount collected. Valid data are defined as data that have not been rejected or considered unusable during validation or data review. For relatively clean, homogeneous matrices, 100% completeness is expected. However, as matrix complexity and heterogeneity increase, completeness may decrease. In addition, simple accidents, such as loss of samples during shipment can result in less than 100 percent completeness. Completeness will be reported for each matrix (number of samples collected versus planed) and analytical group (percent of results that are valid). Completeness will not be reported for various concentration levels because all concentration levels are equally important for attaining project objectives.

Where PQOs are compromised by unavailable data, the ability to achieve project objectives will be evaluated. Whether any particular sample result is critical (i.e., absolutely necessary for the attainment of project objectives) to the investigation will be evaluated in terms of the sample location, the parameter in question, the intended data use, and the effects of an incomplete data set on the attainment of project objectives.

Critical data points may not be identified until all the analytical results are evaluated. If in the evaluation of results it becomes apparent that the data for a specific medium are of insufficient quality (i.e., less than 95% completeness), either with respect to the number of samples or an individual analysis, resampling to replace the deficient data points may be necessary. The Navy and TtNUS will determine whether resampling is necessary.

1.7 SECONDARY DATA EVALUATION

The subject work is being conducted as an SI, the first phase of investigation. If present, secondary data would be listed in UFP QAPP Worksheet 13.

1.8 PROJECT OVERVIEW AND SCHEDULE

The section summarizes project activities and provides the schedule for each task.

1.8.1 Project Overview (Outcome of Project Scoping Activities)

UFP QAPP Worksheet 14 presents a synopsis of the tasks that will be undertaken to complete this project. More detailed information is provided in the Work Plan Sections 3 and 5.1, as well as Section 4 of the SAP.

1.8.2 Project Schedule

The project schedule is displayed in UFP QAPP Worksheet 16.

2.0 MEASUREMENT AND DATA ACQUISITION ELEMENTS

2.1 SAMPLING TASKS

2.1.1 Sampling Process Design and Rationale

The overall sampling design and rationale for collecting various samples is presented in Section 4 of the SAP and location maps can be found in Section 2 of the Work Plan. UFP QAPP Worksheet 17, Sampling Design and Rationale, and UFP QAPP Worksheet 18, Sampling Locations, direct users to the Work Plan and SAP noted above.

Analytical requirements are summarized in UFP QAPP Worksheet 19, which is described in more detail in Section 2.1.2.2.

Collection of quality control samples will include blind duplicates and matrix spikes, temperature blanks, trip blanks, field blanks, and equipment blank samples. A summary of the type and frequency of quality control sampling is included in UFP QAPP Worksheet 20.

2.1.2 Sampling Procedures and Requirements

2.1.2.1 Sampling Collection Procedures

A summary of sample identification, depths, SOPs, and summary of rationale for sampling tasks is provided in the SAP, Sections 4 and 5. UFP QAPP Worksheet 21 lists all project sampling SOPs. These field SOPs are presented in their entirety in Attachment 1-B. Example field forms are included as Attachment 1-C.

2.1.2.2 Sample Containers, Volume, and Preservation

Sample-specific requirements for sample containers, volume, and preservation requirements are provided on UFP QAPP Worksheet 19.

2.1.2.3 Equipment/Sample Containers Cleaning and Decontamination Procedures

All equipment that makes contact with samples will either be obtained in a suitable condition of cleanliness or will be cleaned prior to making contact with samples. Reusable equipment will be cleaned between uses to ensure that samples are not contaminated by the sampling equipment.

Sample containers will be shipped certified clean from the analytical laboratory. Sampling equipment decontamination procedures are presented in SOP SA-7.1 (S8) in Attachment 1-B.

2.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures

Each field crew member must inspect equipment items prior to use to ensure they are capable of meeting their intended use. Some of these inspections are carried out during calibration or routine maintenance described in this section. More casual inspections for equipment integrity must be made routinely to ensure that the equipment is intact, all applicable parts are operating correctly, and the equipment is suitably clean to perform its task. All electronic measurement equipment must be calibrated or must be received with a valid calibration and this must be verified by the field crew member prior to use of the equipment to be suitable for use.

The overall requirements for field equipment usage, maintenance, testing, and inspection for this project are provided in UFP QAPP Worksheet 22. Calibration and maintenance SOPs are provided in Attachment 1-B for measurement instruments that are commonly used. Calibration and standards checks will be conducted on the water quality meter in accordance with the manufacturer's recommendations and SOP S11 (USEPA Region I methods). The calibration procedure for all parameters except ORP will use, at a minimum, two standards to bracket the measurement range. The ORP measurement uses one standard (Zobell standard). For less commonly used equipment or for maintenance not covered by the SOPs, the owner's manual for the equipment will be consulted. If equipment that is out of calibration cannot be brought into calibration, the equipment will not be used. If the equipment cannot be used then new equipment must be acquired before proceeding.

2.1.2.5 Sampling Supply Inspection and Acceptance Procedures

Each person using sampling equipment must ensure that the equipment is capable of adequately serving its intended use. This includes a verification of the equipment cleanliness as described in Section 2.1.2.3, and a verification that the equipment is in good working order and that all accessories necessary for performing the work have been provided by the vendor. If questions arise, the Field Operations Leader (FOL) will be contacted for guidance.

2.1.2.6 Field Documentation Procedures

Field documentation will be preformed in accordance with SOP SA-6.3 (S9) presented in Attachment 1-B.

2.2 ANALYTICAL TASKS

This section provides information with regard to the analytical SOPs, calibration procedures, instrument/equipment maintenance, testing, and inspection procedures for all selected laboratories.

2.2.1 Analytical SOPs

All relevant analytical SOPs are summarized in UFP QAPP Worksheet 23. These laboratory SOPs are provided in Attachment 1-D (on compact disc).

2.2.2 Analytical Instrument Calibration Procedures

All instrument calibration, frequency of calibration, acceptance criteria, corrective action, and person responsible for corrective action are displayed in UFP QAPP Worksheet 24. The SOPs are designed to ensure that laboratory equipment meets the sensitivity, accuracy, and precision requirements of this QAPP.

2.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures

All instrument and equipment maintenance, testing, and inspection procedures are displayed in UFP QAPP Worksheet 25. These procedures are designed to ensure that all necessary equipment is continually available to prevent project delays caused by missing or broken equipment.

2.2.4 Analytical Supply Inspection and Acceptance Procedures

The laboratory must be able to demonstrate that all supplies needed for analytical work will be available when needed and be free of target compounds and any interference. This information can be supplied upon request.

2.3 SAMPLE COLLECTION DOCUMENTATION, HANDLING, TRACKING, AND CUSTODY PROCEDURES

The following sections outline the procedures that will be used to document project activities and sample collection, handling, tracking, and custody procedures during the SI. All forms must be filled in as completely as possible.

2.3.1 Sample Collection Documentation

Documentation of field observations will be recorded in a field logbook and/or field log sheets including sample collection logs, boring logs, and monitoring well construction logs. Field logbooks utilized on this project will consist of a bound, water-resistant logbook. All pages of the logbook will be numbered sequentially and observations will be recorded with indelible ink.

Field sample log sheets will be used to document sample collection details, and other observations and activities will be recorded in the field logbook. Instrument calibration logs will be used to record the daily instrument calibration. Example field forms are included in Attachment 1-C.

For sampling and field activities, the following types of information will be recorded in the field log as appropriate:

- Site name and location
- Date and time of logbook entries
- Personnel and their affiliations
- Weather conditions
- Activities involved with the sampling
- Subcontractor activity summary
- Site observations including site entry and exit times
- Site sketches made onsite
- Visitor names, affiliations, arrival and departure times
- Health and safety issues including personal protective equipment (PPE)

2.3.2 Sample Handling and Tracking System

These subsections outline the procedures that will be used by field and laboratory personnel to document project activities and sample collection procedures during this SI. All forms must be filled in as completely as possible.

2.3.2.1 Sample Handling

Sample handling is described in UFP QAPP Worksheet 26.

2.3.2.2 Sample Delivery

Samples to be delivered to the laboratory will be made by a public courier (i.e., Federal Express). After samples have been collected, they will be sent to the laboratory within 72 hours. Under no circumstances will sample holding times be exceeded.

2.3.3 Sample Custody

To ensure the integrity of a sample from collection through analysis, it is necessary to have an accurate, written record that traces the possession and handling of the sample. This documentation is referred to as the chain-of-custody form. The chain of custody begins at the time of sample collection.

A sample is under custody if:

- The sample is in the physical possession of an authorized person.
- The sample is in view of an authorized person after being in his/her possession.
- The sample is placed in a secure area by an authorized person after being in his/her possession.
- The sample is in a secure area, restricted to authorized personnel only.

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

Integrity of the samples collected during the site investigation will be the responsibility of identified persons from the time the samples are collected until the samples, or their derived data, are incorporated into the final report. Sample custody is described in UFP QAPP 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 chain-of-custody form. This record documents the sample custody transfer from the sampler to the laboratory, often through another person or agency (common carrier). Upon arrival at the laboratory, internal sample custody procedures will be followed as defined in the laboratory SOPs included in Attachment 1-D.

2.4 QUALITY CONTROL SAMPLES

2.4.1 Sampling Quality Control Samples

QC samples will be collected or generated during environmental sampling activities. QC samples will include field duplicates for soil and sediment. A summary of Quality Assurance/Quality Control (QA/QC) samples requirements are provided in Worksheet 20. The four types of field QC samples are defined as follows:

Field Duplicates. Field duplicates are obtained during a single act of sampling and are used to assess the overall precision of the sampling and analysis program. Field duplicates will be collected at a frequency of 10% per sampling matrix. Duplicates are submitted for laboratory analyses for the same parameters as their environmental sample counterparts. For all media these duplicates will be subsample field duplicates unless the sample collection does not permit the collection of enough sample volume to support splitting. For example, it may be impossible to collect enough groundwater sample in a single pass to fill all required sample bottles.

Equipment Rinsate Blanks. Equipment rinsate blanks are obtained under representative field conditions by running analyte-free water through or over decontaminated sample collection equipment [bailer, direct-push technology (DPT) cutting shoe, etc.]. Equipment rinsate blanks will be used to assess the effectiveness of decontamination procedures. Equipment rinsate blanks will be collected for each type of non-dedicated sampling equipment used and will be submitted at a frequency of one per day or one per 20 units of a medium sampled, whichever is less. One equipment rinsate blank will also be collected from each dedicated sampling device for this project. Equipment rinsate blanks will be analyzed for the same suite of analytical parameters as the associated environmental samples and will be collected in the appropriate sample containers.

Source Water Blank (Field Blanks) - Source water blanks are collected to assess the presence of contamination in the water used to decontaminate the sampling equipment. Field personnel prepare source water blanks. One source water blank is to be collected for each type of decontamination water (deionized water, tap water, etc.) used during each sampling phase.

Temperature Blank - Temperature blanks are vials of water inserted into each sample cooler prior to shipment from the field. The temperature of the temperature blank is measured upon receipt at the laboratory to assess whether samples were properly cooled during transit.

2.4.2 Analytical Quality Control Samples

Analytical quality control samples to be used during this SI are provided in UFP QAPP Worksheet 28.

2.5 DATA MANAGEMENT TASKS

This section describes how all project information will be managed, organized, and maintained for efficient use by project personnel. The information management process is outlined from the point of data generation to ultimate storage.

2.5.1 Project Documentation and Records

A summary of project documentation and records to be generated and stored in TtNUS project files is provided in UFP QAPP Worksheet 29.

2.5.2 Data Package Deliverables

2.5.2.1 Sample Collection and Field Measurements Data Package Deliverables

Sample collection data are as required by the sample collection SOPs of Attachment 1-B. In addition, calibration data will also be provided in accordance with the SOPs of Attachment 1-B.

Results will be provided to MEDEP electronically in the Department's Environmental Data Deliverable (EDD) format for the Environmental and Geographic Analysis Database (EGAD). This will include field parameter data, water level and flow data, and laboratory analytical data for soil and groundwater. Laboratory analytical data will include field quality control sample results, surrogate recoveries in percent, and matrix spike/matrix duplicate recoveries in percent. Sample locations and other geographic location data will be provided to sub-meter accuracy in a map file or table for uploading to the Department's GIS (Arc GIS). Universal Transverse Mercator (UTM) coordinated based on North American Datum (NAD) 1983 preferred for coordinates, at a minimum, the projection, units, and datum will be submitted.

2.5.2.2 On-Site Analysis Data Package Deliverables

This project involves on-site analyses beyond the field screening associated with well stabilization and personnel protection. The on-site analyses are associated with XRF sampling of lead at the Machine Gun Boresight Range (by USEPA) and the NASB Skeet Range and Topsham Skeet Range (by TtNUS). Details are described in the Sections 4.6.4 and 4.11.2 of the SAP.

A data package deliverable is not required for XRF field screening; however, the field screening data will be recorded in the format determined by the XRF instrument manufacturer's software, and the resultant output will be managed in a manner consistent with other field data for the project. It is anticipated that resultant output from the XRF will be in a tabular format text file capable of being imported and evaluated in Microsoft Excel. The minimum requirements are a sample identifier, numerical result, and measurement units for every sample identified. If each final numerical result is an average of intermediate results, the intermediate results will be documented in a similar manner. Measures of precision (e.g., standard deviation) based on multiple intermediate results may also be recorded. Also required are the name of the person performing the analysis, the date and time of analysis, and a reference to the calibration (standardization) on which the measured values are based. Field calibration information will also be included with the XRF output. Although the data downloaded from the XRF instrument includes the results for all metals that the XRF analyzes, only lead data for this project will have calibration checks. Therefore, only data for lead will be incorporated in the database.

Results will be provided to MEDEP electronically in the Department's Environmental Data Deliverable (EDD) format for the Environmental and Geographic Analysis Database (EGAD).

2.5.2.2 Off-Site Laboratory Data Package Deliverables

Data package deliverable requirements are detailed in Table 2-1, entitled Analytical Data Deliverable Elements, and UFP QAPP Worksheet 30 Analytical Services.

Data packages will require all the elements specified in Table 2-1. Data packages will be provided as both hardcopy and portable document format (.PDF). All laboratories will provide a Naval Installation Restoration Information Solutions (NIRIS) compatible electronic data deliverable (EDD). Results will be provided to MEDEP electronically in the Department's Environmental Data Deliverable (EDD) format for the Environmental and Geographic Analysis Database (EGAD). Reports will include surrogate recoveries in percent and matrix spike/matrix spike duplicate recoveries in percent. Data packages will be contract laboratory program (CLP)-equivalent (i.e., they will contain CLP-equivalent summary forms and raw data). The standard turnaround time for analytical services is 21 calendar days. Turnaround time will be measured from the laboratory receipt of the last samples in a sample delivery group (SDG). SDGs must contain 20 samples (no more than 20 and only less if the entire sampling event was comprised of less than 20 samples). Data will be stored by the analytical laboratory for 5 years.

2.5.3 Data Reporting Formats

Field data will be recorded in the field logbooks and field forms. All logbook and log sheet entries must be made in indelible ink (black pen is preferred). No erasures or liquid paper or white out are permitted. If an incorrect entry is made, the data will be crossed out with a single strike mark, initialed, and dated. The field personnel will sign and date the logbook pages and field forms. Examples of the forms to be used in the field are presented in Attachment 1-C of this QAPP.

2.5.4 Data Handling and Management

The data-handling procedures to be followed by the laboratories will meet the requirements in the laboratory subcontracts. All analytical and field data will be maintained in the project files. The project files will contain hard copies of the chain-of-custody forms, sample log forms, and sample location maps and documentation of quality assurance of data manipulation. Forms are included in Attachment 1-C of this QAPP.

The overall field data flow is as follows:

1. Field personnel calibrate sampling equipment and document calibrations in accordance with SOPs of Attachment 1-B.

2. Field personnel use the SAP, documentation from the site visit, and magnetometer/Global Positioning System (GPS) survey results to identify sampling locations.

3. As applicable, sampling locations are evaluated to ensure they are safe for personnel to collect samples and the evaluations are documented in accordance with SOPs of Attachment 1-B and the HASP (approved by the Navy as a separate document).

4. If applicable, sampling conditions are monitored with field instruments to ensure that sampling conditions are representative of the intended populations. These measurements are made and documented in accordance with SOPs of Attachment 1-B.

5. Samples are collected and documented and sampling equipment is decontaminated in accordance with SOPs of Attachment 1-B.

6. Samples are shipped to the laboratory for analysis and shipping documentation, including chain of custody records are compiled for later transfer to the Project Manager.

7. Sampling locations are marked for land survey and are later land surveyed or “GPS’d” to establish sample locations within the desired precision and accuracy.

8. Field conditions are recorded throughout the field work.

9. During demobilization field records are collected, double checked for completeness, and forwarded to the TtNUS project manager for inclusion in the project database and the appropriate site report.

The overall laboratory data flow is as follows:

1. Samples are received, inspected and the condition of the samples is logged.

2. Analytical equipment is calibrated for analysis and calibrations are documented.

3. Samples are analyzed and the analyses are documented.

4. The resulting data are reviewed and incorporated into the laboratory information management system.

5. Hardcopy data packages are assembled and electronic data deliverables are prepared to match the hardcopy data packages.

6. The hardcopy and electronic data are transmitted to TtNUS.

The overall TtNUS data flow is as follows:

1. EDDs and hardcopy data packages are received from the laboratory. These data are reviewed for accuracy and are validated.

2. A validation report is prepared to document the data quality.

3. Data qualifiers are assigned to electronic data and the data are transferred, with qualifiers, to the TtNUS project database.

4. Corresponding field data are transferred to the project database.

5. Data are now available for use by TtNUS project personnel. A data usability assessment is performed to ensure the data will be useful as intended. Data usability is assessed continually as various data users have the opportunity to work with the data.

6. Validated data are made available to the Geographical Information System (GIS) for plotting and other manipulations.

Additional details are provided in the subsections below.

2.5.5 Data Tracking and Control

A "cradle-to-grave" sample tracking system will be used from the beginning to the end of the investigation. Before field mobilization, the FOL will coordinate/initiate the sample tracking process. The Project Manager will ensure that sample jar labels are printed before field sampling, as necessary. The FOL and Project Manager or designee will review the labels for completeness of information and adherence to work plan requirements, as well as for accuracy. The Project Manager will coordinate with the analytical laboratory to ensure that they are aware of the number and type of samples and analyses that are about to be requested.

When field sampling is underway, the FOL will forward the chain-of-custody forms to the TtNUS Project Manager or designee via facsimile at the end of each day. The Project Manager or designee will compare the entries on the chain-of-custody forms with the sample tracking database and enter the sample date and other sample information as appropriate. The Project Manager or designee will also confirm that the chain-of-custody forms provide the information required by the Work Plan. This will allow for early detection of errors made in the field so that adjustments can be made while the crew is mobilized. After successful completion of all requested analyses, the laboratory will submit an electronic deliverable for every SDG. When all electronic deliverables have been received from the laboratory, the Project Manager will ensure that the laboratory performed all the requested analyses. Ideally, discrepancies can be noted early enough so that all samples can be analyzed within the prescribed holding times.

2.5.5.1 Sample Information

Data from field measurements will be recorded directly in field notebooks or on sample logs. Reduction of field data entails the summarization and presentation of these data in tabular form. The reduction of laboratory data entails the manipulation of raw data instrument output into reportable results. Field data

will be verified on a daily basis by the FOL. Laboratory data will be verified by the group supervisor and then by the laboratory's QC/Documentation Department.

Before electronic files are received from the laboratory, all sample-specific information will be entered into the data management system. The sample information file will allow the analytical results to be grouped together properly for statistical purposes. The data will be managed in one data structure. For field data, the FOL will coordinate with the GIS lead to ensure that all survey technical specifications are consistent with the underlying coordinate system in the GIS.

Electronic data arriving from the laboratory will pass through to the data validation manager (DVM) for database compilation and validation. The DVM will compile all the formatted laboratory electronic deliverables into a working project database. Data that are to be validated will be printed as data packages, which include the samples as part of each SDG and the appropriate analytical fraction. The data packages will be distributed to the appropriate data validators. The data validators will enter all data qualifiers and qualifier codes into the database and print out a hard copy and return it to the DVM. The DVM will check the data qualifiers and qualifier codes in the project database and print the final validated data for incorporation into the data validation letter. When all samples and analyses have been accounted for and validated, the Project Manager will ensure that the analytical data are incorporated into the project database.

2.5.5.2 Project Data Compilation

The analytical laboratory subcontractor(s) will generate a PDF file of the analytical data packages, as well as electronic database deliverables. The electronic database will be checked against the PDF file provided by the laboratory and updated as required, based on data qualifier flags applied during the data validation process. The data generated during the implementation of this QAPP will be incorporated into the project database and GIS. All data, such as units of measure and chemical nomenclature, will be manipulated to maintain consistency with the project database.

2.5.5.3 Geographical Information System

Data management systems consist of a relational database and GIS that are being used to manage environmental information pertaining to this project. The relational database stores chemical, geological, hydrogeological, and other environmental data collected during environmental investigations. The GIS is built from the relational database and contains subsets of the larger data pool. Using the GIS, environmental data can be posted on base mapping to provide a graphical representation of the information.

TABLE 2-1

LABORATORY DATA PACKAGE ELEMENTS
SITE INVESTIGATION QAPP
NAS BRUNSWICK, MAINE
PAGE 1 OF 4

DATA PACKAGE ELEMENTS	Nitrates	PAHs	Explosives (dinitrotoluene) and Propellants (nitroglycerin)	Metals and TCLP Metals	Perchlorate	TCLP Organic	TOC, pH, CEC
♦ **NARRATIVE (Org. Narrative, Inorg. Cover Page)**	X	X	X	X	X	X	X
♦ **SHIPPING/RECEIVING DOCUMENTS AND INTERNAL LABORATORY COC RECORDS:**							
- Airbills	X	X	X	X	X	X	X
- Chain-of-Custody Records/Forms (Traffic Report)	X	X	X	X	X	X	X
- Sample Log-In Sheet (Org. and Inorg. DC-1 Form)	X	X	X	X	X	X	X
- Miscellaneous Shipping/Receiving Records	X	X	X	X	X	X	X
- Internal Lab. Sample Transfer Records and Tracking Sheets	X	X	X	X	X	X	X
♦ **SAMPLE DATA:**							
- Tabulated Summary Form for Field Sample, Method Blanks, and PE Sample Results (Org. and Inorg. Form I)	X	X	X	X	X	X	X
- Tentatively Identified Compounds Tabulate Summary Form (Org. Form I TIC)							
- Reconstructed Total Ion Chromatogram (RIC) for each sample	X	X				X	
- Raw spectra of target compound and background subtracted spectrum of target compound for each sample	X	X			X	X	
- Mass spectra of all reported TICs/three best library matches for each sample							
- Chromatograms from both columns for each sample							
- Pesticide/PCB Identification Tabulated Summary Form (Org. Form X)							
- For Pest/PCB or Dioxin/Furan results confirmed by GC/MS, copies of raw spectra and background subtracted spectrum of target compounds							
- GPC sample chromatograms (if necessary)		X				X	
- Sample preparation/extraction/digestion log (Inorg. Form XIII) and logbook pages	X	X	X	X	X	X	X
- Sample analysis run log (Inorg. Form XIV) and logbook pages	X	X	X	X	X	X	X
- ICP Raw Data				X			
- Furnace AA Raw Data							

TABLE 2-1

LABORATORY DATA PACKAGE ELEMENTS
SITE INVESTIGATION QAPP
NAS BRUNSWICK, MAINE

DATA PACKAGE ELEMENTS	Nitrates	PAHs	Explosives (dinitrotoluene) and Propellants (nitroglycerin)	Metals and TCLP Metals	Perchlorate	TCLP Organic	TOC, pH, CEC
- Mercury Raw Data				X			
- Cyanide Raw Data							
- Other Analytical Raw Data	X	X		X		X	X
♦ **STANDARDS DATA:**							
- Method Detection Limit Study Tabulated Summary Form	X	X	X	X		X	X
- Initial Calibration Tabulated Summary Form (Org. Form VI, Inorg. Form IIA)	X	X	X	X	X	X	X
- Continuing Calibration Tabulated Summary Form (Org. Form VII, Inorg. Form IIA)	X	X	X	X	X	X	
- RICs and quantitation reports for all GC/MS Standards	X	X				X	
- Pesticides Analyte Resolution tabulated Summary Form (Org. Form VI, Pest-4)							
- Pesticides Calibration Verification Tabulated Summary Form (Org. Form VII, Pest-1 and Pest-2)							
- Analytical Sequence Tabulated Summary Form (e.g. Org. Form VIII-Pest)			X		X		
- GC Chromatograms and data system printouts for all GC standards							
- For Pesticides/Aroclors confirmed by GC/MS, copies of spectra for standards data							
- GPC Calibration Tabulated Summary Form (Org. Form IX, Pest-2)							
- Florisil Cartridge Check Tabulated Summary Form (Org. Form IX, Pest-1)							
- Instrument Detection Limits Tabulated Summary Form (Inorg. Form X)				X			
- ICP Interelement Correction Factors Tabulated Summary Form (Inorg. Form XIA and XIB)				X			
- ICP Linear Ranges Tabulated Summary Form (Inorg. Form XII)				X			
- CRDL Standards for AA and ICP Tabulated Summary Form (Inorg. Form IIB)				X			
- Standards preparation logbook pages	X	X	X	X	X	X	X

TABLE 2-1

LABORATORY DATA PACKAGE ELEMENTS
SITE INVESTIGATION QAPP
NAS BRUNSWICK, MAINE

DATA PACKAGE ELEMENTS	Nitrates	PAHs	Explosives (dinitrotoluene) and Propellants (nitroglycerin)	Metals and TCLP Metals	Perchlorate	TCLP Organic	TOC, pH, CEC
♦ **QC DATA:**							
- Tuning and Mass Calibration Tabulated Summary Form (Org. Form V)	X	X			X	X	
- Window defining mixture							
- Chromatographic resolution							
- Surrogate Percent Recovery Tabulated Summary Form (Org. Form II)	X	X	X			X	
- MS/MSD Recovery Tabulated Summary Form (Org. Form III)	X	X	X			X	
- Method Blank Tabulated Summary Form (Org. Form IV and Inorg. Form III)	X	X	X	X		X	X
- Internal Standard Area and RT Tabulated Summary Form (Org. Form VIII)	X	X			X	X	
- Labeled Compound Recovery Summary Form							
- QC Raw Data – RICs, Chromatograms, Quan Reports, Integration Reports, Mass Spectra, etc.	X	X			X	X	X
- Spike Sample Recovery Tabulated Summary Form (Inorg. Form IV)				X			X
- Duplicates Tabulated Summary Form (Inorg. Form VI)				X			X
- Internal Laboratory Control Sample Tabulated Summary Form (Org Form III and Inorg. Form VII)		X	X	X	X	X	X
- Standard Addition Results Tabulated Summary Form (Inorg. Form VIII)				X			
- ICP Interference Check Sample Tabulated Summary Form (Inorg. Form IV)				X			
- Post Digestion Spike Tabulated Summary Form (Inorg. Form VB)				X			
- ICP Serial Dilutions Tabulated Summary Form (Inorg. Form IV)				X			
- QC Raw Data – ICP, Furnace, Mercury computer printouts, etc.				X			X
- QC sample preparation logbook pages	X	X	X	X	X	X	X
- Toxicity Equivalence Factor							
- Compound Identification Criteria							

TABLE 2-1

LABORATORY DATA PACKAGE ELEMENTS
SITE INVESTIGATION QAPP
NAS BRUNSWICK, MAINE

DATA PACKAGE ELEMENTS	Nitrates	PAHs	Explosives (dinitrotoluene) and Propellants (nitroglycerin)	Metals and TCLP Metals	Perchlorate	TCLP Organic	TOC, pH, CEC
♦ **MISCELLANEOUS DATA:**							
- Original preparation and analysis forms or copies of preparation and analysis logbook pages	X	X	X	X	X	X	X
- Screening records	X	X		X		X	X
- All instrument output, including strip charts from screening activities	X	X	X	X	X	X	X
- Instrument stability							
- Preparation Logs Raw Data	X	X	X	X	X	X	X
- Percent Solids Determination Log	X	X	X	X		X	X
- Other Records (ex. Telephone Communication Log)	X	X		X		X	X

(1) Miscellaneous data package will include information as applicable to the method.
VOC - Volatile Organic Compounds
PAH – Polycyclic Aromatic Hydrocarbons
TOC –Total Organic Carbon
TCLP – Toxicity Characteristics Leaching Procedure
CEC – Cation Exchange Capacity

3.0 ASSESSMENT AND OVERSIGHT

3.1 ASSESSMENT AND RESPONSE ACTIONS

Assessment activities ensure that planned project activities are implemented as described in this QAPP, and that appropriate corrective actions are implemented to address nonconformances and deviations from the QAPP. These activities help to ensure that data are adequate for their intended use.

3.1.1 Planned Assessments

The planned assessments are system audits and field audits. The assessments planned for this project are identified in UFP QAPP Worksheet 31.

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

- The FOL will supervise and check on a daily basis that the field measurements are made accurately, equipment is thoroughly decontaminated, samples are collected and handled properly, and fieldwork is accurately and neatly documented. Documentation includes verifying that the sample names on sample log sheets, field notes, chain-of-custody records, and sample labels are identical matches to sample names in the QAPP. The FOL will update the Project Manager of field activities on a daily basis.

- System audits for the laboratory will be performed regularly and in accordance with NFESC guidance and DOD QSM (January, 2006), as provided in the Laboratory Quality Assurance Plan (LQAP).

- The data validator will review the chemical analytical data packages submitted by the laboratory. The data validator will check that the data were obtained through use of the approved methodology, that the appropriate level of QC effort and reporting was conducted, and whether or not the results are in conformance with QC criteria. On the basis of these factors, the data validator will generate a report describing data limitations that will be reviewed internally by the DVM before submittal to the Project Manager.

- The Project Manager will maintain contact with the FOL and DVM to ensure that management of the acquired data proceeds in an organized and expeditious manner.

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

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

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

3.1.2 Assessment Findings and Corrective Action Responses

Assessment findings that require corrective action initiate a sequence of events that include documentation of deficiencies, notification of findings, request for corrective action, implementation of corrective action, and follow-up assessment of the corrective action effectiveness. UFP QAPP Worksheet 32 summarizes the procedure for handling any QAPP deviations and project deficiencies that are identified through the planned project assessments.

Potential problems may involve nonconformance with the SOPs and/or analytical procedures established for the project or other unforeseen difficulties. Any person identifying a condition adverse to project quality will notify the PM. The PM, with the assistance of the Quality Assurance Manual (QAM), will be responsible for developing and initiating appropriate corrective action. If the identified deficiencies involve field work, this will be done through the FOL; if the deficiencies involve the laboratory, this will be done through the laboratory project manager. The corrective actions will require follow through to the point of verifying that the corrective action has been effective. Corrective actions may include resampling and/or reanalyzing

samples or amending or adjusting project procedures. If warranted by the severity of the problem (for example, if a change in the approved plan is required), the Navy will be notified in writing and the Navy's approval will be obtained before any change is implemented. Minor changes will be documented for the main file by the TtNUS PM. Additional work that depends on a nonconforming activity will not be performed until the problem has been eliminated. The overall corrective action responsibility for system audits will reside with the PM. The overall corrective action responsibility for field audits will reside with the QA officer.

For QA issues involving the analytical laboratory to be used for the project, the laboratory also maintains an internal closed-loop corrective action system that operates under the direction of the laboratory QA coordinator.

3.2 QA MANAGEMENT REPORTS

This section presents the activities that will be performed to keep management updated on the project status. Open communication pathways will benefit the project by allowing all appropriate personnel to be aware of activities and have the ability to provide input in a timely manner. Input from these parties will be used to make necessary corrective actions so project quality objectives are met.

The information to be included in each of the QA Management Reports listed UFP QAPP Worksheet 33 is summarized as follows.

Field Status Reports

The FOL will give verbal status reports to the Project Manager on a daily basis or more frequently if needed. The status reports will include the field activities completed for the day, the personnel who completed each activity, the anticipated activities to be completed during the next day, and any issues or problems identified. A summary of most significant progress in project activities will be sent via electronic mail, facsimile transmission, or other agreed upon mode of documentation to the Project Manager.

Data Validation Reports

Data validation reports will be prepared and formatted as described in Section 4.2. The data validation reports will be included as an appendix to the SI. Field data will not be validated.

When requested by the Project Manager, additional reports may be generated. For example, detailed data usability assessments may be documented in usability reports. However, other data assessments are planned to be included directly in the SI. Therefore, the appropriate sections of the report will identify the following:

- Summary of project QA/QC programs and training conducted during the project.
- Conformance of project activities to QAPP requirements and procedures.
- Status of project and schedule delays.
- Deviations from approved QAPP or QAPP amendments and the impact of such deviations on the attainment of project objectives.
- Description and findings of assessments.
- Required corrective actions and effectiveness of corrective actions.
- Limitations of the data usability.

Major Analysis Problem Identification

If a major analysis problem occurs the laboratory project manager will immediately inform the project chemist and the project chemist will draft and internal memorandum (most likely via email) and inform the Project Manager, DVM, QAM, and project file. If corrective actions are warranted they will be drafted and implemented as soon as possible.

Laboratory Quality Assurance Reports

The laboratory will draft a memorandum documenting deviations from QAPP. This would occur in only situations of instrument failure or some catastrophic nature.

3.3 FINAL PROJECT REPORT

UFP QAPP Worksheet 29 presents a list of reports to be generated for this project.

4.0 DATA REVIEW ELEMENTS

4.1 OVERVIEW

Data review is the process by which data are examined and evaluated by various personnel at various levels of detail. This process includes data verification, data validation and data usability assessment. Data verification is a process of evaluating the completeness, correctness, and contractual compliance of a data set against the method standard, SOP, or contract requirements documented in this QAPP. Data validation is an analyte- and sample-specific process that extends the qualification of data beyond data verification to determine the quality of a specific data set relative to analytical quality control requirements. Data usability assessment extends these other review processes to examine the data in the context of the project objectives to determine whether the data are suitable for supporting the attainment of these objectives.

The internal data verification requirements for this project include the maintenance and periodic review of field documentation (site logbooks, instrument calibration logs, chain-of-custody forms, field summary reports, and field modification records) and laboratory analytical data packages.

Data validation is a systematic review of the analytical data package with respect to sample receipt and handling, compliance with required analytical methods, data reporting and deliverables, and document control. A qualified chemist will review the analytical data packages using USEPA procedures.

Usability assessment will be performed by project level personnel who understand the intended use of the data. These personnel include but are not necessarily limited to project chemists, geologists, and risk assessors.

4.2 DATA REVIEW STEPS

Upon receipt of analytical laboratory results, TtNUS database personnel will evaluate electronic and hardcopy deliverables for conformance to data format specifications and for completeness. TtNUS validation chemists also perform completeness verification and will then perform data validation according to the most current data validation guidelines to ensure that the analytical results meet the data quality objectives for risk assessment. The validation guidance documents that will be used include:

- Region I Inorganic Data Validation Functional Guidelines (2/89)

- Region I Organic Data Validation Functional Guidelines [Volatile/Semivolatile part 2, pesticide/Polychlorinated Biphenyls (PCB) part 3] (12/96), to the extent practicable.

- DOD QSM (January 2006).

- Test Methods for Evaluating Solid Waste; Physical/Chemical Methods (SW-846), 3rd edition, up to and including Update III. Office of Solid Waste and Emergency Response, Washington, DC. 1986.

- DOD Perchlorate Handbook (May 2007).

The TtNUS DVM stays current with available guidelines to ensure that the appropriate validation guidelines are used.

After the data are validated, a list of nonconformities will be generated. Nonconformities require data qualifiers, which are used to alert the data user to inaccurate or imprecise data. For situations in which several quality control criteria are out of specification with regard to the limits specified in the DOD QSM (January, 2006), the data validator may make professional judgments and/or comments on the validity of the overall data package. In situations where the validity of an entire data package is in question, it may be necessary for the sample(s) to be reanalyzed. The reviewer will then prepare a technical memorandum presenting changes in the data, if necessary, and the rationale for making such changes.

The net result is a data package that has been carefully reviewed for its adherence to prescribed requirements. Data validation therefore plays a major role in determining the confidence with which key technical evaluations may be made.

Data validation reports for all parameters will be generated according to the requirements described above. The final data validation report will include a technical memorandum, qualified analytical results, results reported by the laboratory, and documentation to support data qualification. All data will be flagged by an appropriate qualifying symbol.

The data and field records will also be reviewed by project personnel to ensure that the samples represent the intended sampling conditions and populations. Data qualified during validation will be reviewed to assess the impact of the qualifiers on the attainment of project objectives.

4.2.1 Step I: Verification

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

4.2.2 Step II: Validation

Validation of field measurements and laboratory analytical data is discussed in this section. Validation of field data will be limited to real-time checks in the field as data are generated, whereas laboratory analytical data will be validated in accordance with current applicable USEPA guidance. Step IIa, Validation Activities (i.e., compliance with methods, procedures and contracts), are discussed in Section 4.2.2.1. Step IIb, Validation Activities [i.e., comparison of results to measurement performance criteria (MPCs) in this QAPP] are in discussed in Section 4.2.2.2.

4.2.2.1 Step IIa: Validation Activities

One hundred percent of the laboratory data from chemical analyses will be validated. Region I, Tier 3 data validation will be employed. Validation of analytical data will be conducted by the TtNUS or data validation subcontractor. Final review and approval of validation deliverables will be completed by the data validation manager. All parameters will be reviewed using applicable sections of the aforementioned guidelines and the laboratory SOPs. Each analysis will be validated in accordance with UFP QAPP Worksheet 36.

As part of the validation process, the validator will check that the laboratory has provided all the documentation required to support the reported analytical results. If any documentation is missing from the data package, the data validator will contact the laboratory to request a resubmittal. If the laboratory fails to resubmit the requested information, the data validator will note this on the data validation cover letter. The usability of such data will then be determined by TtNUS, the Navy, and regulatory agencies.

Data validation will be completed to ensure that the data are of evidentiary quality. Particular emphasis will be placed on holding time compliance, equipment calibration, spike recoveries, and blank results, although all required elements of the validation process will be considered.

4.2.2.2 Step IIb: Validation Activities

UFP QAPP Worksheet 35 summarizes the Step IIb data validation activities.

4.2.3 Step III: Usability Assessment

Section 1.7.2 contains a PARCCS description and Worksheets 12, 15, and 28 provide project specific MPCs.

After completion of data validation and an overall review of data quality indicators, the data will be reconciled with MPCs to determine whether sufficient data of acceptable quality are available for decision making. A series of inspections and analyses will be performed to estimate several of the data set characteristics. The evaluations will include simple summary statistics for target analytes, such as the maximum and minimum concentrations, the frequency of samples exhibiting detectable analytes, and the proportion of samples with raised results. The data will be presented in a tabular format. These inspections and statistical analyses will be designed to:

- Identify deviations, if any, from the field sampling SOPs.
- Identify deviations, if any, from the laboratory analytical methods.
- Identify deviations, if any, from the QAPP.
- Identify deviations, if any, from the data validation process.
- Evaluate effects of the above-listed deviations from planned procedures and processes on the interpretation and utility of the data.
- Identify elevated detection limits and explain their impacts on the attainment of project objectives.
- Identify unusable data (i.e., data qualified as “R”).
- Evaluate project assumptions.
- Identify unanticipated data set characteristics such as a laboratory variance greater than the sampling variance (i.e., ANOVA, *t*-test) if enough data are available.
- Identify and evaluate potential data outliers (95% confidence goodness-of-fit test on probability plot data). The plotted data will be transformed, if necessary, depending on the observed distribution.
- Evaluate adherence to investigation objectives and decision rules.

- Ensure completion of corrective actions.

- Identify the existence of remaining data gaps.

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

Statistical tests for outliers will be conducted using standard statistical techniques appropriate for this task. Potential outliers will be removed if a review of field and laboratory documents indicates that the results are true outliers. If no physical cause for a statistical outlier can be identified, the data point will not be removed from the data set. However, if the data point is found to truly represent a physical quantity that is different from the rest of the data set, it will be removed. A suspected data outlier will not be removed from consideration unless a sampling or analytical error can be identified.

The suitability of any given statistical test will be assessed based on the completeness of the data sets and the conditions observed at the site. For example, when a single data value is available for soils or water samples at a given sampling location, statistical tests cannot be conducted for that individual sampling location. However, pooling of data across sampling locations may be possible and, if logical to do so, may be implemented at the discretion of the TtNUS Project Manager. Statistical testing will generally be conducted at the five percent significance level. Statistical testing at other significance levels may also be warranted to provide perspective on the results of testing at five percent significance. If other significance levels are used, they will be supported with rationales for their use.

4.2.3.1 Data Limitations and Actions from Usability Assessment

After all data evaluations are completed, any limitations on the use of data will be known and the limitations will be considered during decision making. If necessary, investigation objectives may be revised in anticipation of additional data collection in order to meet project quality objectives for the site.

Because analytical performance is generally similar for groundwater and surface water and for some soil and sediment, two matrix groups (solids and aqueous) may be used when discussing bias and precision. These groupings also cause the number of samples within a group to be greater than when the matrices are discussed separately. The increased number of samples in a group better supports statistical

analyses of the data. Sediment with a high percent moisture (>50%) will be discussed separately from soil as these types of sediment cannot be considered part of the same population as soil samples.

4.2.3.1.1 Precision

Field and laboratory precision indicators will be reviewed. In general, laboratory precision should be no worse than field precision because laboratory replicate samples have had an additional opportunity to be homogenized beyond the homogenization applied to field samples. If the laboratory precision is detected to be worse than field precision and the difference is judged to be significant the cause of the difference will be investigated and an appropriate corrective action will be devised. In general, RPD values (see Section 1.6.2.1) from laboratory and field duplicates will be used for this evaluation.

4.2.3.1.2 Accuracy/Bias

Overall contamination (both field and laboratory) will be reviewed and assessed for impact to the project. In general, this will be reflected in the assignment of data validation qualifiers that have accounted for the effect of blank contamination.

LCS, LCSD, MS, and MSD percent recovery results (see Section 1.6.2.2) will also be used to assess the degree of bias to be expected for various analyte groups and, to the extent that it is warranted, for individual analytes. Whether review and discussion to the analyte level is warranted depends on which analytes exhibit unacceptable bias and whether they are of importance to this project. In general, MS and MSD data will be used as a more accurate indication of bias in site samples than LCS and LCSD data.

Percent recovery values between 75 and 125% will generally be considered not be significant. Biases between 40 and 75% or 125 to 160% will be considered to be moderate. Biases between 10 and 40% or 160 to 190% will generally be considered to be severe. Biases less than 10 percent or greater than 190% will generally be considered to be extreme.

If enough data are available and multiple field events are conducted, precision may be compared for the multiple field events at the discretion of the TtNUS project manager. The usability assessment will strive to indicate limitations of the data caused by positive or negative bias and will attempt to do so based on logical groupings such as individual or multiple sample delivery groups, matrices, etc.

4.2.3.1.3 Representativeness

Representativeness (see Section 1.6.2.4) will be evaluated by reviewing field logs and analytical results to verify that project requirements were stratified for these operations. Special emphasis will be placed

on determining whether or not all intended samples were collected from the intended locations. If they were not, an assessment on the attainment of project objectives will be made. Based on these assessments a qualitative value judgment will be made concerning the representativeness of the samples and associated data. Representativness may also be assessed during the evaluation of precision and accuracy and will be summarized in the precision and accuracy assessments as applicable. An inclusion of representativeness of data for risk assessment may also be made when, for example, chemicals may be partitioned between various physical phases of a sample such as the dissolved and suspended solids of a groundwater or surface water sample.

4.2.3.1.4 Comparability

Comparability (see Section 1.6.2.5) will be assessed by first ensuring that QAPP requirements were satisfied. In addition, the comparability of data from one sample group to another may be assessed if the precision, accuracy or sensitivity indicates that the data are not or may not be comparable in terms of those quality indicators. If multiple sampling rounds are undertaken the comparability of multiple sets of data for the same chemical parameters and matrices will be assessed in terms of precision, accuracy, sensitivity, and representativeness.

In the case of upgradient data, comparability of the upgradient and site data sets will be described in terms of their representativeness for the intended populations, and any other factors such as precision, accuracy, and sensitivity of analytical results that either support or inhibit comparisons of the two types of data.

4.2.3.1.5 Sensitivity and Quantitation Limits

The range of non-detect values, which represent the sensitivity of the analytical measurements, will be compared to QAPP target detection and quantitation limits. This comparison will identify if actual sensitivity was sufficient enough to meet the QAPP targets. If the targets were not met, a discussion will be presented in the final report to establish the impact of not meeting the targets on the attainment of project objectives.

4.2.3.1.6 Completeness

Completeness will be evaluated on the following levels:

- Number of samples collected compared to number of samples scheduled for collection, by matrix.
- Number of valid results compared to number of planned results, by matrix, by analytical group.

In addition, a tally of the rejected values and reasons for rejection will be presented. Where rejections are identified the impact on attainment of project objectives will be described.

Special emphasis will be placed on discussing completeness deficiencies that have the greatest impact on the project. Completeness deficiencies that are inconsequential to the attainment of project objectives will be identified but will receive little discussion.

4.2.3.2 Activities

Several activities to be undertaken by the project team to assess data usability are described above. When the usability assessment is completed the project team will be able to:

- Establish whether intended samples were collected for each matrix and whether the samples and associated data are of sufficient quality to support the attainment of project objectives.
- If data for any scheduled samples are not available (e.g., because the samples were not analyzed or the data were rejected for use), identify the impact of the missing data on the project.
- Identify all PARCSS parameters that were not satisfied and in which samples, matrices, analytical groups, etc., they were not satisfied.
- Identify data use restrictions for any data that did not meet project quality objectives or otherwise are insufficient to support the attainment of project objectives.
- Identify corrective actions necessary to recover from missing data or data of insufficient quality.

4.3 STREAMLINING DATA REVIEW

Streamlining data review is not an applicable task for this project.

4.3.1 Data Review Steps to be Streamlined

Streamlining data review is not an applicable task for this project; therefore, no steps can be presented for streamlining.

4.3.2 Criteria for Streamlining

Streamlining data review is not an applicable task for this project; therefore, criteria for streamlining are not needed.

4.3.3 Amounts and Types of Data Appropriate for Streamlining

Streamlining data review is not an applicable task for this project; therefore, no data reviews will be streamlined.

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QAPP ATTACHMENTS

ATTACHMENT 1-A

UFP-QAPP WORKSHEETS

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WORKBOOK FOR UNIFORM FEDERAL POLICY FOR QUALITY ASSURANCE PROJECT PLANS

INTRODUCTION

The *Workbook for Uniform Federal Policy for Quality Assurance Project Plans* is Part 2A of the *Uniform Federal Policy for Quality Assurance Project Plans* (UFP-QAPP). It provides examples of worksheets to assist with the preparation of QAPPs in accordance with Part 1 of the UFP-QAPP (the UFP-QAPP Manual) and Section 6 (Part B) of *Quality Systems for Environmental Data and Technology Programs - Requirements with guidance for use*, ANSI/ASQ E4 (February 2004). This Workbook was be used by the lead organization and its contractors to assist with the preparation of this QAPP.

Each worksheet addresses specific requirements of the UFP-QAPP. Both the UFP-QAPP Manual and the Workbook are intended to be comprehensive and are not intended to be program-specific. Since the content and level of detail in a specific QAPP varies by program, by the work being performed, and by the intended use of the data, specific worksheets may not be applicable to all projects.

Though the format of each worksheet is not mandatory, the information required on the worksheets must still be presented in the QAPP, as appropriate to the project. QAPP preparers developed additional tables, as appropriate to the project. Written discussion in text format accompanies all tables. Certain sections, by their nature, require more written discussion than others. In particular, an in-depth explanation of the sampling design rationale is provided in the accompanying work plan, and Section 4 of the QAPP describes the procedures and criteria that will be used for data review.

QAPP_Worksheet_#1
(UFP-QAPP Section 2.1)
Title and Approval Page

Site Name/Project Name: Naval Air Station (NAS) Brunswick, Cumberland County, Maine/Site Investigation (SI)
Site Location: NAS Brunswick, Cumberland County, Maine

Document Title: Site Investigation (SI) Quality Assurance Project Plan (QAPP)

Lead Organization: Department of Navy (hereafter refered to as Navy)

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

Preparer's Address, Telephone Number, and E-mail Address: 661 Andersen Drive, Foster Plaza 7, Pittsburgh, PA 15220, 412-921-8250, linda.klink@ttnus.com

Preparation Date (Day/Month/Year): Revision 2 June 2009

Investigative Organization's Project Manager

Printed Name/Organization: Linda Klink, Tetra Tech NUS, Inc.

Investigative Organization's Project QA Officer

Printed Name/Organization: Tom Johnston, Tetra Tech NUS, Inc.

SEE SI WORK PLAN TITLE PAGE FOR SIGNATURES

Document_Control_Numbering_System : ____Not applicable

QAPP Worksheet #2
(UFP-QAPP Section 2.2.4)
QAPP Identifying Information

Site Name/Project Name: Naval Air Station (NAS) Brunswick/Site Investigation
Site Location: Cumberland County, Maine
Site Number/Code: NA
Operable Unit: NA
Contractor Name: Tetra Tech NUS, Inc.
Contractor Number: N62472-03-D-0057

Contract Title: NAVFAC Mid-Atlantic CLEAN
Work Assignment Number: CTO 069

Title: Site Investigation Quality Assurance Project Plan (QAPP)
Revision Number: 2
Revision Date: June 2009
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1. Identify regulatory program: Military Munitions Response Program (MMRP); National Contingency Plan; CERCLA
2. Identify approval entity: Maine Department of Environmental Protection (MEDEP) and USEPA Region 1
3. The QAPP is (select one): ☐Generic ☒Project Specific
4. List dates of scoping sessions that were held: See Malcolm Pirnie Final Site Inspection Work Plan June 2007
5. List dates and titles of QAPP documents written for previous site work, if applicable:

Title	Approval Date
Malcolm Pirnie Final Site Investigation Work Plan for NAS Brunswick,ME (Draft)	June 2007
Malcolm Pirnie Final Sampling and Analysis Plan for NAS Brunswick,ME (Draft)	June 2007

6. List organizational partners (stakeholders) and connection with lead organization:
MEDEP {regulatory stakeholder}, USEPA Region I {regulatory stakeholder}, Navy BRAC PMO NE {property owner}, Brunswick Area Citizens for a Safe Environment (BACSE), and Midcoast Regional Redevelopment Authority (MRRA)
7. List data users:
Navy
8. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusions below:

QAPP_Worksheet_#2
QAPP Identifying Information
(continued)

Identify where each required QAPP element is located in the QAPP (provide section, worksheet, table, or figure number) or other project planning documents (provide complete document title, date, section number, page numbers, and location of the information in the document). Type "NA" for the QAPP elements that are not applicable to the project. Provide an explanation in the QAPP.

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
Project Management and Objectives		
2.1 Title and Approval Page	- Title and Approval Page	UFP QAPP Worksheet 1 SI Work Plan (WP) and Sampling and Analysis Plan (SAP) Title/ Approval Page QAPP Section 1.1
2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.4 QAPP Identifying Information	- Table of Contents - QAPP Identifying Information	QAPP Table of contents UFP QAPP Worksheet 2 SI WP Section 1.2 QAPP Section 1.2
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	- Distribution List - Project Personnel Sign-Off Sheet	UFP QAPP Worksheets 3 and 4 SI Work Plan Title Page QAPP Section 1.3
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table	UFP QAPP Worksheets 5,6,7,and 8 SI WP Section 5.2 QAPP Section 1.4
2.5 Project Planning/Problem Definition 2.5.1 Project Planning (Scoping) 2.5.2 Problem Definition, Site History, and Background	- Project Planning Session Documentation (including Data Needs tables) - Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site Maps (historical and present)	UFP QAPP Worksheets 9 and 10 SI WP Sections 1 and 2 and SAP Section 3.1 QAPP Section 1.5 Attachment 1-E
2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria	- Site-Specific PQOs - Measurement Performance Criteria Table	UFP QAPP Worksheets 11 and 12 and SAP Section 3.0 QAPP Section 1.6

QAPP_Worksheet_#2
QAPP Identifying Information
(continued)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
2.7 Secondary Data Evaluation	- Sources of Secondary Data and Information - Secondary Data Criteria and Limitations Table	UFP QAPP Worksheet 13 and WP Sections 2.2 through 2.5 and SAP Section 3.1 QAPP Section 1.7
2.8 Project Overview and Schedule 2.8.1 Project Overview 2.8.2 Project Schedule	- Summary of Project Tasks - Reference Limits and Evaluation Table - Project Schedule/Timeline Table	UFP QAPP Worksheets 14, 15, and 16 WP Sections 3, 5.1 SAP Section 3 QAPP Section 1.8
Measurement/Data Acquisition		
3.1 Sampling Tasks 3.1.1 Sampling Process Design and Rationale 3.1.2 Sampling Procedures and Requirements 3.1.2.1 Sampling Collection Procedures 3.1.2.2 Sample Containers, Volume, and Preservation 3.1.2.3 Equipment/Sample Containers Cleaning and Decontamination Procedures 3.1.2.3 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures 3.1.2.4 Supply Inspection and Acceptance Procedures 3.1.2.6 Field Documentation Procedures	- Sampling Design and Rationale - Sample Location Map - Sampling Locations and Methods/SOP Requirements Table - Analytical Methods/SOP Requirements Table - Field Quality Control Sample Summary Table - Sampling SOPs - Project Sampling SOP References Table - Field Equipment Calibration, Maintenance, Testing, and Inspection Table	UFP QAPP Worksheets 17, 18, 19, 20, 21, and 22 SAP Section 4 QAPP Section 2.1 Attachment 1-B
3.2 Analytical Tasks 3.2.1 Analytical SOPs 3.2.2 Analytical Instrument Calibration Procedures 3.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures 3.2.4 Analytical Supply Inspection and Acceptance Procedures	- Analytical SOPs - Analytical SOP References Table - Analytical Instrument Calibration Table - Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	UFP QAPP Worksheets 23, 24, and 25 QAPP Section 2.2 Attachment 1-D

QAPP_Worksheet_#2
QAPP Identifying Information
(continued)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Required Documents
3.3 Sample Collection Documentation, Handling, Tracking, and Custody Procedures 3.3.1 Sample Collection Documentation 3.3.2 Sample Handling and Tracking System 3.3.3 Sample Custody	- Sample Collection Documentation Handling, Tracking, and Custody SOPs - Sample Container Identification - Sample Handling Flow Diagram - Example Chain-of-Custody Form and Seal	UFP QAPP Worksheets 26 and 27 QAPP Section 2.3 Attachment 1-B Attachment 1-C SAP Sections 4.8, 4.14 and 5.0
3.4 Quality Control Samples 3.4.1 Sampling Quality Control Samples 3.4.2 Analytical Quality Control Samples	- QC Samples Table - Screening/Confirmatory Analysis Decision Tree	UFP QAPP Worksheet 28 SAP 4.17 QAPP Section 2.4
3.5 Data Management Tasks 3.5.1 Project Documentation and Records 3.5.2 Data Package Deliverables 3.5.3 Data Reporting Formats 3.5.4 Data Handling and Management 3.5.5 Data Tracking and Control	- Project Documents and Records Table - Analytical Services Table - Data Management SOPs	UFP QAPP Worksheets 29 and 30 WP Sections 3.3.1 and 6 SAP Sections 4.8 and 5 QAPP Section 2.5
Assessment/Oversight		
4.1 Assessments and Response Actions 4.1.1 Planned Assessments 4.1.2 Assessment Findings and Corrective Action Responses	- Assessments and Response Actions - Planned Project Assessments Table - Audit Checklists - Assessment Findings and Corrective Action Responses Table	UFP QAPP Worksheets 31 and 32 WP Section 3.3.1 SAP Sections 3.2, 4.1, 4.4, and 5.1 QAPP Section 3.1
4.2 QA Management Reports	- QA Management Reports Table	UFP QAPP Worksheet 33 and SAP Section 5.1 QAPP Section 3.2
4.3 Final Project Report		WP Section 6.0 QAPP Section 3.3

QAPP_Worksheet_#2
QAPP Identifying Information
(continued)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
Data Review		
5.1 Overview		QAPP Section 4.1
5.2 Data Review Steps 5.2.1 Step I: Verification 5.2.2 Step II: Validation 5.2.2.1 Step IIa Validation Activities 5.2.2.2 Step IIb Validation Activities 5.2.3 Step III: Usability Assessment 5.2.3.1 Data Limitations and Actions from Usability Assessment 5.2.3.2 Activities	- Verification (Step I) Process Table - Validation (Steps IIa and IIb) Process Table - Validation (Steps IIa and IIb) Summary Table - Usability Assessment	UFP QAPP Worksheets 34, 35, 36, and 37 SAP Section 4 QAPP Section 4.2
5.3 Streamlining Data Review 5.3.1 Data Review Steps To Be Streamlined 5.3.2 Criteria for Streamlining Data Review 5.3.3 Amounts and Types of Data Appropriate for Streamlining		Not applicable

QAPP_Worksheet_#3

(UFP-QAPP Manual Section 2.3.1)

List those entities to whom copies of the approved QAPP, subsequent QAPP revisions, addenda, and amendments will be sent.

☐ Worksheet Not Applicable (State Reason)

QAPP Recipients	Title	Organization	Telephone Number	E-mail Address	Document Control Number
Mr. Todd Bober	Navy BRAC PMO NE Remedial Project Manager (RPM)	Navy BRAC PMO NE 4911 South Broad Street Philadelphia, PA 19112	215-897-4911	todd.bober@navy.mil	Not applicable
Mr. Paul Burgio (shared copy with Todd Bober)	Navy BRAC PMO NE Coordinator	Navy BRAC PMO NE 4911 South Broad Street Philadelphia, PA 19112	215-897-4915	paul.burgio@nvay.mil	Not applicable
Ms. Carolyn LePage	Technical Advisor to BASCE	LePage Environmental Services 731 Hotel Road Auburn, ME 04210	207-777-1049	calepage@adelphia.net	Not applicable
Victoria Boundy	Planning and Environmental Manager (MRRA)	Midcoast Regional Redevelopment Authority 5450 Fitch Avenue Brunswick, ME 04011	207-798-6512	victoriab@mrra.us	Not applicable
(shared copy) Ms. Jennifer Wright and Ms. Amy Van Dercook	Environmental Technical Support Biologist Environmental Scientist	NAVFAC Atlantic Attn: Code EV32JW 6506 Hampton Blvd Norfolk, VA 23508-1278 Jen (Code EV32JW); Amy (Code EV32AV)	757-322-8428 757-322-4764	Jennifer.H.Wright@navy.mil Amy.vandercook@navy.mil	Not applicable
Mr. David Barclift	Navy BRAC PMO NE Technical Support	Navy BRAC PMO NE 4911 South Broad Street Philadelphia, PA 19112	215-897-4913	david.barclift@navy.mil	Not applicable
Mr. Michael Fagan	NAS Brunswick IR Coordinator	NAS Brunswick Environmental Office 437 Huey Drive, Bldg. 53 Brunswick, ME 04011-5008	207-921-1717	Michael.Fagan1@navy.mil	Not applicable
(shared copy) Ms. Lisa Joy	NAS Brunswick Environmental Director	NAS Brunswick Environmental Office 437 Huey Drive, Bldg. 53 Brunswick, ME 04011-5008	207-921-1720	lisa.joy@navy.mil	Not applicable

QAPP Recipients	Title	Organization	Telephone Number	E-mail Address	Document Control Number
Mr. Michael Daly	Remedial Project Manager	USEPA Region I Federal Facilities Superfund Section 1 Congress Street, Suite 1100 (HBT) Boston, MA 02114-2023	617-918-1386	Daly.Mike@epamail.epa.gov	Not applicable
Ms. Claudia Sait	Remedial Project Manager	Maine Department of Environmental Protection	207-287-7713	claudia.b.sait@maine.gov	Not applicable
Mr. Chris Evans	Project Hydrogeologist	Maine Department of Environmental Protection Bureau of Remediation & Waste Management State House, Station 17 Augusta, ME 04333-0017	207-287-7656	Gordon.C.Evans@maine.gov	Not applicable
Mr. Peter Golonka	EPA Contractor	Gannett Fleming Suite 210 199 Wells Avenue Newton, MA 02459	---	pgolonka@gfnet.com	Not applicable
Mr. Dave McTigue	EPA Contractor	Gannett Fleming Suite 210 199 Wells Avenue Newton, MA 02459	---	dmctigue@gfnet.com	Not applicable
Mr. Richard Roedner	Dept. of Planning and Development	22 Elm Street Topsham, ME 04086	207-725-1724	rroedner@Topshammaine.com	Not applicable
Janice Shilling	Laboratory Project Manager	Empirical Laboratory 227French Landing Nashville, TN 37228	615-345-1115	mmcginnity@empirlabs.com	Not applicable
Deb Patton	Laboratory Project Manager	Columbia Analytical Services 1 Mustard Street, Suite 250 Rochester, NY 14609	585-288-5380	akeefer@caslab.com	Not applicable
Ms. Linda Klink	TtNUS Project Manager (PM)	Tetra Tech NUS, Inc. 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	412-921-8650	linda.klink@ttnus.com	Not applicable
Mr. Charles Race	Tetra Tech Maine Certified Geologist (C.G.)	Tetra Tech NUS, Inc. 55 Jonspin Road Wilmington, MA 01887-1020	978-474-8437	charles.race@tetratech.com	Not applicable

QAPP Recipients	Title	Organization	Telephone Number	E-mail Address	Document Control Number
Mr. Tom Johnston (shared copy with Tetra Tech PM & Chemist) Kelly Carper	Tetra Tech Quality Assurance Manager (QAM) Tetra Tech Project Chemist	Tetra Tech NUS, Inc. 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	412-921-8615 412-921-7273	tom.johnston@tetratech.com kelly.carper@tetratech.com	Not applicable
Mr. Joseph Samchuck	TtNUS Data Validation Manager (DVM)	Tetra Tech NUS, Inc. 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	412-921-8510	Joseph.samchuck@ttnus.com	Not applicable
Mr. Matt Soltis (HASP only)	TtNUS Health and Safety Manager (HSM)	Tetra Tech NUS, Inc. 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	412-921-8912	matt.soltis@ttnus.com	Not applicable
Mr. John Trepanowski	TtNUS Program Manager	Tetra Tech NUS, Inc. King of Prussia, PA	610-491-9688	john.trepanowski@ttnus.com	Not applicable
TBD	TtNUS Field Operations Leader (FOL) and field support	Tetra Tech NUS, Inc. 661 Andersen Drive Foster Plaza 7 Pittsburgh, PA 15220	TBD	TBD	Not applicable
Ms. Janet Fullerton	Head of Reference Desk (NASB Admin Record)	Curtis Memorial Library 23 Pleasant Street Brunswick, Maine 04011	---	---	Not applicable

QAPP Worksheet #4 (UFP-QAPP Manual Section 2.3.2)

Have copies of this form signed by key project personnel from each organization to indicate that they have read the applicable sections of the QAPP and will perform the tasks as described. Ask each organization to forward signed sheets to the central project file.

☐ Worksheet Not Applicable (State Reason)

Project Personnel Sign-Off Sheet

Organization: TtNUS

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Linda Klink	PM	412-921-8650	See Signature Page	
Charles Race	Maine Certified Geologist	978-474-8437	See Certification Page	
To Be Determined (TBD)	FOL, lead geologist, site safety officer	TBD		
Kelly Carper	Project Chemist	412-921-7273		
Matt Soltis	HSM	412-921-8912	See HASP signature page (separate internal Navy document)	
Joseph Samchuck	DVM	412-921-8510		

Project Personnel Sign-Off Sheet

Organization: Empirical Laboratory

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Janice Shilling	Laboratory PM	615-345-1115		
Randy Ward	Quality Assurance (QA) Director	615-345-1115		

Project Personnel Sign-Off Sheet

Organization: Columbia Analytical Services

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Deb Patton	Laboratory PM	585-288-5380		

QAPP_Worksheet_#5_____

(UFP-QAPP Manual Section 2.4.1)

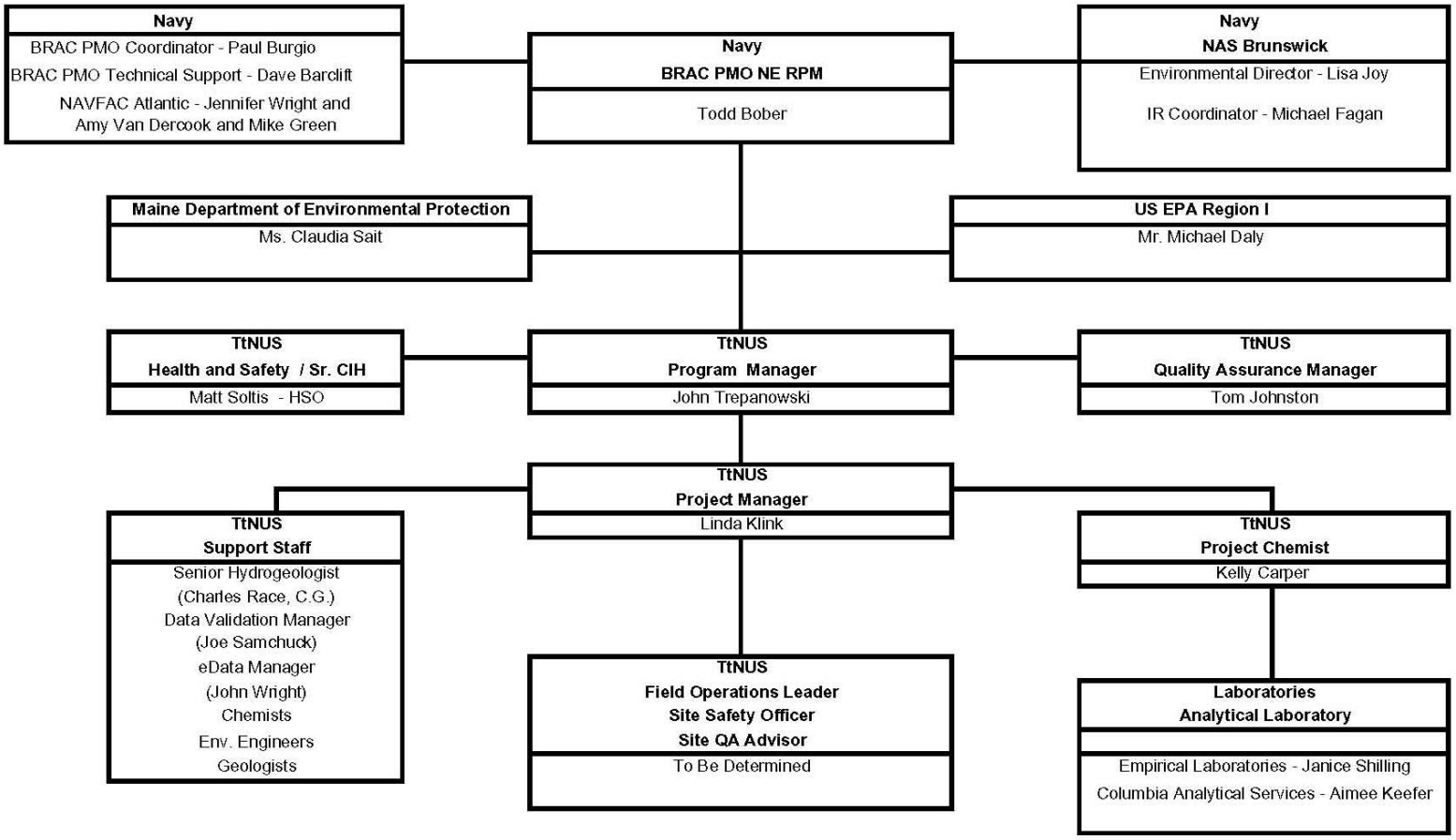
Identify reporting relationships between all organizations involved in the project, including the lead organization and all contractor and subcontractor organizations. Identify the organizations providing field sampling, on-site and off-site analysis, and data review services, including the names and telephone numbers of all project managers, project team members, and/or project contacts for each organization.

☐ Worksheet Not Applicable (State Reason) **See Attached Organization Chart**

QAPP Worksheet #5

☐ Worksheet Not Applicable (State Reason)

PROJECT ORGANIZATION CHART



QAPP_Worksheet_#6

(UFP-QAPP Manual Section 2.4.2)

Describe the communication pathways and modes of communication that will be used during the project, after the QAPP has been approved. Describe the procedures for soliciting and/or obtaining approval between project personnel, between different contractors, and between samplers and laboratory staff. Describe the procedure that will be followed when any project activity originally documented in an approved QAPP requires real-time modifications to achieve project goals or a QAPP amendment is required. Describe the procedures for stopping work and identify who is responsible.

☐ Worksheet Not Applicable (State Reason)

Communication Pathways

Communication_Drivers	Responsible_Entity	Name	Phone Number	Procedure_(Timing,_Pathways,_etc.)
Field task modification requests	TtNUS FOL	TBD	TBD	Immediately get approval from TtNUS PM Document via FTMR form
QAPP amendments	Navy RPM	Todd Bober	215-897-4911	Send scope change to TtNUS Program office
Changes in schedule	TtNUS PM	Linda Klink	412-921-8650	Inform Navy via schedule impact letter as soon as impact is realized
Issues in the field that result in changes in scope of field work	TtNUS FOL TtNUS PM TtNUS Maine C.G.	TBD Linda Klink Charles Race	 412-921-8650 978-474-8437	FOL inform PM and C.G.; PM inform RPM; RPM issue scope change if warranted; Scope change to be implemented before work is executed
Recommendations to stop work and initiate work upon corrective action	TtNUS FOL TtNUS PM TtNUS Maine C.G. TtNUS QAM TtNUS HSM Navy RPM	TBD Linda Klink Charles Race Kelly Carper Matt Soltis Todd Bober	 412-921-8650 978-474-8437 412-921-7273 412-921-8912 2151-897-4911	Responsible Party immediately informs subcontractors, the Navy, and Project Team
Analytical data quality issues	Analytical Laboratory TtNUS Project Chemist	Janice Shilling (Empirical) Aimee Keefer (Columbia Analytical Services) Kelly Carper	615-345-1115 585-288-5380 412-921-7273	Immediately notify TtNUS Project Chemist Notify Data Validation Staff and TtNUS PM if necessary

FTMR= Field Task Modification Request

QAPP Worksheet #7

(UFP-QAPP Manual Section 2.4.3)

Identify project personnel associated with each organization, contractor, and subcontractor participating in responsible roles. Include data users, decision-makers, project managers, QA officers, project contacts for organizations involved in the project, project health and safety officers, geotechnical engineers and hydrogeologists, field operation personnel, analytical services, and data reviewers. Identify project team members with an asterisk (*). Attach resume to this worksheet or note the location of the resumes.

☐ Worksheet Not Applicable (State Reason)

Personnel Responsibilities and Qualification Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Linda Klink*	PM	TtNUS	Oversees project, financial, schedule, and technical day to day management of the project.	M.S. Environmental Engineering, B.S. Chemical Engineering, 26 years professional engineering experience
Charles Race*	Maine Certified Geologist	TtNUS	Oversees geology/hydrogeology related tasks to ensure compliance with State of Maine requirements.	M.S. Geology; B.A. Geological Sciences, Maine and N.H. Certified Geologist, Mass. Site Licensed Professional, 26 years experience
TBD*	FOL, Lead Geologist, Project Safety Officer	TtNUS	Supervise, coordinate, and perform field sampling activities prepare all geological interpretation and text.	
Kelly Carper*	Project Chemist and QAM	TtNUS	Prepare QAPP, prepare lab scope, coordinate with lab, and data quality review. Ensure Quality aspescts of the CLEAN program.	B.S. Biology, 15 years environmental experience
Joseph Samchuck	DVM	TtNUS	Quality assurance of data validation deliverables.	B.S. Chemistry, MBA, M.S. Finance, 24 years environmental experience
Janice Shilling	Chemical Laboratory PM	Empirical Laboratory	Coordinate analyses with lab chemists, ensure the scope is followed, QA data packages, communicate with TtNUS staff.	Can be provided upon request
Matt Soltis	HSM	TtNUS	Oversee CLEAN Program safety	B.S. Industrial Safety Sciences, 24 years of environmental experience

QAPP_Worksheet_#8

(UFP-QAPP Manual Section 2.4.4)

Provide the following information for those projects requiring personnel with specialized training. Attach training records and/or certificates to the QAPP or note their location.

☐ Worksheet Not Applicable (State Reason)

Information provided in Health and Safety Plan and Section 1.4.4 of the QAPP

Special Personnel Training Requirements Table

Project Function	Specialized Training – Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates

QAPP Worksheet #9

(UFP-QAPP Manual Section 2.5.1)

Complete this worksheet for each project scoping session held. Identify project team members who are responsible for planning the project.

☐ Worksheet Not Applicable (State Reason)

Project Name: Projected Date(s) of Field Activity: **23-24 October 2007** Project Manager: **Linda Klink**	Site Name: **Site 12 EOD Area, Former Munitions Bunker West Area, Quarry, Machine Gun Boresight Range, Skeet Range, Topsham Skeet Range** Site Location: **Naval Air Station Brunswick, Brunswick, Maine** **NOTE: The Site 12 EOD, Former Munitions Bunker West, and Quarry have been removed from this document since this meeting.**

Date of Session: On 23-24 October 2007, members of the Tetra Tech team, NAS Brunswick personnel, and Stakeholders for MEDEP, EPA Region I, EPA contractors, and Brunswick Land Redevelopment Authority representatives met to discuss the scope of the SI project and participate in a site walk of the six MRP sites.

Scoping Session Purpose: The purpose of this site walk/meeting session was to verify the site-specific observations previously made by the Tetra Tech team during the initial site visit in May 2007 and obtain input from the Stakeholders.

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Orlando Monaco	Remedial Project Manager (RPM)	Navy BRAC PMO NE	215-897-4911	orlando.monaco@navy.mil	Same as title
Dawn Kincaid	BRAC Environmental Coordinator	Navy BRAC PMO NE	215-897-4915	dawn.kincaid@navy.mil	Same as title
Carolyn LePage	Technical Advisor to BASCE	LePage Environmental Services	207-777-1049	calepage@adelphia.net	Same as title
Carol Warren	Brunswick LRA	Brunswick LRA		carol@wacubu.com	
Amy Van Dercook	Environmental Technical Support – Environmental Scientist	NAVFAC Atlantic	757-322-4764	Amy.vandercook@navy.mil	Same as title
Dale Mosher	NAS Brunswick IR Coordinator	NAS Brunswick Environmental Office	207-921-1719	dale.mosher@navy.mil	Same as title
Lisa Joy	NAS Brunswick Environmental Director	NAS Brunswick Environmental Office	207-921-1720	lisa.joy@navy.mil	Same as title
Michael Daly	Remedial Project Manager	USEPA Region I Federal Facilities Superfund Section	617-918-1386	Daly.Mike@epamail.epa.gov	Same as title
Claudia Sait	Remedial Project Manager	MEDEP Bureau of Remediation & Waste Management	207-287-7713	claudia.b.stait@maine.gov	Same as title
Chris Evans	Project Hydrogeologist	MEDEP Bureau of Remediation & Waste Management	207-287-7656	Gordon.C.Evans@maine.gov	Same as title
Carol Stein	EPA Contractor	Gannett Fleming	---	cstein@gfnet.com	Same as title
Dave McTigue	EPA Contractor	Gannett Fleming	---	dmctigue@gfnet.com	Same as title
Linda Klink	Project Manager (PM)	Tetra Tech	412-921-8650	linda.klink@tetratech.com	Same as title
Charles Race	Maine Certified Geologist (CG)	Tetra Tech	978-474-8437	charles.race@tetratech.com	Same as title
Ralph Brooks	UXO Manager	Tetra Tech	770-413-0965 (Ext. 231)	ralph.brooks@tetratech.com	Same as title
Jim Coffman	Project Geophysicist	Tetra Tech	412-921-8244	jim.coffman@tetratech.com	Same as title

Comments/Decisions: On October 23 through October 24, 2007, a site visit of all 6 MRP areas was conducted for the benefit of the Stakeholders. The agenda, sign-in sheet, and action item list from the site visit is provided in Attachment 1-E.

Action Items: See Table 9-A, Action Items

Consensus Decisions: See Table 9-1, Action Items and Work Plan Appendix D-1 Addendum to Comment Response Letter.

TABLE 9-A
ACTION ITEMS
NAVAL AIR STATION BRUNSWICK, MAINE
OCTOBER 23 - 24, 2007 TEAM MEETING

ITEM NUMBER	PERSON(S) RESPONSIBLE	DESCRIPTION OF ACTION REQUIRED
1	1) Ralph Brooks 2) Mike Daly 3) Jim Coffman/ Linda Klink	**Machine Gun Boresight Range:** 1) Map dirt pile via GPS 2) Pursue EPA XRF sampling 3) Propose magnetic detection strategy for site original berm location, potential current berm location (dirt pile) and area in between
2	1) Chuck Race/Chris Evans 2) Claudia Sait/Chris Evans	**Skeet Range:** 1) Provide Linda Klink with info on adjacent Site 9 nearby existing wells (survey coordinates, boring logs, well construction, and analytical data) for 2) Provide team with analytical data for lead at adjacent Site 6
3	Linda Klink	**Site 12 EOD Area:** Incorporate historical aerials (from PA) on the EOD pit
4	Chris Evans/Claudia Sait	**Quarry:** Provide team with spill reports associated with historical PAH treatment area
5	1) All Team 2) Chris Evans/Claudia Sait 3) Linda Klink 4) Richard Roedner 5) Richard Roedner	**Topsham Skeet Range:** 1) Decide on use of XRF (if aerial photos do not definitively show site location) 2) Provide Linda Klink with Draft Report including info on existing wells (survey coordinates, boring logs, well construction, and analytical data) [not in Admin Record] 3) Evaluate analytical results for lead from 2004 EA Report 4) Provide team with Spring 2006 aerial photos 5) Obtain site access from property owner for field work (staking out sample locations, field work, and temporary monitoring well abandonment events)
6	1) Linda Klink 2) Dave McTigue 3) Linda Klink/Malcolm Pirnie	**General Research:** 1) Research ITRC suggestions on sampling of skeet ranges 2) Provide team with USGS Delaware, Otis Cape Cod report on skeet range 3) Obtain historical aerial photographs for the 6 MRP sites from Malcolm Pirnie PAs and incorporate into MEC and MC SI Work Plans
7	Linda Klink/Lonnie Monaco	**Scope Additions** Obtain funding of scope additions and transfer of MC SI Work Plan from Malcolm Pirnie to Tetra Tech
8	Tetra Tech	**Update of Malcolm Pirnie SI MC Work Plan and Figures:** Make changes to figures and conceptual models as per meeting discussions/site visits: All Figures: - Incorporate significant information from historical aerial photographs (Malcolm Pirnie, CERFA Report, and NAS Brunswick) and determine date of aerial used as background

ITEM NUMBER	PERSON(S) RESPONSIBLE	DESCRIPTION OF ACTION REQUIRED
		Skeet Range: - Show existing Site 9 well(s) and use if located at proposed well(s), as appropriate - Add 2 surface water/sediment samples for lead analyses (1 at tributary & 1 in detention pond; both within shotfall zone) - Move outer soil samples into shotfall zone Machine Gun Boresight Range: - Add dirt pile to figure and locate samples here as potential former berm - Correlate Malcolm Pirnie SI Work Plan figure with historical aerial - Add plans for XRF sampling of lead (no XRF confirmation samples necessary since to be used for screening purposes and for locating soil borings where fixed based laboratory analyses will be employed) - Add text to conceptual model concerning: - No evidence of berm at original location & presence of dirt pile further out - Notation that the Sites 1 and 3 landfills were not in operation at the time of the berm bulldozing operation in 1978 Site 12 EOD Area - Adjust proposed monitoring well locations to allow for better triangulation for development of potentiometric maps - Add text to conceptual model/rationale that wells may be dry based on findings of previous test pit activities. Note that bedrock wells are not warranted for the SI because they would require abandoning during munitions clearance; however, bedrock wells will be warranted for the future RI Former Munitions Bunker West - Evaluate change in vegetated area from historical aerials, if possible - Add perchlorate for aqueous analyses (groundwater and surface water) Quarry - Evaluate change in vegetated area and determine operational area, if possible - More clearly show PAH treatment area on figures - Add text to conceptual model concerning: - Presence of metal and other debris (e.g., barrel) near rock face and note historical use for dumping - Likely 18 inches of till in PAH treatment area - Documentation of spill reports associated w/ PAH treatment area - Documentation of lithology from previous effort (Appx C of PA) Topsham Skeet Range - Add 2 surface water/sediment samples (1 at brook and 1 in shotfall zone) - Delete surface water/sediment sample in detention basin - Move outer soil samples into shotfall zone - Show fill area and site location based on aerials, if possible - Add text to conceptual model/rationale concerning: - Most of the site is outside the NAS Brunswick boundary and so the Navy will not be able to place land use controls on the property - Documentation of previous XRF work in housing area in corner of the site

ITEM NUMBER	PERSON(S) RESPONSIBLE	DESCRIPTION OF ACTION REQUIRED
9	Ralph Brooks/Linda Klink/ Jim Coffman	**Update of Tetra Tech SI MEC Work Plan and Figures** Make changes to figures and conceptual models as per meeting discussions/site visits: Site 12 EOD Area - Add horizontal grid lines in open area for munitions sweep Former Munitions Bunker West - Evaluate change in vegetated area from historical aerials, if possible - Add text to conceptual model/rationale that the wooded area will need to be addressed in the future during an RI (along with intrusive investigation of anomalies likely to occur during the RI phase) Quarry - Evaluate change in vegetated area and determine operational area, if possible - Limitations of geophysical survey instrumentation in PAH treatment area based on 18 inches of till
10	1) Mike Daly 2) Linda Klink	**Regulatory Criteria:** 1) Provide Linda Klink with current sediment criteria to update Malcolm Pirnie MC Work Plan 2) Provide team with proposed aqueous detection limits for antimony and arsenic (concern for skeet range sites)
11	1) Linda Klink 2) All Team	**11/13/07 Conference Call:** 1) Set up conference call and e-mail dial-in information to team 2) Participate in conference call

CERFA	Community Environmental Response Facilitation Act
PA	Preliminary Assessment
EOD	Explosive Ordnance Disposal
PAH	Polynuclear aromatic hydrocarbon
GPS	Global Positioning System
RI	Remedial Investigation
ITRC	Interstate Technology and Regulatory Council
SI	Site Inspection
MC	Munitions Constituents
USGS	United States Geological Survey
MEC	Munitions and Explosives of Concern
XRF	X-Ray Fluorescence
MRP	Munitions Response Program

Several scoping meetings were held via conference calls with TtNUS, Malcolm Pirnie, Navy, and Regulators. Site Visits were conducted by Malcolm Pirnie during the Preliminary Assessment process. Regulatory Agencies have also visited the sites at various times.

QAPP_Worksheet_#10

(UFP-QAPP Manual Section 2.5.2)

Clearly define the problem and the environmental questions that should be answered for the current investigation and develop the project decision "If…, then…" statements in the QAPP, linking data results with possible actions. The prompts below are meant to help the project team define the problem. They are not comprehensive.

☐ Worksheet Not Applicable (State Reason)

Detailed in Section 3.1 of the SAP

Problem Definition

The problem to be addressed by the project:
The environmental questions being asked:
Observations from any site reconnaissance reports:
A synopsis of secondary data or information from site reports:
The possible classes of contaminants and the affected matrices:
The rationale for inclusion of chemical and nonchemical analyses:
Information concerning various environmental indicators:
Project decision conditions ("If..., then..." statements):

QAPP_Worksheet_#11

(UFP-QAPP Manual Section 2.6.1)

Use this worksheet to develop project quality objectives (PQOs) in terms of type, quantity, and quality of data determined using a systematic planning process. Provide a detailed discussion of PQOs in the QAPP. List PQOs in the form of qualitative and quantitative statements. These statements should answer questions such as those listed below. These questions are examples only, however; they are neither inclusive nor appropriate for all projects.

☐ Worksheet Not Applicable (State Reason)

Project_Quality_Objectives /Systematic Planning Process Statements

Who will use the data? Navy and TtNUS and Stakeholders
What will the data be used for? Data will be used to determine the presence or absence of munitions constituents at these sites.
What type of data are needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques) Definitive fixed based laboratory data is required. Soil, groundwater, surface water, and sediment samples will be collected, as applicable, and analyzed for explosives (nitrotoluenes only), propellants (nitroglycerin only), total organic carbon, pH, CEC, metals, perchlorate, nitrates, and polycyclic aromatic hydrocarbons. Analyses vary by site based on site history.
How "good" do the data need to be in order to support the environmental decision? Data must be of the fullest level of quality control and documentation, such that a no further action (NFA) decision can be supported or the data can be used to support a future remedial investigation (RI).
How much data are needed? (number of samples for each analytical group, matrix, and concentration) The number of samples to be collected was developed by the team during the October 2007 meeting/site visit and subsequently revisited during routine Stakeholder meetings, conference calls, and e-mails..
Where, when, and how should the data be collected/generated? Collection will be in accordance with the SAP.
Who will collect and generate the data? Samples will be collected by TtNUS and analyzed for chemical parameters by Empirical Laboratory and perchlorate by Columbia Analytical Services.
How will the data be reported? Chemical data will be delivered in contract laboratory program-like sample delivery groups. Chemical data will also be received in an electronic data deliverable that will be used to create a NAS Brunswick database. Data will be summarized in the Site Inspection report.
How will the data be archived? Once data are validated they will be uploaded to the master NAS Brunswick database and eventually added to the NIRIS database. Hardcopy data packages will be stored by a data storage contractor until they are relinquished to the Navy.

A more detailed description of the project quality objectives for this SI is found in Section 3.1 of the SAP.

QAPP Worksheet #12

(UFP-QAPP Manual Section 2.6.2)
Complete this worksheet for each matrix, analytical group, and concentration level. Identify the data quality indicators (DQIs), measurement performance criteria (MPC), and QC sample and/or activity used to assess the measurement performance for both the sampling and analytical measurement systems. Use additional worksheets if necessary. If MPC for a specific DQI vary within an analytical parameter, i.e., MPC are analyte-specific, then provide analyte-specific MPC on an additional worksheet.

☐ Worksheet Not Applicable (State Reason)

Measurement Performance Criteria Table

Matrix	Groundwater/
Analytical Group	Explosives
Concentration Level	Low/Medium

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
S5 and S13	SW-846 8330A Empirical/SOP-327	Bias / Contamination	No target analytes ≥1/2 QL	Method Blank	A
S5 and S13	SW-846 8330A Empirical/SOP-327	Accuracy / Bias	Statistically derived limits.	Laboratory Control Spike and Surrogate Standards	A
S5 and S13	SW-846 8330A Empirical/SOP-327	Accuracy / Bias / Precision	Statistically derived limits, 30%RPD	Matrix spike/matrix spike duplicate	A
S5 and S13	SW-846 8330A Empirical/SOP-327	Precision	Values > 5X QL: ≤ 30% RPD	Field Duplicates	S & A
S5 and S13	SW-846 8330A Empirical/SOP-327	Bias / Contamination	No target analytes ≥ QL	Equipment/Rinsate Blanks	S
S5 and S13	SW-846 8330A Empirical/SOP-327	Accuracy / Representativeness	Between 2 and 6 degrees C.	Cooler Temperature Indicator	S
S5 and S13	SW-846 8330A Empirical/SOP-327	Data Completeness	95% Overall	Data Completeness Check	S & A
S5 and S13	SW-846 8330A Empirical/SOP-327	Comparability	Values > 5X QL: Field Replicates ≤ 30% RPD	Comparability Check	S & A
S5 and S13	SW-846 8330A Empirical/SOP-327	Sensitivity	Quantiation limits less than project action limits listed in Worksheet 15	Comparability and sensitivity check	S&A

Matrix	Groundwater
Analytical Group	Nitroglycerin
Concentration Level	Low/Medium

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
S5 and S13	SW-846 8332/ Empirical SOP-327	Bias / Contamination	No target analytes ≥1/2 QL	Method Blank	A
S5 and S13	SW-846 8332/ Empirical SOP-327	Accuracy / Bias	Statistically derived limits.	Laboratory Control Spike and Surrogate Standards	A
S5 and S13	SW-846 8332/ Empirical SOP-327	Accuracy / Bias / Precision	Statistically derived limits, 30% RPD	Matrix spike/matrix spike duplicate	A
S5 and S13	SW-846 8332/ Empirical SOP-327	Precision	Values > 5X QL: ≤ 30% RPD	Field Duplicates	S & A
S5 and S13	SW-846 8332/ Empirical SOP-327	Bias / Contamination	No target analytes ≥ QL	Equipment/Rinsate Blanks	S
S5 and S13	SW-846 8332/ Empirical SOP-327	Accuracy / Representativeness	Between 2 and 6 degrees C.	Cooler Temperature Indicator	S
S5 and S13	SW-846 8332/ Empirical SOP-327	Data Completeness	95% Overall	Data Completeness Check	S & A
S5 and S13	SW-846 8332/ Empirical SOP-327	Comparability	Values > 5X QL: Field Replicates ≤ 30% RPD	Comparability Check	S & A
S5 and S13	SW-846 8332/ Empirical SOP-327	Sensitivity	Quantiation limits less than project action limits listed in Worksheet 15	Comparability and sensitivity check	S&A

Matrix	Groundwater/Surface water
Analytical Group	Polycyclic Aromatic Hydro carbons (PAHs)
Concentration Level	Low/Medium

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
S5 and S13	SW-846 8270C/ Empirical SOP-231/300	Bias / Contamination	No target analytes ≥1/2 QL	Method Blank	A
S5 and S13	SW-846 8270C/ Empirical SOP-231/300	Accuracy / Bias / Precision	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Internal Standards	A
S5 and S13	SW-846 8270C / Empirical SOP-231/300	Accuracy / Bias	Statistically derived limits.	Laboratory Control Spike and Surrogate Standards	A
S5 and S13	SW-846 8270C / Empirical SOP-231/300	Accuracy / Bias / Precision	Statistically derived limits, 30% RPD	Matrix spike/matrix spike duplicate	A
S5 and S13	SW-846 8270C/ Empirical SOP-231/300	Precision	Values > 5X QL: ≤ 30% RPD	Field Duplicates	S & A
S5 and S13	SW-846 8270C/ Empirical SOP-231/300	Bias / Contamination	No target analytes ≥ QL	Equipment/Rinsate Blanks	S
S5 and S13	SW-846 8270C/ Empirical SOP-231/300	Accuracy / Representativeness	Between 2 and 6 degrees C.	Cooler Temperature Indicator	S
S5 and S13	SW-846 8270C/ Empirical SOP-231/300	Data Completeness	95% Overall	Data Completeness Check	S & A
S5 and S13	SW-846 8270C/ Empirical SOP-231/300	Comparability	Values > 5X QL: Field Replicates ≤ 30%	Comparability Check	S & A
S5 and S13	SW-846 8270C/ Empirical SOP-231/300	Sensitivity	Quantiation limits less than project action limits listed in Worksheet 15	Comparability and sensitivity check	S&A

Matrix	Groundwater
Analytical Group	Perchlorate
Concentration Level	Low/Medium

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
S5 and S13	SW-846 6850 / CAS HPLC-DoDPerchlorate	Bias / Contamination	No target analytes ≥1/2 QL;	Method Blank	A
S5 and S13	SW-846 6850 / CAS HPLC-DoDPerchlorate	Accuracy / Bias	80-120% Recovery LCS	Laboratory Control Spike	A
S5 and S13	SW-846 6850 / CAS HPLC-DoDPerchlorate	Accuracy / Bias / Precision	80-120% Recovery, 15% RPD	Matrix spike/matrix spike duplicate	A
S5 and S13	SW-846 6850 / CAS HPLC-DoDPerchlorate	Precision	Values > 5X QL: ≤ 30% RPD	Field Duplicates	S & A
S5 and S13	SW-846 6850 / CAS HPLC-DoDPerchlorate	Bias / Contamination	No target analytes ≥ QL	Equipment/Rinsate Blanks	S
S5 and S13	SW-846 6850 / CAS HPLC-DoDPerchlorate	Accuracy / Representativeness	Between 2 and 6 degrees C.	Cooler Temperature Indicator	S
S5 and S13	SW-846 6850 / CAS HPLC-DoDPerchlorate	Data Completeness	95% Overall	Data Completeness Check	S & A
S5 and S13	SW-846 6850 / CAS HPLC-DoDPerchlorate	Comparability	Values > 5X QL: Field Replicates ≤ 30% RPD	Comparability Check	S & A
S5 and S13	SW-846 6850 / CAS HPLC-DoDPerchlorate	Sensitivity	Quantiation limits less than project action limits listed in Worksheet 15	Comparability and sensitivity check	S&A

Matrix	Groundwater/Surface Water
Analytical Group	TAL Metals
Concentration Level	Low/Medium

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
S5 and S13	SW-846 6010c/7470A/ Empirical SOP-100/105/103	Bias / Contamination	No target analytes ≥1/2 QL	Method Blank	A
S5 and S13	SW-846 6010B6010B/7470A/ Empirical SOP-100/105/103	Accuracy / Bias	± 20% Recovery of true value	Laboratory Control Sample	A
S5 and S13	SW-846 6010B/7470A/ Empirical SOP-100/105/103	Accuracy / Bias	± 25% Recovery of true value if sample < 4x spike added	Matrix Spike Sample	A
S5 and S13	SW-846 6010B/7470A/ Empirical SOP-100/105/103	Precision	Values ≥ 5X QL: RPD ≤20%	Duplicate	S & A
S5 and S13	SW-846 6010B/7470A/ Empirical SOP-100/105/103	Accuracy / Bias	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	ICP Serial Dilution	A
S5 and S13	SW-846 6010B/7470A/ Empirical SOP-100/105/103	Precision	Values ≥ 5X QL: RPD ≤ 30%	Field Duplicates	S & A
S5 and S13	SW-846 6010B/7470A/ Empirical SOP-100/105/103	Bias / Contamination	No target analytes ≥ QL	Equipment/Rinsate Blanks	S
S5 and S13	SW-846 6010B/7470A/ Empirical SOP-100/105/103	Accuracy / Representativeness	Between 2 and 6 degrees C.	Cooler Temperature Indicator	S
S5 and S13	SW-846 6010B/7470A/ Empirical SOP-100/105/103	Data Completeness	95% Overall	Data Completeness Check	S & A
S5 and S13	SW-846 6010B/7470A/ Empirical SOP-100/105/103	Comparability	Values ≥ 5X QL: Field Duplicates; RPD ≤ 30%	Comparability Check	S & A

Matrix	Groundwater/Surface Water
Analytical Group	TAL Metals
Concentration Level	Low/Medium

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
S5 and S13	SW-846 6010B/7470A/ Empirical SOP-100/105/103	Sensitivity	Quantiation limits less than project action limits listed in Worksheet 15	Comparability and sensitivity check	S&A

Matrix	Soil
Analytical Group	Explosives
Concentration Level	Low/Medium

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
S3 and S12	SW-846 8330A Empirical/SOP-327	Bias / Contamination	No target analytes ≥1/2 QL; with the exception of common field/laboratory contaminants	Method Blank	A
S3 and S12	SW-846 8330A Empirical/SOP-327	Bias / Contamination	No target analytes ≥ QL; with the exception of common field/laboratory contaminants	Trip Blank	S &A
S3 and S12	SW-846 8330A Empirical/SOP-327	Accuracy / Bias	Statistically derived limits.	Laboratory Control Spike and Surrogate Standards	A
S3 and S12	SW-846 8330A Empirical/SOP-327	Accuracy / Bias / Precision	Statistically derived limits, ≤ 30% RPD	Matrix spike/matrix spike duplicate	A
S3 and S12	SW-846 8330A Empirical/SOP-327	Precision	Values > 5X QL: ≤ 50% RPD	Field Duplicates	S & A
S3 and S12	SW-846 8330A Empirical/SOP-327	Bias / Contamination	No target analytes ≥ QL; with the exception of common field/laboratory contaminants	Equipment/Rinsate Blanks	S
S3 and S12	SW-846 8330A Empirical/SOP-327	Accuracy / Representativeness	Between 2 and 6 degrees C.	Cooler Temperature Indicator	S
S3 and S12	SW-846 8330A Empirical/SOP-327	Data Completeness	95% Overall	Data Completeness Check	S & A
S3 and S12	SW-846 8330A Empirical/SOP-327	Comparability	Values > 5X QL: Field Replicates ≤ 50% RPD	Comparability Check	S & A
S3 and S12	SW-846 8330A Empirical/SOP-327	Sensitivity	Quantiation limits less than project action limits listed in Worksheet 15	Comparability and sensitivity check	S&A

Matrix	Soil
Analytical Group	Nitroglycerin
Concentration Level	Low/Medium

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
S3 and S12	SW-846 8332/ Empirical SOP-327	Bias / Contamination	No target analytes ≥1/2 QL	Method Blank	A
S3 and S12	SW-846 8332/ Empirical SOP-327	Accuracy / Bias	Statistically derived limits.	Laboratory Control Spike and Surrogate Standards	A
S3 and S12	SW-846 8332/ Empirical SOP-327	Accuracy / Bias / Precision	Statistically derived limits, 30% RPD	Matrix spike/matrix spike duplicate	A
S3 and S12	SW-846 8332/ Empirical SOP-327	Precision	Values > 5X QL: ≤ 50% RPD	Field Duplicates	S & A
S3 and S12	SW-846 8332/ Empirical SOP-327	Bias / Contamination	No target analytes ≥ QL	Equipment/Rinsate Blanks	S
S3 and S12	SW-846 8332/ Empirical SOP-327	Accuracy / Representativeness	Between 2 and 6 degrees C.	Cooler Temperature Indicator	S
S3 and S12	SW-846 8332/ Empirical SOP-327	Data Completeness	95% Overall	Data Completeness Check	S & A
S3 and S12	SW-846 8332/ Empirical SOP-327	Comparability	Values > 5X QL: Field Replicates ≤ 50% RPD	Comparability Check	S & A
S3 and S12	SW-846 8332/ Empirical SOP-327	Sensitivity	Quantiation limits less than project action limits listed in Worksheet 15	Comparability and sensitivity check	S&A

Matrix	Soil/Sediment
Analytical Group	Polycyclic Aromatic Hydro carbons (PAHs)
Concentration Level	Low/Medium

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
S3 and S12	SW-846 8270C / Empirical SOP-231/329	Bias / Contamination	No target analytes ≥1/2 QL; with the exception of common field/laboratory contaminants which should be <RL	Method Blank	A
S3 and S12	SW-846 8270C / Empirical SOP-231/329	Accuracy / Bias / Precision	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Internal Standards	A
S3 and S12	SW-846 8270C / Empirical SOP-231/329	Accuracy / Bias	Statistically derived limits.	Laboratory Control Spike and Surrogate Standards	A
S3 and S12	SW-846 8270C / Empirical SOP-231/329	Accuracy / Bias / Precision	Statistically derived limits, 30% RPD	Matrix spike/matrix spike duplicate	A
S3 and S12	SW-846 8270C / Empirical SOP-231/329	Precision	Values > 5X QL: ≤ 50% RPD	Field Duplicates	S & A
S3 and S12	SW-846 8270C / Empirical SOP-231/329	Bias / Contamination	No target analytes ≥ QL; with the exception of common field/laboratory contaminants which should be <RL	Equipment/Rinsate Blanks	S
S3 and S12	SW-846 8270C / Empirical SOP-231/329	Accuracy / Representativeness	Between 2 and 6 degrees C.	Cooler Temperature Indicator	S
S3 and S12	SW-846 8270C / Empirical SOP-231/329	Data Completeness	95% Overall	Data Completeness Check	S & A
S3 and S12	SW-846 8270C / Empirical SOP-231/329	Comparability	Values > 5X QL: Field Replicates ≤ 50%	Comparability Check	S & A
S3 and S12	SW-846 8270C / Empirical SOP-231/329	Sensitivity	Quantiation limits less than project action limits listed in Worksheet 15	Comparability and sensitivity check	S&A

Matrix	Soil/Sediment
Analytical Group	TAL Metals
Concentration Level	Low/Medium

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
S3 and S12	SW-846 6010C/7471A/ Empirical SOP-100/105/104	Bias / Contamination	No target analytes ≥1/2 QL	Method Blank	A
S3 and S12	SW-846 6010B/7471A/ Empirical SOP-100/105/104	Accuracy / Bias	± 20% Recovery of true value	Laboratory Control Sample	A
S3 and S12	SW-846 6010B/7471A/ Empirical SOP-100/105/104	Accuracy / Bias	± 25% Recovery of true value if sample < 4x spike added	Matrix Spike Sample	A
S3 and S12	SW-846 6010B/7471A/ Empirical SOP-100/105/104	Precision	Values ≥ 5X QL: RPD ≤20%	Duplicate	S & A
S3 and S12	SW-846 6010B/7471A/ Empirical SOP-100/105/104	Accuracy / Bias	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	ICP Serial Dilution	A
S3 and S12	SW-846 6010B/7471A/ Empirical SOP-100/105/104	Precision	Values ≥ 5X QL: RPD ≤ 50%	Field Duplicates	S & A
S3 and S12	SW-846 6010B/7471A/ Empirical SOP-100/105/104	Bias / Contamination	No target analytes ≥ QL	Equipment/Rinsate Blanks	S
S3 and S12	SW-846 6010B/7471A/ Empirical SOP-100/105/104	Accuracy / Representativeness	Between 2 and 6 degrees C.	Cooler Temperature Indicator	S
S3 and S12	SW-846 6010B/7471A/ Empirical SOP-100/105/104	Data Completeness	95% Overall	Data Completeness Check	S & A
S3 and S12	SW-846 6010B/7471A/ Empirical SOP-100/105/104	Comparability	Values ≥ 5X QL: Field Duplicates; RPD ≤ 50%	Comparability Check	S & A

Matrix	Soil/Sediment
Analytical Group	TAL Metals
Concentration Level	Low/Medium

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
S3 and S12	SW-846 6010B/7471A/ Empirical SOP-100/105/104	Sensitivity	Quantiation limits less than project action limits listed in Worksheet 15	Comparability and sensitivity check	S&A

Matrix	Soil
Analytical Group	TCLP VOC
Concentration Level	Low/Medium

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
S3	SW-846 1311/8260B/ Empiircal SOP-198/202	Bias / Contamination	No target analytes ≥ QL; with the exception of common field/laboratory contaminants	Method Blank	A
S3	SW-846 1311/8260B/ Empiircal SOP-198/202	Accuracy / Bias / Precision	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Internal Standards	A
S3	SW-846 1311/8260B/ Empiircal SOP-198/202	Accuracy / Bias	Statistically derived limits.	Laboratory Control Spike and Surrogate Standards	A
S3	SW-846 1311/8260B/ Empiircal SOP-198/202	Accuracy / Representativeness	Between 2 and 6 degrees C.	Cooler Temperature Indicator	S
S3	SW-846 1311/8260B/ Empiircal SOP-198/202	Data Completeness	95% Overall	Data Completeness Check	S & A
S3	SW-846 1311/8260B/ Empiircal SOP-198/202	Comparability	Values > 5X QL: Field Replicates ≤ 50% RPD	Comparability Check	S & A
S3	SW-846 1311/8260B/ Empiircal SOP-198/202	Sensitivity	Quantiation limits less than project action limits listed in Worksheet 15	Comparability and sensitivity check	S&A

Matrix	Soil
Analytical Group	TCLP SVOC
Concentration Level	Low/Medium

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
S3	SW-846 1311/8270C/ Empirical SOP-198/300/201	Bias / Contamination	No target analytes ≥ QL; with the exception of common field/laboratory contaminants	Method Blank	A
S3	SW-846 1311/8270C/ Empirical SOP-198/300/201	Accuracy / Bias / Precision	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Internal Standards	A
S3	SW-846 1311/8270C/ Empirical SOP-198/300/201	Accuracy / Bias	Statistically derived limits.	Laboratory Control Spike and Surrogate Standards	A
S3	SW-846 1311/8270C/ Empirical SOP-198/300/201	Accuracy / Representativeness	Between 2 and 6 degrees C.	Cooler Temperature Indicator	S
S3	SW-846 1311/8270C/ Empirical SOP-198/300/201	Data Completeness	95% Overall	Data Completeness Check	S & A
S3	SW-846 1311/8270C/ Empirical SOP-198/300/201	Comparability	Values > 5X QL: Field Replicates ≤ 50% RPD	Comparability Check	S & A
S3	SW-846 1311/8270C/ Empirical SOP-198/300/201	Sensitivity	Quantiation limits less than project action limits listed in Worksheet 15	Comparability and sensitivity check	S&A

Matrix	Soil
Analytical Group	TCLP Pesticide
Concentration Level	Low/Medium

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
S3	SW-846 1311/8081A/ Empirical SOP-198/302/211	Bias / Contamination	No target analytes ≥ QL; with the exception of common field/laboratory contaminants	Method Blank	A
S3	SW-846 1311/8081A/ Empirical SOP-198/302/211	Accuracy / Bias	Statistically derived limits.	Laboratory Control Spike and Surrogate Standards	A
S3	SW-846 1311/8081A/ Empirical SOP-198/302/211	Accuracy / Representativeness	Between 2 and 6 degrees C.	Cooler Temperature Indicator	S
S3	SW-846 1311/8081A/ Empirical SOP-198/302/211	Data Completeness	95% Overall	Data Completeness Check	S & A
S3	SW-846 1311/8081A/ Empirical SOP-198/302/211	Comparability	Values > 5X QL: Field Replicates ≤ 50% RPD	Comparability Check	S & A
S3	SW-846 1311/8081A/ Empirical SOP-198/302/211	Sensitivity	Quantiation limits less than project action limits listed in Worksheet 15	Comparability and sensitivity check	S&A

Matrix	Soil
Analytical Group	TCLP Herbicides
Concentration Level	Low/Medium

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
S3	SW-846 1311/8151A/ Empirical SOP-198/304/208	Bias / Contamination	No target analytes ≥ QL	Method Blank	A
S3	SW-846 1311/8151A/ Empirical SOP-198/304/208	Accuracy / Bias	Statistically derived limits.	Laboratory Control Spike and Surrogate Standards	A
S3	SW-846 1311/8151A/ Empirical SOP-198/304/208	Accuracy / Representativeness	Between 2 and 6 degrees C.	Cooler Temperature Indicator	S
S3	SW-846 1311/8151A/ Empirical SOP-198/304/208	Data Completeness	95% Overall	Data Completeness Check	S & A
S3	SW-846 1311/8151A/ Empirical SOP-198/304/208	Comparability	Values > 5X QL: Field Replicates ≤ 50% RPD	Comparability Check	S & A
S3	SW-846 1311/8151A/ Empirical SOP-198/304/208	Sensitivity	Quantiation limits less than project action limits listed in Worksheet 15	Comparability and sensitivity check	S&A

Matrix	Soil
Analytical Group	TCLP Explosives
Concentration Level	Low/Medium

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
S3	SW-846 1311/8330A/ Empirical SOP-198/327	Bias / Contamination	No target analytes ≥ QL	Method Blank	A
S3	SW-846 1311/8330A/ Empirical SOP-198/327	Accuracy / Bias	Statistically derived limits.	Laboratory Control Spike and Surrogate Standards	A
S3	SW-846 1311/8330A/ Empirical SOP-198/327	Accuracy / Representativeness	Between 2 and 6 degrees C.	Cooler Temperature Indicator	S
S3	SW-846 1311/8330A/ Empirical SOP-198/327	Data Completeness	95% Overall	Data Completeness Check	S & A
S3	SW-846 1311/8330A/ Empirical SOP-198/327	Comparability	Values > 5X QL: Field Replicates ≤ 50% RPD	Comparability Check	S & A
S3	SW-846 1311/8330A/ Empirical SOP-198/327	Sensitivity	Quantiation limits less than project action limits listed in Worksheet 15	Comparability and sensitivity check	S&A

Matrix	Soil
Analytical Group	TCLP Metals
Concentration Level	Low/Medium

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
S3	SW-846 1311/6010B/7470A/ Empirical SOP-198/100/105/103	Bias / Contamination	No target analytes ≥ QL	Method Blank	A
S3	SW-846 1311/6010B/7470A/ Empirical SOP-198/100/105/103	Accuracy / Bias	± 20% Recovery of true value	Laboratory Control Spike	A
S3	SW-846 1311/6010B/7470A/ Empirical SOP-198/100/105/103	Accuracy / Representativeness	Between 2 and 6 degrees C.	Cooler Temperature Indicator	S
S3	SW-846 1311/6010B/7470A/ Empirical SOP-198/100/105/103	Data Completeness	95% Overall	Data Completeness Check	S & A
S3	SW-846 1311/6010B/7470A/ Empirical SOP-198/100/105/103	Comparability	Values > 5X QL: Field Replicates ≤ 50% RPD	Comparability Check	S & A
S3	SW-846 1311/6010B/7470A/ Empirical SOP-198/100/105/103	Sensitivity	Quantiation limits less than project action limits listed in Worksheet 15	Comparability and sensitivity check	S&A

Matrix	Soil/Sediment
Analytical Group	TOC
Concentration Level	Low/Medium

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
S3	Lloyd Kahn Method Empirical SOP-221	Bias / Contamination	$\leq$100 mg/kg	Method Blank	A
S3	Lloyd Kahn Method Empirical SOP-221	Accuracy / Bias	± 20% Recovery of true value	Laboratory Control Spike	A
S3	Lloyd Kahn Method Empirical SOP-221	Accuracy / Bias / Precision	± 25% Recovery of true value	Matrix spike/matrix spike duplicate	A
S3	Lloyd Kahn Method Empirical SOP-221	Precision	Values > 5X QL: $\leq$ 50% RPD	Field Duplicates	S & A
S3	Lloyd Kahn Method Empirical SOP-221	Bias / Contamination	No target analytes $\geq$QL	Equipment/Rinsate Blanks	S
S3	Lloyd Kahn Method Empirical SOP-221	Accuracy / Representativeness	Between 2 and 6 degrees C.	Cooler Temperature Indicator	S
S3	Lloyd Kahn Method Empirical SOP-221	Data Completeness	95% Overall	Data Completeness Check	S & A
S3	Lloyd Kahn Method Empirical SOP-221	Comparability	Values > 5X QL: Field Replicates $\leq$ 50% RPD	Comparability Check	S & A
S3	Lloyd Kahn Method Empirical SOP-221	Sensitivity	Quantiation limits less than project action limits listed in Worksheet 15	Comparability and sensitivity check	S&A

[1]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[2]Reference number from QAPP Worksheet #23 (see Section 3.2).
QL – Quantitation limit
RPD – Relative Percent Difference
EICP – Extracted ion current profile
IS – Internal Standard
TCL – Target Compound List
TAL – Target Analyte List
IDL – Instrument Detection Limit
MDL – Method Detection Limit
TCLP – Toxicity Characteristic Leaching Procedure
TOC – Total Organic Carbon
CEC – Cation Exchange Capacity

QAPP_Worksheet_#13

(UFP-QAPP Manual Section 2.7)

Identify all secondary data and information that will be used for the project and their originating sources. Specify how the secondary data will be used and the limitations on their use.

☐ Worksheet Not Applicable (State Reason)

The planned SI is the first formal phase of investigation. Refer to the SAP, Section 3.1 for historical information and the Work Plan Appendix C.

Secondary Data Criteria and Limitations Table

Secondary_Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/ Collection Dates)	How Data Will Be Used	Limitations on Data Use

QAPP_Worksheet_#14

(UFP-QAPP Manual Section 2.8.1)

Provide a brief overview of the listed project activities.

☐ Worksheet Not Applicable (State Reason) **More detailed information is provided in the Work Plan Sections 3 and 5.1 as well as Section 4 of the SAP**

Summary of Project Tasks

Sampling Tasks: Soil, groundwater, surface water, and sediment samples are being collected
Analysis Tasks: Definitve fixed based laboratory data is required. Soil, groundwater, surface water, and sediment samples will be collected and analyzed for explosives, nitroglycerin, pH, total organic carbon, cation exchange capacity, metals, perchlorate, nitrates, and polycyclic aromatic hydrocarbons. Analyses varies by site based on site history. TCLP regulatory list plus explosives for IDW disposal determination purposes.
Quality Control Tasks: QA/QC field samples including duplicates, rinsate blanks, and field blanks. MS/MSD samples. Chain of custody procedures implemented.
Secondary Data: (this should be previous data/data from other investigations) Available data were used "as is" no chemistry data were available.
Data Management Tasks: Assign data qualifiers, verify sample IDs, run statistical calculations, ensure data are transferred to the project database for safekeeping.
Documentation and Records: Keep and accurate and neat field log book. Record all field data in logbook and on associated field forms. Data validation reporting.
Assessment/Audit Tasks: Routine audits of field personnel, field procedures, 100% data validation for laboratory analytical data.
Data Review Tasks: Data validation, database QA, calculation/data input technical review.

QAPP_Worksheet_#15

(UFP-QAPP Manual Section 2.8.1)

Complete this worksheet for each matrix, analytical group, and concentration level. Identify the target analytes/contaminants of concern and project-required action limits. Next, determine the quantitation limits (QLs) that must be met to achieve the project quality objectives. Finally, list the published and achievable detection and quantitation limits for each analyte.

☐ Worksheet Not Applicable (State Reason) **See Attached Table 15**

SAP Worksheet #15 -- Reference Limits and Evaluation Table

Matrix: Soil
Analytical Group: TAL Metals

Analyte	CAS Number	Project Action Limit (PAL) [1] (mg/kg)	PAL Reference	Project Quantitation Limit Goal (PQLG) (mg/kg)	Laboratory [2]	
					Quantitation Limit (QL) (mg/kg)	Method Detection Limit (MDL) (mg/kg)
Aluminum	7429-90-5	7700	Residential	40	40	10
Antimony	7440-36-0	3.1	Residential	1	1	0.25
Arsenic	**7440-38-2**	**0.39**	**Residential**	**0.39**	**1**	**0.15**
Barium	7440-39-3	1500	Residential	10	10	1
Beryllium	7440-41-7	16	Residential	1	1	0.25
Cadmium	7440-43-9	7	Residential	0.25	0.25	0.05
Calcium	7440-70-2	NA	Residential	250	250	50
Chromium	7440-47-3	280	Residential	0.5	0.5	0.1
Cobalt	**7440-48-4**	**2.3**	**Residential**	**2.3**	**2.5**	**0.25**
Copper	7440-50-8	310	Residential	1.25	1.25	0.25
Iron	7439-89-6	5500	Residential	5	5	1.5
Lead	7439-92-1	40	Residential	0.25	0.25	0.1
Mercury	7439-97-6	0.67	Residential	0.67	0.03	0.01
Magnesium	7439-95-4	NA	Residential	250	250	50
Manganese	7439-96-5	180	Residential	1	1	0.25
Nickel	7440-02-0	160	Residential	2	2	0.25
Potassium	7440-09-7	NA	Residential	250	250	50
Selenium	7782-49-2	39	Residential	0.25	0.25	0.15
Silver	7440-22-4	39	Residential	0.5	0.5	0.1
Sodium	7440-23-5	NA	Residential	250	250	50
Thallium	7440-28-0	0.51	Residential	0.5	0.5	0.15
Vanadium	7440-62-2	39	Residential	25	25	0.25
Zinc	7440-66-6	2300	Residential	1	1	0.25

Matrix: Soil
Analytical Group: PAHs

Analyte	CAS Number	Project Action Limit (1) (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory (2)	
					Quantitation Limit (mg/kg)	Method Detection Limit (mg/kg)
1-methylnaphthalene	90-12-0	31	Residential	0.010	0.010	0.003
2-Methylnaphthalene	91-57-6	31	Residential	0.010	0.010	0.003
Acenaphthene	83-32-9	340	Residential	0.010	0.010	0.003
Acenaphthylene	208-96-8	340	Residential	0.010	0.010	0.003
Anthracene	120-12-7	1700	Residential	0.010	0.010	0.003
Benzo(a)anthracene	56-55-3	0.15	Residential	0.010	0.010	0.003
Benzo(a)pyrene	50-32-8	0.015	Residential	0.010	0.010	0.003
Benzo(b)fluoranthene	205-99-2	0.15	Residential	0.010	0.010	0.003
Benzo(g,h,i)perylene	191-24-2	170	Residential	0.010	0.010	0.003
Benzo(k)fluoranthene	207-08-9	1.5	Residential	0.010	0.010	0.003
Chrysene	218-01-9	15	Residential	0.010	0.010	0.003
Dibenzo(a,h)anthracene	**53-70-3**	**0.0015**	**Residential**	**0.0015**	**0.010**	**0.003**
Fluoranthene	206-44-0	230	Residential	0.010	0.010	0.003
Fluorene	86-73-7	230	Residential	0.010	0.010	0.003
Indeno(1,2,3-cd)pyrene	193-39-5	0.15	Residential	0.010	0.010	0.003
Naphthalene	91-20-3	3.9	Residential	0.010	0.010	0.003
Phenanthrene	85-01-8	170	Residential	0.010	0.010	0.003
Pyrene	129-00-0	170	Residential	0.010	0.010	0.003

Matrix: Soil
Analytical Group: Explosives

Analyte	CAS Number	Project Action Limit [1] (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory [2]	
					Quantitation Limit (mg/kg)	Method Detection Limit (mg/kg)
2,4-Dinitrotoluene	121-14-2	0.71	Residential	0.50	0.50	0.10
2,6-Dinitrotoluene	606-20-2	0.71	Residential	0.50	0.50	0.10
2,4,6-Trinitrotoluene	118-96-7	3.6	Residential	0.50	0.50	0.10
2-Amino-4,6-Dinitrotoluene	35572-78-2	15	Residential	0.50	0.50	0.10
2-Nitrotoluene	**88-72-2**	**0.29**	**Residential**	**0.29**	**0.50**	**0.10**
3-Nitrotoluene	99-08-1	120	Residential	0.50	0.50	0.10
4-Amino-2,6-Dinitrotoluene	19406-51-0	15	Residential	0.50	0.50	0.10
4-Nitrotoluene	99-99-0	24	Residential	0.50	0.50	0.10
Nitroglycerin	**55-63-0**	**0.61**	**Residential**	**0.61**	**5.0**	**1.0**

Matrix: Soil
Analytical Group: TOC

Analyte	CAS Number	Project Action Limit [1] (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory [2]	
					Quantitation Limit (mg/kg)	Method Detection Limit (mg/kg)
TOC	NA	NA	NA	3.0	3.0	1.0

PAL References

Residential – Oak Ridge National Laboratory (ORNL) Regional direct contact residential soil screening levels (September 2008)

1 Project Action Limits (PALs) are the ORNL Regional Screening Level table (September 12, 2008). Tables summarizing all reference limits are provided in Appendic C-2.

2 The QLs and MDLs from Empirical Laboratory are presented. These are subject to change and actual limits will be evaluated during the data usability assessment to ensure that the detection and quantitation limits actually achieved are satisfactory to support the data evaluations.

SAP Worksheet #15 -- Reference Limits and Evaluation Table

Matrix: Sediment
Analytical Group: TAL Metals

Analyte	CAS Number	Project Action Limit (PAL) (3,4) (mg/kg)	PAL Reference	Project Quantitation Limit Goal (PQLG) (mg/kg)	Laboratory (2)	
					Quantitation Limit (QL) (mg/kg)	Method Detection Limit (MDL) (mg/kg)
Aluminum	7429-90-5	26000	FACILITY - EA 2006	40	40	10
Antimony	**7440-36-0**	**0.16**	FACILITY - EA 2006	**0.16**	**1**	**0.25**
Arsenic	7440-38-2	9.8	FACILITY - EA 2006	1	1	0.15
Barium	**7440-39-3**	**0.7**	FACILITY - EA 2006	**0.7**	**10**	**1**
Beryllium	**7440-41-7**	**0.55**	FACILITY - EA 2006	**0.55**	**1**	**0.25**
Cadmium	7440-43-9	0.99	FACILITY - EA 2006	0.25	0.25	0.05
Calcium	7440-70-2	NA	FACILITY - EA 2006	250	250	50
Chromium	7440-47-3	43.4	FACILITY - EA 2006	0.5	0.5	0.1
Cobalt	7440-48-4	10	FACILITY - EA 2006	2.5	2.5	0.25
Copper	7440-50-8	31.6	FACILITY - EA 2006	1.25	1.25	0.25
Iron	7439-89-6	10000	FACILITY - EA 2006	5	5	1.5
Lead	7439-92-1	35.8	FACILITY - EA 2006	0.25	0.25	0.1
Mercury	7439-97-6	0.18	FACILITY - EA 2006	0.03	0.03	0.01
Magnesium	7439-95-4	NA	FACILITY - EA 2006	250	250	50
Manganese	7439-96-5	630	FACILITY - EA 2006	1	1	0.25
Nickel	7440-02-0	22.7	FACILITY - EA 2006	2	2	0.25
Potassium	7440-09-7	NA	FACILITY - EA 2006	250	250	50
Selenium	7782-49-2	0.29	FACILITY - EA 2006	0.25	0.25	0.15
Silver	**7440-22-4**	**0.5**	FACILITY - EA 2006	**0.5**	**0.5**	**0.1**
Sodium	7440-23-5	NA	FACILITY - EA 2006	250	250	50
Thallium	7440-28-0	NA	FACILITY - EA 2006	0.5	0.5	0.15
Vanadium	7440-62-2	43	FACILITY - EA 2006	25	25	0.25
Zinc	7440-66-6	121	FACILITY - EA 2006	1	1	0.25

Matrix: Sediment
Analytical Group: PAHs

Analyte	CAS Number	Project Action Limit (3,4) (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory (2)	
					Quantitation Limit (mg/kg)	Method Detection Limit (mg/kg)
1-methylnaphthalene	90-12-0	20.2	NOAA	0.010	0.010	0.003
2-Methylnaphthalene	91-57-6	20.2	NOAA	0.010	0.010	0.003
Acenaphthene	83-32-9	290	NOAA	0.010	0.010	0.003
Acenaphthylene	208-96-8	160	NOAA	0.010	0.010	0.003
Anthracene	120-12-7	57.2	TEC	0.010	0.010	0.003
Benzo(a)anthracene	56-55-3	108	TEC	0.010	0.010	0.003
Benzo(a)pyrene	50-32-8	150	TEC	0.010	0.010	0.003
Benzo(b)fluoranthene	205-99-2	1800	NOAA	0.010	0.010	0.003
Benzo(g,h,i)perylene	191-24-2	170	OMOE	0.010	0.010	0.003
Benzo(k)fluoranthene	207-08-9	240	OMOE	0.010	0.010	0.003
Chrysene	218-01-9	166	TEC	0.010	0.010	0.003
Dibenzo(a,h)anthracene	53-70-3	33	TEC	0.010	0.010	0.003
Fluoranthene	206-44-0	423	TEC	0.010	0.010	0.003
Fluorene	86-73-7	77.4	TEC	0.010	0.010	0.003
Indeno(1,2,3-cd)pyrene	193-39-5	200	OMOE	0.010	0.010	0.003
Naphthalene	91-20-3	176	TEC	0.010	0.010	0.003
Phenanthrene	85-01-8	204	TEC	0.010	0.010	0.003
Pyrene	129-00-0	195	TEC	0.010	0.010	0.003

Matrix: Sediment
Analytical Group: TOC

Analyte	CAS Number	Project Action Limit (1) (mg/kg)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/kg)	Laboratory (2)	
					Quantitation Limit (mg/kg)	Method Detection Limit (mg/kg)
TOC	NA	NA	NA	3	3.0	1.0

(1) Where available, sediment project action limits reflect facility-specific values previously developed for other sites at NAS Brunswick (EA, January 31, 2006).
(2) The QLs and MDLs from Empirical laboratories are presented. These are subject to change and actual limits will be evaluated during the data usability assessment to ensure that the detection and quantitation limits actually achieved are satisfactory to support the data evaluations.
(3) Where facility-specific project action limits are not available for sediment, a variety of other sources were used and is noted as such.
- NOAA – National Oceanographic and Atmospheric Administration sediment benchmarks lowest observed effects level (Buchman, 1999) (ecological risk based value).
- OMOE – Ontario Ministry of the environment Lowest Effects Level (Persaud, etal., 1993) (ecological risk based values).
- TEC – Threshold Effects Concentrations (MacDonald, et al., 2000) (ecological risk based values).

Bolded values indicate that the project action limit is below the project quantitation limit.
Bolded and shaded values indicate that the project action limit is below both the project quantitation limit and method detection limit.

NA - Not available

SAP Worksheet #15 -- Reference Limits and Evaluation Table

Matrix: Groundwater
Analytical Group: TAL Metals

Analyte	CAS Number	Project Action Limit (1) (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory (2)	
					Quantitation Limit (µg/L)	Method Detection Limit (µg/L)
Aluminum	7429-90-5	1430	MEG	200	200	50
Antimony	**7440-36-0**	**1.5**	**Tap Water SL**	**1.5**	**4**	**1.3**
Arsenic	**7440-38-2**	**0.045**	**Tap Water SL**	**0.045**	**1.3**	**0.75**
Barium	7440-39-3	730	Tap Water SL	200	200	5
Beryllium	7440-41-7	7.3	Tap Water SL	5	5	1
Cadmium	**7440-43-9**	**1.8**	**Tap Water SL**	**1.8**	**5**	**1**
Calcium	7440-70-2	NA	Tap Water SL	1000	1000	200
Chromium	7440-47-3	35	MEG	10	10	2
Cobalt	**7440-48-4**	**1.1**	**Tap Water SL**	**1.1**	**15**	**5**
Copper	7440-50-8	150	Tap Water SL	25	25	5
Iron	7439-89-6	2600	Tap Water SL	100	100	30
Lead	7439-92-1	10	MEG	3	3	1.5
Mercury	**7439-97-6**	**0.063**	**Tap Water SL**	**0.063**	**0.2**	**0.08**
Magnesium	7439-95-4	NA	Tap Water SL	5000	5000	1000
Manganese	7439-96-5	88	Tap Water SL	15	15	5
Nickel	7440-02-0	140	MEG	40	40	5
Potassium	7440-09-7	NA	Tap Water SL	1000	1000	500
Selenium	7782-49-2	35	MEG	5	5	3
Silver	7440-22-4	35	MEG	10	10	1
Sodium	7440-23-5	NA	MEG	5000	5000	1000

Analyte	CAS Number	Project Action Limit (1) (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory (2)	
					Quantitation Limit (µg/L)	Method Detection Limit (µg/L)
Thallium	**7440-28-0**	**0.5**	**MEG**	**0.5**	**2**	**0.75**
Vanadium	7440-62-2	18	Tap Water SL	15	15	5
Zinc	7440-66-6	1100	MEG	20	20	5

Matrix: Groundwater
Analytical Group: PAHs

Analyte	CAS Number	Project Action Limit (1) (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ugL)	Laboratory (2)	
					Quantitation Limit (ug/L)	Method Detection Limit (ug/L)
1-Methylnaphthalene		2.3	Tap Water SL	0.05	0.05	0.008
2-Methylnaphthalene	91-57-6	15	Tap Water SL	0.05	0.05	0.006
Acenaphthene	83-32-9	220	Tap Water SL	0.05	0.05	0.007
Acenaphthylene	208-96-8	220	Tap Water SL	0.05	0.05	0.005
Anthracene	120-12-7	1100	Tap Water SL	0.05	0.05	0.001
Benzo(a)anthracene	**56-55-3**	**0.029**	**Tap Water SL**	**0.029**	**0.05**	**0.013**
Benzo(a)pyrene	**50-32-8**	**0.0029**	**Tap Water SL**	**0.0029**	**0.05**	**0.007**
Benzo(b)fluoranthene	**205-99-2**	**0.029**	**Tap Water SL**	**0.029**	**0.05**	**0.012**
Benzo(g,h,i)perylene	191-24-2	110	Tap Water SL	0.05	0.05	0.007
Benzo(k)fluoranthene	207-08-9	0.29	Tap Water SL	0.05	0.05	0.01
Chrysene	218-01-9	2.9	Tap Water SL	0.05	0.05	0.011
Dibenzo(a,h)anthracene	**53-70-3**	**0.0029**	**Tap Water SL**	**0.0029**	**0.05**	**0.006**
Fluoranthene	206-44-0	150	Tap Water SL	0.05	0.05	0.013
Fluorene	86-73-7	150	Tap Water SL	0.05	0.05	0.007
Indeno(1,2,3-cd)pyrene	**193-39-5**	**0.029**	**Tap Water SL**	**0.029**	**0.05**	**0.004**
Naphthalene	91-20-3	0.14	Tap Water SL	0.05	0.05	0.013
Phenanthrene	85-01-8	110	Tap Water SL	0.05	0.05	0.008
Pyrene	129-00-0	110	Tap Water SL	0.05	0.05	0.013

Matrix: Groundwater
Analytical Group: Explosives

Analyte	CAS Number	Project Action Limit (1) (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Laboratory (2)	
					Quantitation Limit (ug/L)	Method Detection Limit (ug/L)
2,4-Dinitrotoluene	**121-14-2**	**0.5**	**MEG**	**0.5**	**0.5**	**0.1**
2,6-Dinitrotoluene	**606-20-2**	**0.5**	**MEG**	**0.5**	**0.5**	**0.1**
2,4,6-Trinitrotoluene	118-96-7	1.8	Tap Water SL	0.5	0.5	0.1
2-Amino-4,6-Dinitrotoluene	35572-78-2	7.3	Tap Water SL	0.5	0.5	0.1
2-Nitrotoluene	**88-72-2**	**0.31**	**Tap Water SL**	**0.31**	**0.5**	**0.1**
3-Nitrotoluene	99-08-1	73	Tap Water SL	0.5	0.5	0.1
4-Amino-2,6-Dinitrotoluene	1946-51-0	7.3	Tap Water SL	0.5	0.5	0.1
4-Nitrotoluene	99-99-0	4.2	Tap Water SL	0.5	0.5	0.1
Nitroglycerin	**55-63-0**	**0.37**	**Tap Water SL**	**0.37**	**4.0**	**1.0**

Matrix: Groundwater
Analytical Group: Perchlorate

Analyte	CAS Number	Project Action Limit (1) (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Laboratory (2)	
					Quantitation Limit (ug/L)	Method Detection Limit (ug/L)
Perchlorate	14797-73-0	1	MEG	0.50	0.50	0.085

Matrix: Groundwater
Analytical Group: Nitrate and Nitrite

Analyte	CAS Number	Project Action Limit (1) (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ug/L)	Laboratory (2)	
					Quantitation Limit (ug/L)	Method Detection Limit (ug/L)
Nitrate	14797-55-8	10000	MEG	0.1	0.1	0.01
Nitrite	14797-65-0	1000	MEG	0.05	0.05	0.01

Matrix: Leachate (all)
Analytical Group: Toxicity Characteristic Leachate Procedure (TCLP) Regulatory List

Analyte	CAS Number	Project Action Limit (3) (mg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/L)	Laboratory (2)	
					Quantitation Limit (mg/L)	Method Detection Limit (mg/L)
Benzene	71-43-2	0.5	40 CFR 260.11	0.020	0.020	0.005
Carbon tetrachloride	56-23-5	0.5	40 CFR 260.11	0.020	0.020	0.005
Chlorobenzene	108-90-7	100	40 CFR 260.11	0.020	0.020	0.005
Chloroform	67-66-3	6	40 CFR 260.11	0.020	0.020	0.005
1,2-Dichloroethane	107-06-2	0.5	40 CFR 260.11	0.020	0.020	0.005
1,1-Dichloroethene	75-35-4	0.7	40 CFR 260.11	0.020	0.020	0.005
2-Butanone	78-93-3	200	40 CFR 260.11	0.100	0.100	0.04
Tetrachloroethene	127-18-4	0.7	40 CFR 260.11	0.020	0.020	0.005
Trichloroethene	79-01-6	0.5	40 CFR 260.11	0.020	0.020	0.005
Vinyl chloride	75-01-4	0.2	40 CFR 260.11	0.040	0.040	0.02
o-Cresol	95-48-7	200	40 CFR 260.11	0.050	0.050	0.02
m-Cresol	95-48-7	200	40 CFR 260.11	0.050	0.050	0.02
p-Cresol	106-44-5	200	40 CFR 260.11	0.050	0.050	0.02
1,4-Dichlorobenzene	106-46-7	7.5	40 CFR 260.11	0.050	0.050	0.02
2,4-Dinitrotoluene	121-14-2	0.13	40 CFR 260.11	0.050	0.050	0.02
Hexachlorobenzene	118-74-1	0.13	40 CFR 260.11	0.050	0.050	0.02
Hexachloro-1,3-butadiene	87-68-3	0.5	40 CFR 260.11	0.050	0.050	0.02
Hexachloroethane	67-72-1	3	40 CFR 260.11	0.050	0.050	0.02
Nitrobenzene	98-95-3	2	40 CFR 260.11	0.050	0.050	0.02
Pentachlorophenol	87-86-5	100	40 CFR 260.11	0.050	0.050	0.02
Pyridine	110-86-1	5	40 CFR 260.11	0.250	0.250	0.1
2,4,5-Trichlorophenol	95-95-4	400	40 CFR 260.11	0.1	0.1	0.05
2,4,6-Trichlorophenol	88-06-2	2	40 CFR 260.11	0.1	0.1	0.05
Chlordane	57-74-9	0.03	40 CFR 260.11	0.0025	0.0025	0.0010
Endrin	72-20-8	0.02	40 CFR 260.11	0.0025	0.0025	0.0010
Heptachlor	76-44-8	0.008	40 CFR 260.11	0.00025	0.00025	0.0001
Heptachlor epoxide	1024-57-3	0.008	40 CFR 260.11	0.00025	0.00025	0.0001
Lindane	58-89-9	0.4	40 CFR 260.11	0.00025	0.00025	0.0001
Methoxychlor	72-43-5	10	40 CFR 260.11	0.0025	0.0025	0.001
Toxaphene	8001-35-2	0.5	40 CFR 260.11	0.010	0.010	0.005
2,4-D	94-75-7	10	40 CFR 260.11	0.015	0.015	0.005
2,4,5-TP (silvex)	93-72-1	1	40 CFR 260.11	0.015	0.015	0.005
Arsenic	7440-38-2	5	40 CFR 260.11	0.10	0.10	0.05
Barium	7440-39-3	100	40 CFR 260.11	0.10	0.10	0.05
Cadmium	7440-43-9	1	40 CFR 260.11	0.10	0.10	0.05
Chromium	7440-47-3	5	40 CFR 260.11	0.10	0.10	0.05

Analyte	CAS Number	Project Action Limit (3) (mg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/L)	Laboratory (2)	
					Quantitation Limit (mg/L)	Method Detection Limit (mg/L)
Lead	7439-92-1	5	40 CFR 260.11	0.10	0.10	0.05
Mercury	7439-97-6	0.2	40 CFR 260.11	0.002	0.002	0.001
Selenium	7782-49-2	1	40 CFR 260.11	0.10	0.10	0.05
Silver	7440-22-4	5	40 CFR 260.11	0.10	0.10	0.05

PAL References

Residential - Oak Ridge National Laboratory Regional direct contact residential soil screening levels (September 2008)

Tap Water SL - ORNL Screening Levels for tap water (September 2008)

MEG - Maine CDC Maximum Exposure Guidelines (MEG) for Drinking Water (December 5, 2008)

(4) Project Action Limits (PALs) are the ORNL Regional Screening Level table (September 12, 2008). Tables summarizing all reference limits are provided in Appendic C-2. For carcinogens in both soil and groundwater, the PRG reference value is provided and for noncarcinogens the PRG reference value is divided by ten. For groundwater, in some cases, the Maine CDC Maximum Exposure Guidelines (MEG) for Drinking Water (December 5, 2008) was lower than the ORNL Tap water value; in these instances the MEG is presented as the project action limit and is noted as such.

(5) The QLs and MDLs from Empirical laboratories are presented. These are subject to change and actual limits will be evaluated during the data usability assessment to ensure that the detection and quantitation limits actually achieved are satisfactory to support the data evaluations.

(6) The PAL is the TCLP regulatory level.

Bolded values indicate that the project action limit is below the project quantitation limit.
Bolded and shaded values indicate that the project action limit is below both the project quantitation limit and method detection limit.

NA - Not available

SAP Worksheet #15 -- Reference Limits and Evaluation Table

Matrix: Surface Water
Analytical Group: TAL Metals

Analyte	CAS Number	Project Action Limit (1) (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory (2)	
					Quantitation Limit (µg/kg)	Method Detection Limit (µg/L)
Aluminum	**7429-90-5**	**87**	**FACILITY - EA 2006**	**87**	**200**	**50**
Antimony	7440-36-0	5.5	SWQC	4	4	1.3
Arsenic	7440-38-2	150	FACILITY - EA 2006	1.3	1.3	0.75
Barium	**7440-39-3**	**3.9**	**FACILITY - EA 2006**	**3.9**	**200**	**5**
Beryllium	**7440-41-7**	**0.66**	**FACILITY - EA 2006**	**0.66**	**5**	**1**
Cadmium	**7440-43-9**	**0.08**	**FACILITY - EA 2006**	**0.08**	**5**	**1**
Calcium	7440-70-2	NA	FACILITY - EA 2006	1000	1000	200
Chromium	7440-47-3	11	FACILITY - EA 2006	10	10	2
Cobalt	7440-48-4	23	FACILITY - EA 2006	15	15	5
Copper	**7440-50-8**	**2.36**	**FACILITY - EA 2006**	**2.36**	**25**	**5**
Iron	7439-89-6	1000	FACILITY - EA 2006	100	100	30
Lead	**7439-92-1**	**0.41**	**FACILITY - EA 2006**	**0.41**	**3**	**1.5**
Mercury	7439-97-6	0.91	FACILITY - EA 2006	0.2	0.2	0.08
Magnesium	7439-95-4	NA	FACILITY - EA 2006	5000	5000	1000
Manganese	7439-96-5	120	FACILITY - EA 2006	15	15	5
Nickel	**7440-02-0**	**13.4**	**FACILITY - EA 2006**	**13.4**	**40**	**5**
Potassium	7440-09-7	NA	FACILITY - EA 2006	1000	1000	500
Selenium	7782-49-2	5.0	FACILITY - EA 2006	5	5	3
Silver	**7440-22-4**	**0.23**	**FACILITY - EA 2006**	**0.23**	**10**	**1**
Sodium	7440-23-5	NA	FACILITY - EA 2006	5000	5000	1000
Thallium	7440-28-0	12	FACILITY - EA 2006	2	2	0.75
Vanadium	7440-62-2	20	FACILITY - EA 2006	15	15	5
Zinc	7440-66-6	30.6	FACILITY - EA 2006	20	20	5

Matrix: Surface Water
Analytical Group: PAHs

Analyte	CAS Number	Project Action Limit (3) (ug/L)	Project Action Limit Reference	Project Quantitation Limit Goal (ugL)	Laboratory (2)	
					Quantitation Limit (ug/L)	Method Detection Limit (ug/L)
1-methylnaphthalene	90-12-0	72.16	FCV	0.05	0.05	0.008
2-Methylnaphthalene	91-57-6	72.16	FCV	0.05	0.05	0.006
Acenaphthene	83-32-9	55.85	FCV	0.05	0.05	0.007
Acenaphthylene	208-96-8	306.9	FCV	0.05	0.05	0.005
Anthracene	120-12-7	20.73	FCV	0.05	0.05	0.001
Benzo(a)anthracene	**56-55-3**	**0.003**	**SWQC**	**0.003**	**0.05**	**0.013**
Benzo(a)pyrene	**50-32-8**	**0.003**	**SWQC**	**0.003**	**0.05**	**0.007**
Benzo(b)fluoranthene	**205-99-2**	**0.003**	**SWQC**	**0.003**	**0.05**	**0.012**
Benzo(g,h,i)perylene	**191-24-2**	**0.003**	**SWQC**	**0.003**	**0.05**	**0.007**
Benzo(k)fluoranthene	**207-08-9**	**0.003**	**SWQC**	**0.003**	**0.05**	**0.01**
Chrysene	**218-01-9**	**0.003**	**SWQC**	**0.003**	**0.05**	**0.011**
Dibenzo(a,h)anthracene	**53-70-3**	**0.003**	**SWQC**	**0.003**	**0.05**	**0.006**
Fluoranthene	206-44-0	7.109	FCV	0.05	0.05	0.013
Fluorene	86-73-7	39.3	FCV	0.05	0.05	0.007
Indeno(1,2,3-cd)pyrene	**193-39-5**	**0.003**	**SWQC**	**0.003**	**0.05**	**0.004**
Naphthalene	91-20-3	193.5	FCV	0.05	0.05	0.013
Phenanthrene	85-01-8	19.13	FCV	0.05	0.05	0.008
Pyrene	129-00-0	10.11	FCV	0.05	0.05	0.013

(7) Where available, surface water project action limits reflect facility-specific values previously developed for other sites at NAS Brunswick (EA, January 31, 2006).
(8) The QLs and MDLs from Empirical laboratories are presented. These are subject to change and actual limits will be evaluated during the data usability assessment to ensure that the detection and quantitation limits actually achieved are satisfactory to support the data evaluations.
(9) Where facility-specific project action limits are not available for surface water, a variety of other sources were used and is noted as such. FCV – Final chronic values (USEPA, 2003) (ecological risk based value), SWQC – State water quality criteria based on consumption for protection of human health.

Bolded values indicate that the project action limit is below the project quantitation limit.
Bolded and shaded values indicate that the project action limit is below both the project quantitation limit and method detection limit.

NA - Not available

(10) The project action limit is the Region IX Preliminary Remediation Goal (PRG), USEPA October 2004 with December 20, 2006 update. The target project action limit for soil is the PRG for residential soil and for groundwater is the PRG for tap water. For carcinogens in both soil and groundwater, the PRG reference value is provided and for noncarcinogens the PRG reference value is divided by ten.
(11) For groundwater, in some cases, the Maine CDC Maximum Exposure Guidelines (MEG) for Drinking Water (September 7, 2007) was lower than the Region IX PRG; in these instances the MEG is presented as the project action limit and is noted as such.
(12) Where available, surface water and sediment project action limits reflect facility-specific values previously developed for other sites at NAS Brunswick (EA, January 31, 2006).
(13) Where facility-specific project action limits are not available for surface water and sediment, a variety of other sources were used and is noted as such.
- EcoTox – Ecotox Thresholds (USEPA, 1996) (ecological based values)
- FCV – Final chronic values (USEPA, 2003) (ecological risk based value)
- NOAA – National Oceanographic and Atmospheric Administration sediment benchmarks lowest observed effects level (Buchman, 1999) (ecological risk based value)
- NRWQC – National recommended Water Quality Criteria, continuous concentration (CCC) for fresh water
- OMOE – Ontario Ministry of the environment Lowest Effects Level (Persaud, etal., 1993) (ecological risk based values)
- SCV – Secondary chronic value (Suter and Tsao, 1996) (ecological risk based level)
- SWQC – State (Maine) Water Quality Criteria for Human Health consumption of water and organisms
- TEC – Threshold Effects Concentrations (MacDonald, etal., 2000) (ecological risk based values)

(14) The project action limit is the TCLP Regulatory level.

Bolded values indicate that the project action limit is below the project quantitation limit.

NA - Not available

QAPP_Worksheet_#16

(UFP-QAPP Manual Section 2.8.2)

List all project activities as well as the QA assessments that will be performed during the course of the project. Include the anticipated start and completion dates.

☐ Worksheet Not Applicable (State Reason) **See Work Plan Table 5-1**

Project Schedule Timeline Table

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		

QAPP_Worksheet_#17

(UFP-QAPP Section 3.1.1)

Describe the project sampling approach. Provide the rationale for selecting sample locations and matrices for each analytical group and concentration level.

☐ Worksheet Not Applicable (State Reason)

Sampling Design and Rationale

Describe and provide a rationale for choosing the sampling approach (e.g., grid system, biased statistical approach): **See Section 4 of the SAP.**
Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations) [May refer to map or Worksheet #18 for details]: **See the location maps in Section 2 of the Work Plan. Also see Table 3-1 of the SAP. For the MC sites where XRF sampling is planned (NASB Skeet Range, Topsham Skeet Range, and Machine Gun Boresight Range) firm sample locations will not be determined until after the XRF sampling is completed. The regulatory agencies will be involved in determining sample locations.**

QAPP_Worksheet_#18

(UFP-QAPP Manual Section 3.1.1)

List all site locations that will be sampled and include sample/ID number, if available. (Provide a range of sampling locations or ID numbers if a site has a large number.) Specify matrix and, if applicable, depth at which samples will be taken. Only a short reference for the sampling location rationale is necessary for the table. The text of the QAPP should clearly identify the detailed rationale associated with each reference. Complete all required information, using additional worksheets if necessary.

☐ Worksheet Not Applicable (State Reason)

See the sample location maps in Section 2 of the Work Plan and Table 3-1 of the SAP. Sampling locations have not yet been firmly determined. For the MC sites where XRF sampling is planned (NASB Skeet Range, Topsham Skeet Range, and Machine Gun Boresight Range) firm sample locations will not be determined until after the XRF sampling is completed. The regulatory agencies will be involved in determining sample locations.
See Sections 4 and 5 of the SAP.

Sampling Locations and Methods/SOP Requirements Table

Sampling Location/ID Number	Matrix	Depth ( )	Analytical Group	Concentration Level	Number of Samples (identify field duplicates	Sampling SOP Reference[1]	Rationale for Sampling Location

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet_#21).

QAPP_Worksheet_#19

(UFP-QAPP Manual Section 3.1.1)

For each matrix, analytical group, and concentration level, list the analytical and preparation method/SOP and associated sample volume, container specifications, preservation requirements, and maximum holding time.

☐ Worksheet Not Applicable (State Reason)

Analytical SOP Requirements Table (Fixed-Base Laboratory)

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference[1]	Containers (number, size, and type)	Sample volume[2] (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[3] (preparation / analysis)
Water	PAHs	SW-846 3510C/8270C SIM Empirical SOP-300/231	Two 1-liter glass amber bottles	1000 milliliters	Cool to 4° C	7 days until extraction/40 days to analysis
Soil	PAHs	SW-846 3541/8270C SIM Empirical SOP-329/231	4 ounce glass	15 grams	Cool to 4° C	14 days until extraction/40 days to analysis
Water	Metals	SW-846 3005A/6010B/7470A, Empirical SOP-100/103/105	500 milliliter plastic	50 milliliters/30 milliliters mercury	Nitric acid to a pH<2; Cool to 4 ° C	180 days to analysis, 28 days mercury
Soil	Metals	SW-846 3050B/6010B/7471A, Empirical SOP-100/104/105	4 ounce glass	1-2 grams/ 0.3 grams mercury	Cool to 4 ° C	180 days to analysis, 28 days mercury
Solid Investigationally Derived Waste (IDW)	Toxicity Characteristic Leaching Procedure (TCLP) Organics	SW-846 1311/3510C/8260B/8270C /8081A/ 8151B/ Empirical SOP-198/300/302/304/202 /201/211/208	4 ounce glass	100 grams	Cool to 4 ° C	14 days to TCLP extraction/7 days to extraction for Semis ,40 days analysis/ 14 days for volatiles to analysis.

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference[1]	Containers (number, size, and type)	Sample volume[2] (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[3] (preparation / analysis)
Solid IDW	TCLP Metals	SW-846 1311/3005A/6010B/ 7470A Empirical SOP-198/100/105/ 103	4 ounce glass	100 grams	Cool to 4 ° C	14 days to TCLP extraction/180 days to analysis, 28 days Hg
Water	Dinitrotoluenes and Nitroglycerin	SW-846 8330A/Empirical SOP-327	2- One Liter Amber Glass	1000 milliliters	Sulfuric acid to a pH< 2; Cool to 4 ° C	7 days to extraction, 40 days to analysis
Soil	Dinitrotoluenes and Nitroglycerin	SW-846 8330A/Empirical SOP-327	4 ounce glass jar	2 grams	Cool to 4 ° C	14 days to extraction, 40 days to analysis
Water	Perchlorate	SW-846 6850 CAS SOP-HPLC-6850	125 milliliters pre-cleaned sterile, plastic containers	10 milliliters	Shipped via FedEx within 12 hours of sample collection with /3 volume head space to remove and/or minimize microbial and anaerobic bacteria. Laboratory to filter immediately upon receipt. 4 + 2 ^{o}C	28 days from sampling to analysis
Water	Nitrate and Nitrite	EPA 300 Empirical SOP-145	250 milliliters pre-cleaned sterile, plastic containers	5 milliliters	Cool to 4 ° C	48 hours to analysis
Soil	pH	SW-846 9045C Empirical SOP-187	4 oz glass	4 oz.	Cool to 4 ° C	Analyze as soon as possible
Soil	Total Organic Carbon(TOC)	Lloyd Kahn Empirical SOP-221	4 ounce glass	5 grams	Cool to 4 ° C	28 days to analysis

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference[1]	Containers (number, size, and type)	Sample volume[2] (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[3] (preparation / analysis)
Soil	Cation Exchange Capacity (CEC)	SW-845 9081 Empirical SOP	4 ounce glass	4-6 grams	Cool to 4 ° C	6 months to analysis

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).
[2] Provide the minimum sample volume or mass requirement if it differs from the container volume.
[3] Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.

HCl - Hydrochloric acid
C - Celcius
TCL – Target Compound List
TAL – Target Analyte List
TCLP – Toxicity Characteristic Leaching Procedure

Analytical SOP Requirements Table (Field)

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference [1]	Sample Volume	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)
Soil	Lead	Low	XRF SOP (see SOP S15)	1 oz.	Zip-lock bag	None	180 days to analysis

QAPP_Worksheet_#20

(UFP-QAPP Manual Section 3.1.1)

Summarize by matrix, analytical group, and concentration level the number of field QC samples that will be collected and sent to the laboratory.

☐ Worksheet Not Applicable (State Reason)

Field Quality Control Sample Summary Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations	No. of Field Duplicate Pairs	No. of MS	No. of Field Blanks	No. of Equip. Blanks	No. of PT Samples	Total No. of Samples to Lab*
Soil	Lead	Low	XRF	270	27	None	None	None	None	0
Surface Soil	TAL Metals	low	SW-846 3005A/6010B/ 7471A Empirical SOP-100/105/104	48	5	5	0	0	0	58
Surface Soil	Explosives	low	SW-846 8330A/Empirical SOP-327	8	2	2	0	0	0	12
Surface Soil	Nitroglycerin	low	SW-846 8332/Empirical SOP-327	21	3	3	0	0	0	27
Surface Soil	PAHs	low	SW-846 3541/8270C/ Empirical SOP-329/231	36	4	4	0	0	0	44
Surface Soil	pH	Low		8	0	0	0	0	0	48
Surface Soil	TOC	Low		8	0	0	0	0	0	8
Surface Soil	CEC	Low		8	0	0	0	0	0	8
Subsurface Soil	TAL Metals	low	SW-846 3005A/6010B/ 7471A Empirical SOP-100/105/104	25	3	3	0	0	0	31
Subsurface Soil	Explosives	low	SW-846 8330A/Empirical SOP-327	3	1	1	0	0	0	5

Field Quality Control Sample Summary Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations	No. of Field Duplicate Pairs	No. of MS	No. of Field Blanks	No. of Equip. Blanks	No. of PT Samples	Total No. of Samples to Lab*
Subsurface Soil	Nitroglycerin	low	SW-846 8330A/Empirical SOP-327	15	2	2	0	0	0	19
Subsurface Soil	PAHs	low	SW-846 3541/8270C/ Empirical SOP-329/231	13	2	2	0	0	0	17
Groundwater /Aqueous*	TAL Metals	low	SW-846 3005A/6010B/ 7470A Empirical SOP-100/105/103	17	3	3	1	9	0	33
Groundwater /Aqueous*	Nitroglycerin	low	SW-846 8330A/Empirical SOP-327	7	2	2	1	7	0	19
Groundwater /Aqueous*	PAHs	low	SW-846 3510C/8270C/ Empirical SOP-300/231	12	2	2	1	6	0	23
Groundwater /Aqueous*	Explosives	low	SW-846 8330A	2	1	1	1	4	0	9
Groundwater /Aqueous*	Perchlorate	low	SW-846 6850	5	1	1	1	1	0	9
Groundwater /Aqueous*	Nitrates	Low		12	2	2	1	2	0	19
Surface water	TAL Metals	low	SW-846 3005A/6010B/ 7470A Empirical SOP-100/105/103	5	2	2	0	2	0	11
Surface water	PAHs	low	SW-846 3510C/8270C/ Empirical SOP-300/231	3	1	1	0	1	0	6
Sediment	TAL Metals	low	SW-846 3005A/6010B/ 7471A Empirical SOP-100/105/104	7	2	2	0	2	0	13
Sediment	PAHs	low	SW-846 3541/8270C/ Empirical SOP-329/231	3	1	1	0	1	0	6

Field Quality Control Sample Summary Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations	No. of Field Duplicate Pairs	No. of MS	No. of Field Blanks	No. of Equip. Blanks	No. of PT Samples	Total No. of Samples to Lab*
Sediment	TOC	low	Lloyd Kahn Empirical SOP-221	7	0	0	0	0	0	7
Soil/ Sediment	TCLP Organic	low	SW-846 1311/3510C/8260B/8270C/8081A/ 8151B/ Empirical SOP-198/300/302/304/202/201/211/208	5	NA	NA	0	NA	0	5
Soil/ Sediment	TCLP Inorganic	low	SW-846 1311/3005A/6010B/7470A Empirical SOP-198/100/105/ 103	5	NA	NA	0	NA	0	5

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).
Not Applicable.

* Approximate. Quantities subject to change.

TCL – Target Compound List
SVOCs – Semivolatile Organic Compounds
PAHs – Polycyclic Aromatic Hydrocarbons
TAL – Target Analyte List
TCLP – Toxicity Characteristic Leaching Procedure

QAPP_Worksheet_#21

(UFP-QAPP Manual Section 3.1.2)

List all SOPs associated with project sampling including, but not limited to, sample collection, sample preservation, equipment cleaning and decontamination, equipment testing, inspection and maintenance, supply inspection and acceptance, and sample handling and custody. Include copies of the SOPs as attachments or reference all in the QAPP. Sequentially number sampling SOP references in the Reference Number column. The reference number can be used throughout the QAPP to refer to a specific SOP.

☐ Worksheet Not Applicable (State Reason)

Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Check if yes)	Comments
S1	Utility Locating And Excavation Clearance, HS-1.0	TtNUS	Magnetometer	☐	
S2	Direct Push Technology, SA-2.5	TtNUS		☐	
S3	Soil Sampling, SA-1.3	TtNUS	Sampling Procedures, Methods	☐	
S4	Borehole and Sample Logging, GH-1.5	TtNUS		☐	
S5	Groundwater Sample Acquisition and On-site Water Quality Testing, SA-1.1	TtNUS	Sampling Procedures, Methods	☐	
S6	Evaluation of Exiting Monitoring Wells and Water Level Measurements, GH-1.2	TtNUS	Water level indicators	☐	
S7	Well Abandonment, GH-2.9	TtNUS		☐	
S8	Decontamination of Field Equipment and Waste Handling, SA-7.1	TtNUS	Decontamination Equipment (scrub brushes, phosphate free detergent, deionized water)	☐	
S9	Field Documentation, SA-6.3	TtNUS	Field Logbook, Field Sample Forms, Boring Logs	☐	
S10	USEPA Region I Low-Flow Purging and Sampling Methods	USEPA	Sampling Procedures, Methods	☐	
S11	USEPA Region I Draft Calibration of Field Instruments	USEPA	Water quality meters	☐	
S12	Surface Water and Sediment Sampling. SA-1.2	TtNUS	Sampling Procedures, Methods	☐	
S13	Groundwater Monitoring Well Installation, GH-2.8	TtNUS		☐	
S14	Soil and Rock Drilling Methods, GH-1.3	TtNUS	Hand auger	☐	

Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Check if yes)	Comments
S15	XRF SOP for Field Portable X-Ray Fluorescence	TtNUS	XRF unit	☐	

QAPP_Worksheet_#22

(UFP-QAPP Manual Section 3.1.2.4)

Identify all field equipment and instruments (other than analytical instrumentation) that require calibration, maintenance, testing, or inspection and provide the SOP reference number for each type of equipment. In addition, document the frequency of activity, acceptance criteria, and corrective action requirements on the worksheet.

☐ Worksheet Not Applicable (State Reason)

Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Water Quality Meter	Prepared Standards	NA	Calibrated in accordance with manufacture specifications and SOP S11(USEPA Region I methods). All parameters except ORP will use a minimum of two standards to bracket the measurement range. The ORP measurement uses one standard (Zobell standard).	Visual Inspection	Daily	Manufacture's Guidance	Replace	FOL	S5 and S11
XRF	Manufacture's Guidance	NA	Calibrated in accordance with manufacture specifications	Visual Inspection	Daily	Manufacture's Guidance	Replace	FOL	S15

[1]Specify the appropriate reference letter or number from the Project Sampling SOP References table (Worksheet_#21).

QAPP Worksheet #23

(UFP-QAPP Manual Section 3.2.1)

List all SOPs that will be used to perform on-site or off-site analysis. Indicate whether the procedure produces screening or definitive data. Sequentially number analytical SOP reference in the Reference Number column. Include copies of the SOPs as attachments or reference in the QAPP. The reference number can be used throughout the QAPP to refer to a specific SOP.

☐ Worksheet Not Applicable (State Reason)

Analytical SOP References Table

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
Empirical SOP-100	Metals Digestion/Preparation Methods 3005A,3010A, 3020A, 3030, 3040A, 3050B, USEPA CLP ILMO 4.1 Aqueous & Soil/Sediment, USEPA Method 200.7 (Standard Methods) 3030C. Rev. 19	Definitive	Soil, water, and leachate Metals digestion	Preparation	Empirical Laboratories, LLC	N
Empirical SOP-105	Metals Analysis by ICP Technique Methods 200.7, SW846 6010B, SM 19th Edition 2340B, USEPA ILMO 4.1, Rev. 14	Definitive	Soil, water, and leachate Metals	Inductively Coupled Plasma (ICP)	Empirical Laboratories, LLC	N
Empirical SOP-103	Mercury Analysis in Water by Manual Cold Vapor Technique Methods SW846 7470A & 245.1, CLP-M 4.1, Rev. 16	Definitive	Leachate and Water Mercury	Flow injection mercury analyzer	Empirical Laboratories, LLC	N
Empirical SOP-104	Mercury Analysis in Soil/Sediment by Manual Cold Vapor Technique Methods SW846 7471A & 245.5, CLP-ILM 4.1, Rev. 17	Definitive	Soil Mercury	Flow injection mercury analyzer	Empirical Laboratories, LLC	N
Empirical SOP-145	Determination of Inorganic Anions in water by ION Chromatography using Dionex DX-500 Ion Chromatograph with Hydroxide Eluent And Dionex Column AS18, Method 300.0 Guidance, Rev. 6	Definitive	Water Anions	Ion Chromatograph	Empirical Laboratories, LLC	N
Empirical SOP-198	Toxicity Characteristic Leaching Procedure (Method 1311), Rev. 6	Definitive	Solid IDW	Preparation	Empirical Laboratories, LLC	N
Empirical SOP-201	GC/MS Semivolatiles by Method 625 and SW846 Method 8270C, Rev. 18	Definitive	Leachate Semivolatile	Agilent/HP Gas Chromatorgraphy/Mass Spectroscopy (GC/MS)	Empirical Laboratories, LLC	N
Empirical SOP-202	GC/MS Volatiles by Method 624 and SW846 Method 8260B, Rev. 21	Definitive	Leachate Volatile	Agilent/HP GC/MS	Empirical Laboratories, LLC	N

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
EmpiricalSOP -211	GC/ECD Organochlorine Pesticides/PCBs by EPA Method 608 and SW846 Method 8081A/8082, Rev. 19	Definitive	Leachate Pesticide/PCBs	Agilent/HP GC/Electron Capture Detector (ECD)	Empirical Laboratories, LLC	N
EmpiricalSOP -208	GC/ECD Chlorinated Acid Herbicides by SW-846, Method 8150B/8151A, Rev. 13	Definitive	Leachate Herbicides	Agilent/HP GC/Electron Capture Detector (ECD)	Empirical Laboratories, LLC	N
Empirical SOP-231	GC/MS Low Level PAHs by Method SW-846 8270C SIM, Rev. 3	Definitive	Soil Volatiles Extraction	Agilent/HP GC/MS	Empirical Laboratories, LLC	N
Empirical SOP-300	GC/MS- Semivolatile BNA-Aqueous Matrix Extraction Using SW-846 Method 3510C for 8270C/625 Analysis, Rev. 17	Definitive	Leachate Semivolatiles Extraction	NA/Extraction	Empirical Laboratories, LLC	N
Empirical SOP-302	Pesticide/PCBs- Aqueous Matrix Extraction for EPA 608 and SW846 Method 8081A/8082 Using Method 3510C, Rev. 16	Definitive	Leachate Pesticide/PCB Extraction	NA/Extraction	Empirical Laboratories, LLC	N
Empirical SOP-304	Herbicides Aqueous Matrix by Methods EPA SW-846 Method 8151A, Rev. 11	Definitive	Leachate Herbicides Extraction	NA/Extraction	Empirical Laboratories, LLC	N
Empirical SOP-329	Soxhlet Extraction- BNA and Pest/PCB Using SW-846 Method 3541, Rev. 16	Definitive	Soil PAH Extraction	NA/Extraction	Empirical Laboratories, LLC	N
Empirical SOP-404	Laboratory Sample Receiving Log-in and Storage Standard Operating Procedures, Rev. 12	Definitive	Log-in	NA/ Log-in	Empirical Laboratories, LLC	N
Empirical SOP-405	Analytical Laboratory Waste Disposal, Rev. 4	Definitive	Log-in	NA/ Log-in	Empirical Laboratories, LLC	N
Empirical SOP-410	Standard Operating Procedures for Laboratory Sample Storage, Secure Areas, and Sample Custody, Rev. 6	Definitive	Log-in	NA/ Log-in	Empirical Laboratories, LLC	N
Empirical SOP-327	Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC) Method 8330A and 8332, Rev. 14	Definitive	Water and Soil Dinitrotoluenes and Nitroglycerin	HPLC/UV	Empirical Laboratories, LLC	N
HPLC-6850	Standard Operating Procedure for Perchlorate in Water, Soils, and Solid Wastes Using High Performance Liquid Chromatography/Electrospray Ionization/Mass Spectrometry (HPLC/ESI/MS), Rev. 3	Definitive	Water Perchlorate	LC/MS	Columbia Analytical Services	N

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?[1] (Y/N)
Empirical SOP-187	Electrometric Determination of pH, Methods 150.1, Standard Methods 4500H+B and 9040B for Waters, Liquids and Liquid Wastes, 9045C for Soils and Solid Wastes, Rev. 6	Definitive	Soil pH	pH Meter	Empirical Laboratories	N
Empirical SOP-221	Total Organic Carbon SM5310C, USEPA Method 415.1 and SW846 Method 9060 and Lloyd Kahn Method, Rev. 7	Definitive	Soil TOC	TOC Analyzer	Empirical Laboratories	N
EPA 9081	Cation Exchange Capacity of Soils	Definitive	Soil CEC	ICP	Empirical Laboratories	N

TCL – Target Compound List
TAL – Target Analyte List
TCLP – Toxicity Characteristic Leaching Procedure
GC/MS – Gas Chromatography/Mass Spectroscopy
GC/ECD - Gas Chromatography/Electron Capture Detector

QAPP Worksheet #24

(UFP-QAPP Manual Section 3.2.2)

Identify all analytical instrumentation that requires calibration and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

☐ Worksheet Not Applicable (State Reason)

Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA[2]	SOP Reference[1]
Gas Chromatograph/ Mass Spectrometer (GC/MS) Volatiles	Initial Calibration - A minimum 5 point calibration is required.	Calibrate the instrument when it is received and after a major change or if the daily calibration fails.	The percent relative standard deviation (RSD) for each Calibration Check Compound (CCCs) must be ≤ 30%. The minimum mean response factor (RF) for each System Performance Check Compound (SPCCs) must meet that stated in 8260B. If the RSD for an analyte is > 15% use a linear curve (≥ 0.995 correlation) or quadratic curve (≥0.99 correlation, minimum 6 points) for quantitation.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/Supervisor	Empirical SOP-202

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA[2]	SOP Reference[1]
	Initial calibration Verification (ICV)	Once after each initial calibration.	±20% recovery of individual compounds.	Identify source of problem, correct, repeat calibration, rerun samples		
	Continuing Calibration (CCV)	Analyze a standard at the beginning of each 12 hour shift after a Bromofluorobenzene (BFB) tune.	The minimum RF for SPCCs must meet that stated in method. The CCCs must be < 20 percent difference (%D).	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.		
GC/MS Semivolatiles and PAHs	Initial Calibration - A minimum 5 point calibration is required.	Calibrate the instrument when it is received and after a major change or if the daily calibration fails. A minimum 5 point calibration is required.	The RSD for each CCCs must be ≤ 30%. The minimum mean RF for each SPCCs must meet that stated in 8270C. If the RSD for an analyte is > 15% use a linear curve (≥ 0.995 correlation) or quadratic curve (≥0.99 correlation, minimum 6 points) for quantitation.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/Supervisor	Empirical SOP-201
	Initial Calibration Verification (ICV)	Once after each initial calibration.	±20% recovery individual compounds.	Identify source of problem, correct, repeat calibration, rerun samples		
	Continuing Calibration(CCV)	Analyze a standard at the beginning of each 12 hour shift after a decafluorotriphenyl phosphine (DFTPP) tune.	The minimum RF for SPCCs must meet those stated in method. The CCCs must be < 20 %D.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA[2]	SOP Reference[1]
GC/Electron Capture Detector (ECD) Pesticides/ Herbicides	Initial calibration - A minimum 5 point calibration is required.	Calibrate the instrument when it is received and after a major change or if the daily calibration fails.	The RSD for each compound must be ≤ 20%. A linear curve (≥ 0.995 correlation) or quadratic curve (≥0.99 correlation, minimum 6 points) for quantitation for all except toxaphene and chlordane. One point for toxaphene and chlordane.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst/ Supervisor	Empirical SOP-211/208
	Continuing Calibration (CCV)	Analyze standard at the beginning and end of sequence and every 10 samples.	≤ 15 %D.	If % D > +15% and samples are < the QL, narrate, If %D > ± 15% only on one column, narrate, If % D > ± 15% for closing CCV, and is likely due to matrix interference, narrate Otherwise reanalyze all samples back to the last acceptable CCV.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA[2]	SOP Reference[1]
Inductively Coupled Plasma (ICP) Metals	Initial Calibration	The instrument is calibrated at the beginning of each day or if the QC is out of criteria.	The instrument is calibrated by a one point calibration per manufacturer's guidelines. Analytes ran at their calibration levels must fall within 90-110% of the true values.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/Supervisor	Empirical SOP-105
	Initial Calibration Blank (ICB)	Before beginning a sample sequence.	No analytes detected > 2x MDL.	Correct the problem, then re-prepare and reanalyze.		
	Continuing Calibration(CCV)	Analyze a standard at the beginning and end of the sequence and after every 10 samples.	The acceptance criterion for the continuing calibration standard is 90-110% recovery of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.		
	Continuing Calibration Blank (CCB)	After every ten samples and at the end of the sequence.	No analytes detected > 2x MDL.	Correct the problem, then re-prepare and reanalyze calibration blank and previous ten samples.		
Flow Injection Mercury System	Initial Calibration	The instrument is calibrated at the beginning of each day or if the QC is out of criteria.	The calibration correlation coefficient is $\geq$0.995.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/Supervisor	Empirical SOP-103/104
	Continuing Calibration(CCV)	The CCV is analyzed at the beginning and end of the sequence and after every 10 samples.	The acceptance criteria are 80-120% recovery of the true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA[2]	SOP Reference[1]
High Perfomance Liquid Chromatography/ Ultraviolet Detector (HPLC/UV) Explosives	Initial Calibration - A minimum 5 point calibration is required.	Calibrate the instrument when it is received and after a major change or if the daily calibration fails.	The RSD for each compound must be ≤ 20%. A linear curve (≥ 0.995 correlation) or quadratic curve (≥0.99 correlation, minimum 6 points) for quantitation.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst/ Supervisor	Empirical SOP-233/327
	Initial calibration Verification (ICV)	Once after initial calibration.	85-115% recovery.	Identify source of problem, correct, repeat calibration, rerun samples.		
	Continuing Calibration (CCV)	Analyze standard at the beginning and end of sequence and every 10 samples.	≤ 15% difference.	If % D > +15% and samples are < the QL, narrate, If %D > ± 15% only on one column, narrate, If % D > ± 15% for closing CCV, and is likely due to matrix interference, narrate Otherwise reanalyze all samples back to the last acceptable.		
LC/MS/MS Perchlorate	Minimum six-point initial calibration for target analytes, lowest concentration standard at or near the quantitation limit.	Initial calibration prior to sample analysis.	Linear: r^2 > 0.990 (r>0.995), b < MDL. Quadratic: r^2 > 0.995.	Correct problem, then repeat initial calibration.	Lab Manager / Analyst	CAS HPLC-6850
	Second-source calibration verification	Once per five-point initial calibration.	Less than 15% difference for target analytes.	Correct problem, then repeat. If still fails, repeat initial calibration.		
	Daily calibration verification	Before sample analysis and every 10 samples or every 12 hours, as	Low-range standard: Perchlorate within	Correct problem, then repeat. If still fails, repeat initial calibration.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA[2]	SOP Reference[1]
		specified by the method, and at the end of the sequence.	±50% of the true value. Mid-range standard: Perchlorate within ±15% of the true value.			
	Initial Calibration Blank and Continuing Calibration Blank (ICB/CCB)	Following ICV/CCVs.	No target analyte concentrations above ½ the quantitation limit.	Rerun ICB. Isolate and correct problem. Reanalyze associated samples.		
	Ion Chromatography Calibration Standard (ICCS)/Interference check standard	Before sample analysis.	70% - 130% recovery	Rerun ICCS If problem still exists, recalibrate.		
	Maximum Conductivity Threshold Standard (MCT)/High TDS standard	Before sample analysis.	80% -120% recovery	Rerun MCT. If problem still exists, recalibrate.		
	Internal Standard	Every calibration standard.	ICAL: IS for each standard must be within ± 50% of the average area of the ICAL. ICV, CCV: Within ± 50% of the average area of the ICAL or within ± 50% of the 1st CCV of the run, if the ICAL is not run the same day.	Evaluate the system. Reanalyze/repeat the calibration.		
pH Meter	Calibration	Calibrate before use with two buffers in	0.05 pH units.	Recalibrate and/or perform necessary	Analyst/Supervisor	Empirical SOP-187

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA[2]	SOP Reference[1]
		the area to be measured. Check with a third buffer.		equipment maintenance.		
Soil TOC Analyzer	Five point calibration including blank	Instrument receipt, instrument change, when CCV does not meet criteria.	Correlation coefficient > 0.995	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst/ Supervisor	Empirical SOP-221
	Continuing calibration	After every 5 samples.	80-120%	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.		
CEC/ICP	Initial Calibration, One point calibration per manufacturer's guidelines	At the beginning of each day or if QC is out of criteria.	90-110% of true values.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst/ Supervisor	Empirical SOP-105
	Continuing calibration	After every 10 samples and at end of analysis sequence.	90-110% of true values.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards, Reanalyze affected data.	Analyst/ Supervisor	
Ion Chromatograph (nitrate and nitrite)	Eight point initial calibration	Instrument receipt, instrument change, when CCV does not meet criteria.	15% RSD or ≥ 0.995 correlation	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst/ Supervisor	Empirical SOP-145
	Initial Calibration Verification/Continuing Calibration Verfication	At the beginning of the sequence and after every ten samples	90-110% of true values.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards, Reanalyze affected data.	Analyst/ Supervisor	

[1] Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

[2] Name or title of responsible person may be used.

QC – Quality Control
CCC- Continuing calibration compound
RSD – Relative Standard Deviation
CCV – Continuing calibration verification
SPCC – System performance check compounds
SOP – Standard Operating Procedure
HPLC – High performance liquid chromatography
CAS – Columbia Analytical Services
%D – percent difference
BFB – Bromofluorobenzene
DFTPP - Decafluorotriphenylphosphine
RF – Response factor
Hg – Mercury
r^2 – Correlation Coeffiecient
GC/MS – Gas Chromatography/Mass Spectroscopy
GC/ECD - Gas Chromatography/Electron Capture Detector
ICP – Inductively Coupled Plasma
PQL – Practical Quantitation Limit
SVOC – Semivolatile Organic Compounds
FIMS – Flow Injection Mercury System

QAPP Worksheet #25

(UFP-QAPP Manual Section 3.2.3)

Identify all analytical instruments that require maintenance, testing, or inspection and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

☐ Worksheet Not Applicable (State Reason)

Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[2]	SOP Reference[1]
GC/MS	Clean the source and replace the filaments. Replace the seal, liner and septum. Change the column.	Volatiles/ Semivolatiles/PAHs	Check the gas supply. Check the seal, liner, and septum.	Source cleaning is performed when the instrument response deteriorates. Other instrument maintenance is done as needed to keep the instrument performing at peak performance.	The minimum RF for SPCCs must meet those stated in method. The CCCs must be < 20 %D.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	Empirical SOP-201/202
GC	Check pressure and gas supply daily. Bake out column, change septa, liner, seal as needed, cut column as needed.	Pesticides/ Herbicides	Liner, seal, septum, column	Prior to initial calibration or as necessary.	≤ 15 %D	If % D > +15% and samples are < QL, narrate, If %D > ± 15% only on one column, narrate. If % D > ± 15% for closing CCV, and is likely due to matrix interference, narrate Otherwise reanalyze all samples back to the last acceptable CCV.	Analyst/ Supervisor	Empirical SOP-211/208

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[2]	SOP Reference[1]
ICP	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 and CEC	Inspect the torch, nebulizer chamber, pump, and tubing.	Maintenance is performed prior to initial calibration or as necessary.	The acceptance criteria for the continuing calibration standard are 90-110% of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	Empirical SOP-105
Flow Injection Mercury System	Change the tubing, filter, clean windows, and check gas flow. Check the reagents and standards.	Mercury	Inspect the tubing, filter, and the optical cell.	Maintenance is performed prior to initial calibration or as necessary.	The acceptance criteria are 80-120% of the true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	Empirical SOP-103/104
HPLC/UV	Check pressure and gas supply daily – change when <200psi, change analytical column as needed, change mobile phase when insufficient for run or contamination, change inlet filters as needed for contamination.	Dinitrotoluenes and Nitroglycerin	Check pump pressure, check for leaks, check for adequate mobile phase.	Prior to initial calibration or as necessary.	CCV $\leq$ 15% difference.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst/ Supervisor	Empirical SOP-327
LC/MS/MS	Replace columns as needed, check eluent reservoirs.	Perchlorate Sensitivity check	Instrument performance and sensitivity.	Daily or as needed.	CCV pass criteria	Recalibrate	CAS Chemist	HPLC-6850

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[2]	SOP Reference[1]
TOC Analyzer	Check gas pressure, check permeation tube and scrubbers, clean and condition sample cups.	TOC	Check gas pressure, check permeation tube and scrubbers.	Prior to initial calibration or as necessary.	The midpoint standard must be within 20% difference.	Recalibrate and/or perform necessary equipment maintenance. Reanalyze samples not bracketed by passing continuing calibration.	Analyst/ Supervisor	Empirical SOP-221
pH Meter	Clean, drain, and refill reference electrode as needed.	pH	Reference electrode for white crystals, Inspect electrode for damage.	Before use.	0.05 pH units	Recalibrate and/or perform necessary equipment maintenance.	Analyst/ Supervisor	Empirical SOP-187
Ion Chromatograph	Change column, frits, tubing	Nitrate and Nitrite	Check gases, reagents	Prior to initial calibration or as necessary	ICV/CCV within10%	Recalibrate and/or perform necessary equipment maintenance. Reanalyze samples not bracketed by passing continuing calibration.	Analyst/ Supervisor	Empirical SOP-145

[1] Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

[2] Name or title of responsible person may be used.

BFB – Bromofluorobenzene
DFTPP - Decafluorotriphenylphosphine
SOP – Standard Operating Procedure
PCB – Polychlorinated biphenyls
Hg – Mercury
GC/MS – Gas Chromatography/Mass Spectroscopy
GC/ECD - Gas Chromatography/Electron Capture Detector
ICP – Inductively Coupled Plasma
QC – Quality Control
VOC – Volatile Organic Cmpounds
SVOC – Semivolatile Organic Compounds
FIMS – Flow Injection Mercury System

QAPP_Worksheet_#26

(UFP-QAPP Manual Appendix A)

Use this worksheet to identify components of the project-specific sample handling system. Record personnel, and their organizational affiliations, who are primarily responsible for ensuring proper handling, custody, and storage of field samples from the time of collection, to laboratory delivery, to final sample disposal. Indicate the number of days field samples and their extracts/digestates will be archived prior to disposal.

☐ Worksheet Not Applicable (State Reason)

Sample Handling System

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

QAPP_Worksheet_#27

(UFP-QAPP Manual Section 3.3.3)

Describe the procedures that will be used to maintain sample custody and integrity. Include examples of chain-of-custody forms, traffic reports, sample identification, custody seals, laboratory sample receipt forms, and laboratory sample transfer forms. Attach or reference applicable SOPs.

☐ Worksheet Not Applicable (State Reason)

Sample Custody Requirements

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory): Following sample collection in the appropriate bottleware, all samples will be immediately placed on ice in a cooler. The glass sample containers will be enclosed in bubble-wrap in order to protect the bottleware during shipment. The cooler will be secured using duct or clear packaging tape along with a signed custody seal. Sample coolers will be delivered to a local courier location for priority overnight delivery to the selected laboratory for analysis. Samples will be preserved as appropriate based on the analytical method. Laboratories will provide pre-preserved sample containers for sample collection. Samples will be maintained at 4° ± 2° Celsius (C) until delivery to the laboratories. Proper custody procedures will be followed throughout all phases of sample collection and handling. Chain of custody (COC) protocols will be used throughout sample handling to establish the evidentiary integrity of sample containers. These protocols will be used to demonstrate that the samples were handled and transferred in a manner that prevents tampering. Samples for the laboratory will be packaged and shipped in accordance with TtNUS SOP SA-6.1.
Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal): **Laboratory sample custody procedures (receipt of samples, archiving, and disposal) will be used according to Empirical SOPs 404,405,410. Coolers are 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 LIMS and given a unique log number which can be tracked thru processing. The client will be notified of any problems.**
Sample Identification Procedures: Sample will be named in accordance SAP Section 5.3 Empirical SOP-404
Chain-of-custody Procedures: After recovery, each sample will be maintained in the sampler's custody until formally transferred to another party (e.g., Federal Express). For all samples recovered, custody records will document the date and time of sample collection, the sampler's name, and the names of all others who subsequently held custody of the sample. Specifications for chemical analyses will also be documented on the custody record. Attached SOP SA-6.3 (Field Documentation) provides further details on the COC procedure. COC requirements are also documented with instructions contained in each shipment from the laboratory. Empirical SOP 404

QAPP Worksheet #28

(UFP-QAPP Manual Section 3.4)

Complete a separate worksheet for each sampling technique, analytical method/SOP, matrix, analytical group, and concentration level. If method/SOP QC acceptance limits exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

☐ Worksheet Not Applicable (State Reason)

Laboratory QC Samples Table

Matrix	Water/Soil
Analytical Group	PAHs
Analytical Method / SOP Reference	SW-846 8270C SIM/Empirical SOP-231

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per batch of 20 or less.	No target compounds should be ≥1/2 the quantitation limit (QL) except common lab contaminants which should be < QL.	Re-clean, retest, re-extract, reanalyze, and/or qualify the data.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	No target compounds > than the QL.
Surrogates	2 per sample.	Percent recoveries: Water: 2-Fluorobiphenyl 34-167 Terphenyl-d14 34-167 Soil: 2-Fluorobiphenyl 14-129 Terphenyl-d14 14-129	(1) Re-prepare and reanalyze for confirmation of matrix interference when appropriate.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Percent recoveries: Water: 2-Fluorobiphenyl 34-167 Terphenyl-d14 34-167 Soil: 2-Fluorobiphenyl 14-129 Terphenyl-d14 14-129
Laboratory Control Sample (LCS)	One per batch of 20 or less.	Compound specific as per SOP-231.	(1) Evaluate and reanalyze if possible. (2) If an MS/MSD was performed in the same 12 hour clock and acceptable	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Compound specific as per SOP-231.

Matrix	Water/Soil					
			narrate. (3) If the LCS recoveries are high but the sample results are <QL, then narrate, otherwise re-prepare and reanalyze the batch.			
Internal Standards (IS)	2 per sample Perylene-d12 Phenanthrene-d10	Retention times for internal standards must be ± 30 seconds and the responses within -50% to +100% of last calibration verification (12 hours) for each internal standard.	Inspect mass spectrometer or gas chromatograph for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Retention times for internal standards must be ± 30 seconds and the responses within -50% to +100% of last calibration verification (12 hours) for each internal standard.
Matrix spike / Matrix spike duplicate (MS/MSD)	One per SDG or every 20 samples.	Compound specific as per SOP-231.	1) Corrective action will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. 2) If both the LCS and MS/MSD are unacceptable re-prepare the samples and QC.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Compound specific as per SOP-231.

Matrix	Leachate
Analytical Group	Volatiles
Analytical Method / SOP Reference	SW-846 8260B/Empirical SOP-202

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per batch of 20 or less.	No target compounds should be ≥1/2 the quantitation limit (QL) except common lab contaminants which should be < the QL.	Reclean, retest, re-extract, reanalyze, and/or qualify the data.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	No target compounds should be > the QL.
Surrogates	3 per sample	Percent recoveries: Water: Dibromofluoromethane 85-120 1,2-dichloroethane-d4 85-130 Toluene-d8 85-115 Bromofluorobenzene 80-120	(1) Re-prepare and reanalyze for confirmation of matrix interference when ppropriate.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Percent recoveries: Water: Dibromofluoromethane 85-120 1,2-dichloroethane-d4 85-130 Toluene-d8 85-115 Bromofluorobenzene 80-120
Laboratory Control Sample (LCS)	One per batch of 20 or less.	Compound specific as per SOP-202.	(1) Evaluate and reanalyze if possible. (2) If an MS/MSD was performed in the same 12 hour clock and acceptable narrate. (3) If the LCS recoveries are high but the sample results are <QL, narrate. Otherwise reprepare and reanalyze the	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Compound specific as per SOP-202.

Matrix	Leachate					
			batch.			
Internal Standards	3 per sample- Fluorobenzene Chlorobenzene-d5 1,4-dichlorobezene-d4	Retention times for internal standards must be ± 30 seconds and the responses within -50% to +100% of last calibration verification (12 hours) for each internal standard.	Inspect mass spectrometer or gas chromatograph for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Retention times for internal standards must be ± 30 seconds and the responses within -50% to +100% of last calibration verification (12 hours) for each internal standard.
Matrix spike / Matrix spike duplicate (MS/MSD)	One per sample delivery group (SDG) or every 20 samples.	Compound specific as per SOP-202.	Corrective actions will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. If both the LCS and MS/MSD are unacceptable reprepare the samples and QC.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Compound specific as per SOP-202.

Matrix	Leachate
Analytical Group	Semivolatile
Analytical Method / SOP Reference	SW-846 8270C/Empirical SOP-201

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per batch of 20 or less.	No target compounds should be ≥1/2 the QL except common lab contaminants which should be < the QL.	Reclean, retest, re-extract, reanalyze, and/or qualify the data.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	No target compounds should be > the QL.
Surrogates	6 per sample	Water: Nitrobenzene-d5 30-110 2-Fluorobiphenyl 35-110 Terphenyl-d14 55-125 Phenol-d6 15-110 2-Fluorophenol 15-110 2,4,6-Tribromophenol 45-125	(1) Re-prepare and reanalyze for confirmation of matrix interference when appropriate.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Water: Nitrobenzene-d5 30-110 2-Fluorobiphenyl 35-110 Terphenyl-d14 55-125 Phenol-d6 15-110 2-Fluorophenol 15-110 2,4,6-Tribromophenol 45-125
Laboratory Control Sample (LCS)	One per batch of 20 or less.	Compound specific as per SOP-201.	(1) Evaluate and reanalyze if possible. (2) If an MS/MSD was performed in the same 12 hour clock and acceptable narrate. (3) If the LCS recoveries are high but the sample results are < QL, then narrate. Otherwise reprepare and	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Compound specific as per SOP-201.

Matrix	Leachate

			reanalyze the batch.			
Internal Standards	6 per sample – 1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	Retention times for internal standards must be ± 30 seconds and the responses within -50% to +100% of last calibration verification (12 hours) for each internal standard.	Inspect mass spectrometer or Gas chromatograph for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Retention times for internal standards must be ± 30 seconds and the responses within -50% to +100% of last calibration verification (12 hours) for each internal standard.
Matrix spike / Matrix spike duplicate (MS/MSD)	One per SDG or every 20 samples.	Compound specific as per SOP-201.	(1)Corrective action will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. (2) If both the LCS and MS/MSD are unacceptable reprepare the samples and QC.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Compound specific as per SOP-201.

Matrix	Water/TCLP
Analytical Group	Metals
Analytical Method / SOP Reference	SW-846 3005A, 6010B/7470A/SOP-100/103/105

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per digestion batch of 20 or	Contaminants in the	1) Investigate	Analyst, Laboratory	Bias /	Contaminants in the

Matrix	Water/TCLP					
	fewer samples.	method blank must be less than ½ the QL.	the source of the contamination. Redigest and reanalyze all associated samples if the sample concentration ≥ the QL and <10x the blank concentration.	Supervisor and Data Validator	Contamination	method blank must be less than the QL.
Laboratory Control Sample(LCS)	One per digestion batch of 20 or fewer samples.	Recovery must be within ± 20% of the true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias / Contamination	Recovery must be within ± 20% of the true value, unless vendor-supplied or statistical limits have been established.
Duplicate Sample	One per digestion batch of 20 or fewer samples.	The relative percent difference should be within ≤20% for duplicate spikes.	Flag results	Analyst, Laboratory Supervisor and Data Validator	Precision	The relative percent difference should be within ≤20% for duplicate spikes.
Matrix Spike (MS)	One per digestion batch of 20 or fewer samples.	Recovery should be ± 25% of the true value, if sample < 4x spike added.	Flag results.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Recovery should be ± 25% of the true value, if sample < 4x spike added.
ICP Serial Dilution	One per digestion batch.	If original sample result is at least 50x the instrument detection limit, 5-fold dilution must agree within ± 10% of the original result.	Flag result or dilute and reanalyze sample to eliminate interference.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	If original sample result is at least 50x the instrument detection limit, 5-fold dilution must agree within ± 10% of the original result.

Matrix	Soil
Analytical Group	Metals
Analytical Method / SOP Reference	SW-846 3050B, 6010B/SOP-100/105

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per digestion batch of 20 or fewer samples.	Contaminants in the method blank must be less than ½ the QL.	Investigate the source of the contamination. Re-digest and reanalyze all associated samples if the sample concentration ≥ the reporting limit and <10x the blank concentration.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	Contaminants in the method blank must be less than the QL.
Laboratory Control Sample(LCS)	One per digestion batch of 20 or fewer samples.	Recovery must be within ± 20% of the true value, unless vendor-supplied or statistical limits have been established.	Investigate source of problem. Redigest and reanalyze all associated samples.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias / Contamination	Recovery must be within ± 20% of the true value, unless vendor-supplied or statistical limits have been established.
Duplicate Sample	One per digestion batch of 20 or fewer samples.	The relative percent difference should be within ≤20% for duplicate spikes.	Flag results.	Analyst, Laboratory Supervisor and Data Validator	Precision	The relative percent difference should be within ≤20% for duplicate spikes.
Matrix Spike	One per digestion batch of 20 or fewer samples.	Recovery should be ± 25% of the true value, if sample < 4x spike added.	Flag results.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Recovery should be ± 25% of the true value, if sample < 4x spike added.

Matrix	Soil					
ICP Serial Dilution	One per digestion batch.	If original sample result is at least 50x the instrument detection limit, 5-fold dilution must agree within ± 10% of the original result.	Flag result or dilute and reanalyze sample to eliminate interference.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	If original sample result is at least 50x the instrument detection limit, 5-fold dilution must agree within ± 10% of the original result.

Matrix	Soil
Analytical Group	Metals
Analytical Method / SOP Reference	SW-846 3050B, 6010B/7471A/SOP-100/104/105

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per digestion batch of 20 or fewer samples.	Contaminants in the method blank must be less than ½ QL.	1) Investigate the source of the contamination. Redigest and reanalyze all associated samples if the sample concentration ≥ the QL and <10x the blank concentration.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	Contaminants in the method blank must be less than the QL.
Laboratory Control Sample(LCS)	One per digestion batch of 20 or fewer samples.	Recovery must be within ± 20% of the true value, unless vendor-supplied or statistical limits have been established.	3) Investigate source of problem. 4) Redigest and reanalyze all associated samples.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias / Contamination	Recovery must be within ± 20% of the true value, unless vendor-supplied or statistical limits have been established.
Duplicate Sample	One per digestion batch of 20 or fewer samples.	The relative percent difference should be within ≤20% for duplicate spikes.	Flag results	Analyst, Laboratory Supervisor and Data Validator	Precision	The relative percent difference should be within ≤20% for duplicate spikes.
Matrix Spike	One per digestion batch of 20 or fewer samples.	Recovery should be ± 25% of the true value, if sample < 4x spike added.	Flag results.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Recovery should be ± 25% of the true value, if sample < 4x spike added.

Matrix	Soil					
ICP Serial Dilution	One per digestion batch.	If original sample result is at least 50x the instrument detection limit, 5-fold dilution must agree within ± 10% of the original result.	Flag result or dilute and reanalyze sample to eliminate interference.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	If original sample result is at least 50x the instrument detection limit, 5-fold dilution must agree within ± 10% of the original result.

Matrix	Leachate
Analytical Group	Pesticides
Analytical Method / SOP Reference	SW-846 8081A/SOP-302/211

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per 20 samples or less.	No target compounds ≥ 1/2 QL.	Re-clean, retest, re-extract, reanalyze, and/or qualify data.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	Contaminants in the method blank must be less than the QL.
Surrogates	2 per sample.	Water: Tetrachloro-m-xylenes 25-120 Decachlorobiphenyl 25-130	(1) Re-prepare and reanalyze for confirmation of matrix interference when appropriate.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	Water: Tetrachloro-m-xylenes 25-120 Decachlorobiphenyl 25-130
Laboratory Control Sample (LCS)	One per 20 samples or less.	Compound specific as per SOP-211.	(1) Evaluate and reanalyze if possible. (2) If an MS was performed in the same 12 hour clock and acceptable narrate.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Compound specific as per SOP-211.

Matrix	Leachate					
			(3) If the LCS recoveries are high but the sample results are <QL narrate otherwise re-prepare and reanalyze.			
Matrix spike/Matrix Spike Duplicate (MS/MSD)	One per sample matrix.	Compound specific as per SOP-211.	(1) Corrective action will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. (2) If both the LCS and MS are unacceptable re-prep the samples and QC.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Compound specific as per SOP-211.

Matrix	Leachate
Analytical Group	Herbicides
Analytical Method / SOP Reference	SW-846 8151A/SOP-304/208

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per 20 samples or less	No target compounds ≥ 1/2 QL.	Re-clean, retest, re-extract, reanalyze, and/or qualify data.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	No target compounds ≥ QL.
Surrogates	1 per sample	2,4-Dichlorophenylacetic acid. water: 20-140% recovery	(1) Re-prepare and reanalyze for confirmation of matrix interference when appropriate.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	2,4-Dichlorophenylacetic acid. water: 20-140% recovery
Laboratory Control Sample (LCS)	One per 20 samples or less.	Compound specific as per SOP- 208.	(1) Evaluate and reanalyze if possible. (2) If an MS was performed in the same 12 hour clock and acceptable narrate. (3) If the LCS recoveries are high but the sample results are <QL narrate otherwise re-prepare and reanalyze.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Compound specific as per SOP- 208.

Matrix	Leachate					
Matrix spike/Matrix Spike Duplicate (MS/MSD)	One per sample matrix.	Compound specific as per SOP- 208.	(1) Corrective action will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. (2) If both the LCS and MS are unacceptable then the sample and QC will be re-prepared.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Compound specific as per SOP- 208.

Matrix	Water/Soil
Analytical Group	Dinitrotoluenes and Nitroglycerin
Analytical Method / SOP Reference	8330AEmpirical SOP-327

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per batch of 20 or less.	No target compounds >QL.	Reclean, retest, re-extract, reanalyze, and/or qualify data	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	No target compounds > QL.
Surrogates	1.per sample	1,2-dinitrobenzene 60%-140% for water 50-150% for soil	Re-prepare and reanalyze for confirmation of matrix interference when appropriate.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	1,2-dinitrobenzene 60%-140% for water 50-150% for soil
Laboratory Control Spike	One per batch of 20 or less	Water: 60 - 120% Soil: 60 - 120%	Evaluate and reanalyze if possible. If an MS/MSD was performed in the same 12 hour clock and is acceptable narrate. If the LCS recoveries are high but the sample results are <QL then narrate, otherwise re-prepare and reanalyze.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Water: 60 - 120% Soil: 60 - 120%

Matrix	Water/Soil					
Matrix spike / Matrix spike duplicate	One per sample delivery group or every 20 samples.	%Recovery = 50 - 140 RPD ≤ 50	Corrective action will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. If both the LCS and MS/MSD are unacceptable then re-prepare the samples and QC.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	%Recovery = 50 - 140 RPD ≤ 50

Matrix	Water
Analytical Group	Perchlorate
Analytical Method / SOP Reference	SW-846 6850/CAS HPLC-6850

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per preparation batch.	No target analytes ≥ 1/2 QL.	Correct problem, then re-extract and reanalyze method blank and all samples processed with the contaminated blank in accordance with DoD QSM requirements.	Lab Manager / Analyst	Accuracy/Bias Contamination	No target analytes ≥ ½ QL.
Internal standards	During acquisition of calibration standard, samples, and QC check samples.	Areas within -50% of the average of the last initial calibration for each sample and QC in accordance with DoD QSM requirements.	Inspect LCMS for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning in accordance with DoD QSM requirements.	Lab Manager / Analyst	Precisions and Accuracy/Bias	Areas within -50% of the average of the last initial calibration for each sample and QC in accordance with DoD QSM requirements.
Isotope Ratio	Every Sample and QC.	83/85 ratio within the established window of 2.3-3.8	Samples must be repeated. Re-extract using cleanup procedures or alternative techniques	Lab Manager / Analyst	Qualitative Idenitfication	83/85 ratio within the established window of 2.3-3.8

Matrix	Water					
			such as post spikes or dilution to reduce interference			
MS/MSD	One MS/MSD pair per analytical/ preparation batch.	QC acceptance criteria: 80% to 120% recovery, 15% relative percent difference; or laboratory statistically derived control limits.	Identify problem; if not related to matrix interference, re-extract and reanalyze MS/MSD and all associated batch samples in accordance with DoD QSM requirements.	Lab Manager / Analyst	Precisions and Accuracy/Bias	QC acceptance criteria: 80% to 120% recovery, 15% relative percent difference; or laboratory statistically derived control limits.
LCS	One LCS per analytical/preparation batch.	QC acceptance criteria: 80% to 120% recovery, 15% relative percent difference; or laboratory statistically derived control limits.	Correct problem, then re-extract and reanalyze the LCS and all associated batch samples in accordance with DoD QSM requirements.	Lab Manager / Analyst	Precisions and Accuracy/Bias	QC acceptance criteria: 80% to 120% recovery, 15% relative percent difference; or laboratory statistically derived control limits.
MDL study	Once per 12-month period	Detection limits established will be below the QLs	Correct problem, then repeat the MDL study in accordance with DoD QSM requirements	Lab Manager / Analyst	Sensitivity	Detection limits will be below the QLs

Matrix	Soil
Analytical Group	Total Organic Carbon
Analytical	Lloyd Kahn/

Matrix	Soil
Method / SOP Reference	SOP-221

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples	No TOC detected > 1/2 QL.	Investigate source of contamination and re-analyze all affected samples.	Analyst, Supervisor, and QA Manager	Accuracy/bias-Contamination	No TOC detected > QL.
Laboratory Duplicate	One per preparation batch of 10 or fewer samples	≤Relative percent difference ≤ 50	Narrate any results that are outside control limits.	Analyst, Supervisor, and QA Manager	Precision	Relative percent difference ≤ 50
Matrix Spike	One per preparation batch of 10 or fewer samples	50-150 % recovery	No corrective action will be taken for samples where recoveries are outside acceptance limits and LCS criteria are met. Narrate noncompliances.	Analyst, Supervisor, and QA Manager	Accuracy/bias	50-150 % recovery
Laboratory Control Sample	One per preparation batch of 20 or fewer samples	80-120% recovery	Re-analyze associated samples. If the sample is within holding time, re-analyze affected sample batch. If the LCS recovery is high but the sample results are < the QL, narrate. Otherwise, re-prepare the blank and affected sample batch.	Analyst, Supervisor, and QA Manager	Accuracy/bias	80-120% recovery

Matrix	Soil
Analytical Group	pH
Analytical Method / SOP Reference	SW-846 9045C/ SOP-187

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Duplicate	One per preparation batch of 10 or fewer samples.	± 0.09 units	Recalibrate and reanalyze samples.	Analyst, Supervisor, and QA Manager	Precision	± 0.09 units
Laboratory Control Sample	Once per 20 samples.	± 0.25 units	Re-analyze associated samples. If sample is within holding time, re-analyze affected sample batch.	Analyst, Supervisor, and QA Manager	Accuracy/bias	± 0.25 units

Matrix	Soil
Analytical Group	CEC
Analytical Method / SOP Reference	SW-846 9081

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch of 10 or fewer samples.	Less than the QL.	Correct and reanalyze batch	Analyst, Supervisor, QA Manager	Accuracy/bias-Contamination	Less than the QL.
Laboratory Duplicate	One per preparation batch of 10 or	Relative percent difference < 20	Report	Analyst, Supervisor, QA Manager	Precision	Relative percent difference < 20

Matrix	Soil					
	fewer samples.					

Matrix	Water
Analytical Group	Nitrate and Nitrite
Analytical Method / SOP Reference	EPA 300/ SOP-145

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Reagent Blank	One per batch	No analytes > ½ QL.	Investigate source of contamination and re-analyze all affected samples.	Analyst, Supervisor, and QA Manager	Accuracy/bias-Contamination	No analytes > ½ QL.
Laboratory Fortified Blank (LCS)	One per batch	90-110% recovery	Re-analyze associated samples. If sample is within holding time, re-analyze affected sample batch.	Analyst, Supervisor, and QA Manager	Accuracy/bias	90-110% recovery
Laboratory Fortified Sample Matrix (MS)	10% of samples	80-120% recovery	No corrective action will be taken for samples where recoveries are outside acceptance limits and LCS criteria are met. Narrate noncompliances.	Analyst, Supervisor, and QA Manager	Accuracy/bias	80-120% recovery
Laboratory Duplicate	10% of samples	20% RPD	If the RPD fails to meet these criteria, the samples must be reported with a	Analyst, Supervisor, QA Manager	Precision	20% RPD

Matrix	Water					
			qualifier identifying the sample analysis result as yielding a poor duplicate analysis RPD.			

QL – Quantitation Limit
VOC – Volatile Organic Cmpounds
SVOC – Semivolatile Organic Compounds
TAL – Target Analyte List
TCL – Target Compound List
TCLP – Toxicity Characteristic Leaching Procedure
TBD – To be Determined
RPD – Relative Percent Difference
IDL – Instrument Detection Limit
SDG – Sample Delivery Group
RL - Reporting Limit
QC - Quality Control
EICP - Extracted Ion Current Profile
ICP – Inductively Coupled Plasma
EICP – Extracted ion current profile
MRL – Method Reporting Limit
LODV – Level of detection verification
FOL – Field Operation Leader
LODV – Limit of Detection Verification

QAPP_Worksheet_#29

(UFP-QAPP Manual Section 3.5.1)

Identify the documents and records that will be generated for all aspects of the project including, but not limited to, sample collection and field measurement, on-site and off-site analysis, and data assessment.

☐ Worksheet Not Applicable (State Reason)

Project Documents and Records Table

Sample Collection Documents and Records	On-site Analysis Documents and Records	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
Field Logbook Field Sample Forms Chain of Custody Records Airbills Sampling Instrument Calibration Logs Sample Location and Sampling Plan Sampling Notes and Drilling Logs Sampling Report Photographs Field Task Modification Forms	Field Sample Data Field Logbook Field Sample Forms Sampling Instrument Calibration Logs Field Sample Data	-Sample receipt, custody, and tracking record -Standards tracability logs -Equipment calibration logs -Sample preparation logs -Run logs -Equipment maintenance, testing, and inspection logs -Corrective action 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 (stored electronically)	Field Sampling Audit Checklist (if audit is conducted) Analytical Audit Checklist (if Audit is conducted) Data Review Reports Tabulated Data Summary Forms Data Validation Memoranda	

QAPP Worksheet #30

(UFP-QAPP Manual Section 3.5.2.3)

Complete this worksheet for each matrix, analytical group, and concentration level. Identify all laboratories or organizations that will provide analytical services for the project, including on-site screening, on-site definitive, and off-site laboratory analytical work. If applicable, identify the subcontractor laboratories and backup laboratory or organization that will be used if the primary laboratory or organizations cannot be used.

☐ Worksheet Not Applicable (State Reason)

Analytical Services Table

Matrix	Analytical Group	Sample Locations/ID Numbers	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
Water and Soil	PAHs	See Worksheet 18	SW-846 8270C SIM	21 calendar days	Janice Shilling Empirical Laboratories 227 French Landing Nashville, TN 37228 615-345-1115	NA
	Metals	See Worksheet 18	SW-846 6010B 7470A/7471A			
	Dinitrotoluenes and nitroglycerin	See Worksheet 18	SW-846 8330A			
Water	Nitrate and Nitrite	See Worksheet 18	EPA 300			
Soil	TCLP Organics	See Worksheet 18	SW-846 1311 followed by 8260B, 8270C, 8081A, 8151A			
	TCLP Inorganics	See Worksheet 18	SW-846 1311 followed by 6010B, 7470A			
	pH	See Worksheet 18	SW-846 9045C			

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)
	CEC	See Worksheet 18	SW-846 9081			
	TOC	See Worksheet 18	Lloyd Kahn			
Water	Perchlorate	See Worksheet 18	SW-846 6850		Deb Patton Columbia Analytical Services 1 Mustard Street, Suite 250 Rochester, NY 14609 585-288-5380	NA

TAL – Target Analyte List
TCL – Target Compound List
PAHS – Polycyclic Aromatic Hydrocarbons
TOC - Total Organic Carbon
TCLP – Toxicity Characteristic Leaching Procedure
NA – Not applicable
TBD – To be determined

QAPP_Worksheet_#31

(UFP-QAPP Manual Section 4.1.1)

Identify the type, frequency, and responsible parties of planned assessment activities that will be preformed for the project.

☐ Worksheet Not Applicable (State Reason)

Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)	Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (Title and Organizational Affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation)
Laboratory Systems Audit	Every 18 months	External	NFESC	TBD	Laboratory QA Manager	Laboratory QAM	Laboratory QAM
Field Sampling Systems Audit	1 per contract year	Internal	TtNUS	TBD	PM	Auditor and Project Chemist (Kelly Carper TtNUS)	Project Chemist (Kelly Carper TtNUS)

QAPP_Worksheet_#32

(UFP-QAPP Manual Section 4.1.2)

For each type of assessment describe procedures for handling QAPP and project deviations encountered during the planned project assessments.

☐ Worksheet Not Applicable (State Reason)

Assessment Findings and Corrective Action Responses

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, Org.)	Timeframe for Response
Field sampling system audit	Audit checklist and written audit finding summary	PM TtNUS, FOL TtNUS, and Program management TtNUS	Dependant on findings, if major a stop work maybe issued immediately, however if minor within 1 week of audit	Written memo	QAM TtNUS, Auditor TtNUS, Program Manager TtNUS	Within 4 weeks of notification
Laboratory systems Audit	Written audit report	Laboratory QAM	Not specified by NFESC	Letter	NFESC	Specified by NFESC

QAPP_Worksheet_#33

(UFP-QAPP Manual Section 4.2)

Identify the frequency and type of planned QA Management Reports, the projected delivery date, the personnel responsible for report preparation, and the report recipients.

☐ Worksheet Not Applicable (State Reason)

QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Title and Organizational Affiliation)	Report Recipient(s) (Title and Organizational Affiliation)
Data validation report	Per SDG	Once validation is complete	DVM or designee (TtNUS)	PM (TtNUS), project file
Major analysis problem identification (Internal Memorandum)	When persistent analysis problems are detected	immediately	Project Chemist	PM (TtNUS), CG (TtNUS), DVM (TtNUS), QAM (TtNUS), and project file
Project monthly progress report	Monthly for duration of the project	monthly	PM (TtNUS)	Navy, project file
Field status reports	Daily, oral, during the course of sampling	everyday that field sampling is occurring	FOL (TtNUS)	PM and CG (TtNUS)
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	immediately	Subcontracted laboratories	TtNUS, project file

QAPP_Worksheet_#34

(UFP-QAPP Manual Section 5.2.1)

Describe the processes that will be followed to verify project data. Verification inputs include items such as those listed in Table 9 of the UFP-QAPP Manual (Section 5.1). Describe how each item will be verified, when the activity will occur, and what documentation is necessary, and identify the persons responsible. *Internal* or *external* is in relation to the data generator.

☐ Worksheet Not Applicable (State Reason)

Verification (Step I) Process Table

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Sample Tables	Proposed samples verified to have been collected and are documented on the chain of custody forms.	Internal	FOL or Designee TtNUS
Sample Coordinates	Sample locations have been verified to be correct and in accordance with the QAPP (overlay maps proposed locations against actual locations)	Internal	FOL, PM, or designee TtNUS
Data package	Verify that the data package contains all the elements required by the scope of work, this occurs as part of the data validation process	Internal	Data validator TtNUS
Sample logsheets	Logsheets completed as samples are collected in the field are verified for completeness and are compiled for inclusion in the RI/FS	Internal	PM or designee TtNUS
Audit Report	Audit checklist and narrative noting any deficiencies (if an audit is performed)	Internal	QAM, FOL, PM

QAPP_Worksheet_#35

(UFP-QAPP Manual Section 5.2.2)

Describe the processes that will be followed to validate project data. Validation inputs include items such as those listed in Table 9 of the UFP-QAPP Manual (Section 5.1). Describe how each item will be validated, when the activity will occur, and what documentation is necessary and identify the person responsible. Differentiate between steps IIa and IIb of validation.

☐ Worksheet Not Applicable (State Reason)

Validation (Steps IIa and IIb) Process Table

Step IIa/IIb	Validation Input	Description	Responsible for Validation (Name, Organization)
IIa	Data package	Validator will verify that elements of the data package that are required for validation are present and if not the lab will be contacted and the missing info will be requested. validation will be performed as per worksheet 36.	Validator TtNUS
IIa	Field logs/sample coordinates	Verify that the sampling plan was implemented and carried out as written and any deviations are documented	PM TtNUS
IIa	Electronic Data	Verify all data have been transferred correctly and completely to the final SQL database	PM or designee TtNUS
IIa	QAPP, SOPs/Field Logs, chains of custody	Verify that deviations have been documented and MPCs have been achieved.	PM, FOL, or designee TtNUS

QAPP_Worksheet_#36

(UFP-QAPP Manual Section 5.2.2)

Identify the matrices, analytical groups, and concentration levels that each entity performing validation will be responsible for, as well as criteria that will be used to validate those data.

☐ Worksheet Not Applicable (State Reason)

Validation (Steps IIa and IIb) Summary Table

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIB	Groundwater, Surface water, Sediment, Surface and Subsurface Soil	TAL Metals	low	SW-846, Region I Inorganic Data Validation Functional Guidelines (2/89), DOD QSM, and QAPP Worksheets 12, 15, and 28.	TtNUS Staff Chemist
IIa and IIB	Groundwater, Surface and Subsurface Soil	Explosives	low	SW-846, DOD QSM, Region I Part 2 Volatile/semivolatile Data Validation Functional Guidelines (12/96) and QAPP Worksheets 12, 15, and 28.	TtNUS Staff Chemist
IIa and IIB	Groundwater, Surface and Subsurface Soil	Nitroglycerin	low	SW-846, DOD QSM, Region I Part 2 Volatile/semivolatile Data Validation Functional Guidelines (12/96) and QAPP Worksheets 12, 15, and 28.	TtNUS Staff Chemist
IIa and IIB	Groundwater, Surface water, Sediment, Surface and Subsurface Soil	PAHs	low	SW-846, Region I Part 2 Volatile/semivolatile Data Validation Functional Guidelines (12/96), DOD QSM, and Worksheets 12, 15, and 18.	TtNUS Staff Chemist
IIa and IIB	Groundwater	Perchlorate	low	SW-846, DOD Perchlorate Handbook, and QAPP Worksheets 12, 15, and 28.	TtNUS Staff Chemist
IIa and IIB	Groundwater	Nitrates	low		TtNUS Staff Chemist

Validation (Steps IIa and IIb) Summary Table

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIB	Surface Soil and Sediment	TOC	low	method-specific, DOD QSM to extent practicable, and QAPP Worksheets 12, 15, and 28.	TtNUS Staff Chemist
IIa and IIB	Surface Soil	pH and CEC	low		TtNUS Staff Chemist
IIa and IIB	Soil	TCLP Organic	low	SW-846, Region I Parts 2 and 3 Volatile/semivolatile and Pesticide/PCB Data Validation Functional Guidelines (12/96), DOD QSM, and QAPP Worksheets 12, 15, and 28.	TtNUS Staff Chemist
IIa and IIB	Soil	TCLP Inorganic	low	SW-846, Region I Inorganic Data Validation Functional Guidelines (2/89), DOD QSM, and QAPP Worksheets 12, 15, and 28.	TtNUS Staff Chemist

QAPP_Worksheet_#37

(UFP-QAPP Manual Section 5.2.3)

Describe the procedures/methods/activities that will be used to determine whether data are of the right type, quality, and quantity to support environmental decision-making for the project. Describe how data quality issues will be addressed and how limitations of the use of the data will be handled.

☐ Worksheet Not Applicable (State Reason)

Usability Assessment

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used: See QAPP Section 4.2.3 text
Describe the evaluative procedures used to assess overall measurement error associated with the project: See QAPP Section 4.2.3 text
Identify the personnel responsible for performing the usability assessment: Project Chemist or designee
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: See QAPP Section 4.2.3 text

ATTACHMENT 1-B

FIELD SOPS

S1 HS-1.0 UTILITY LOCATING AND EXCAVATION CLEARANCE

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1.0 PURPOSE

Utilities such as electric service lines, natural or propane gas lines, water and sewage lines, telecommunications, and steam lines are very often in the immediate vicinity of work locations. Contact with underground or overhead utilities can have serious consequences including employee injury/fatality, property and equipment damage, substantial financial impacts, and loss of utility service to users.

The purpose of this procedure is to provide minimum requirements and technical guidelines regarding the appropriate procedures to be followed when performing subsurface and overhead utility locating services. It is the policy of Tetra Tech NUS, Inc. (TtNUS) to provide a safe and healthful work environment for the protection of our employees. The purpose of this Standard Operating Procedure (SOP) is to aid in achieving the objectives of this policy, to present the acceptable procedures pertaining to utility locating and excavation clearance activities, and to present requirements and restrictions relevant to these types of activities. This SOP must be reviewed by any employee potentially involved with underground or overhead utility locating and avoidance activities.

2.0 SCOPE

This procedure applies to all TtNUS field activities where there may be potential contact with underground or overhead utilities. This procedure provides a description of the principles of operation, instrumentation, applicability, and implementability of typical methods used to determine the presence and avoidance of contact with utility services. This procedure is intended to assist with work planning and scheduling, resource planning, field implementation, and subcontractor procurement. Utility locating and excavation clearance requires site-specific information prior to the initiation of any such activities on a specific project. This SOP is not intended to provide a detailed description of methodology and instrument operation. Specialized expertise during both planning and execution of several of the methods presented may also be required.

3.0 GLOSSARY

Electromagnetic Induction (EMI) Survey - A geophysical exploration method whereby electromagnetic fields are induced in the ground and the resultant secondary electromagnetic fields are detected as a measure of ground conductivity.

Magnetometer – A device used for precise and sensitive measurements of magnetic fields.

Magnetic Survey – A geophysical survey method that depends on detection of magnetic anomalies caused by the presence of buried ferromagnetic objects.

Metal Detection – A geophysical survey method that is based on electromagnetic coupling caused by underground conductive objects.

Vertical Gradiometer – A magnetometer equipped with two sensors that are vertically separated by a fixed distance. It is best suited to map near surface features and is less susceptible to deep geologic features.

Ground Penetrating Radar – Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture.

4.0 RESPONSIBILITIES

Project Manager (PM)/Task Order Manager (TOM) - Responsible for ensuring that all field activities are conducted in accordance with this procedure.

Site Manager (SM)/Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved SOPs or as otherwise directed by the approved project plan(s).

Site Health & Safety Officer (SHSO) – Responsible to provide technical assistance and verify full compliance with this SOP. The SHSO is also responsible for reporting any deficiencies to the Corporate Health and Safety Manager (HSM) and to the PM/TOM.

Health & Safety Manager (HSM) – Responsible for preparing, implementing, and modifying corporate health and safety policy and this SOP.

Site Personnel – Responsible for performing their work activities in accordance with this SOP and the TtNUS Health and Safety Policy.

5.0 PROCEDURES

This procedure addresses the requirements and technical procedures that must be performed to minimize the potential for contact with underground and overhead utility services. These procedures are addressed individually from a buried and overhead standpoint.

5.1 Buried Utilities

Buried utilities present a heightened concern because their location is not typically obvious by visual observation, and it is common that their presence and/or location is unknown or incorrectly known on client properties. This procedure must be followed prior to beginning any subsurface probing or excavation that might potentially be in the vicinity of underground utility services. In addition, the Utility Clearance Form (Attachment 3) must be completed for every location or cluster of locations where intrusive activities will occur.

Where the positive identification and de-energizing of underground utilities cannot be obtained and confirmed using the following steps, the PM/TOM is responsible for arranging for the procurement of a qualified, experienced, utility locating subcontractor who will accomplish the utility location and demarcation duties specified herein.

1. A comprehensive review must be made of any available property maps, blue lines, or as-builts prior to site activities. Interviews with local personnel familiar with the area should be performed to provide additional information concerning the location of potential underground utilities. Information regarding utility locations shall be added to project maps upon completion of this exercise.

2., A visual site inspection must be performed to compare the site plan information to actual field conditions. Any findings must be documented and the site plan/maps revised. The area(s) of proposed excavation or other subsurface activities must be marked at the site in white paint or pin flags to identify those locations of the proposed intrusive activities. The site inspection should focus on locating surface indications of potential underground utilities. Items of interest include the presence of nearby area lights, telephone service, drainage grates, fire hydrants, electrical service vaults/panels, asphalt/concrete scares and patches, and topographical depressions. Note the location of any emergency shut off switches. Any additional information regarding utility

locations shall be added to project maps upon completion of this exercise and returned to the PM/TOM.

3. If the planned work is to be conducted on private property (e.g., military installations, manufacturing facilities, etc.) the FOL must identify and contact appropriate facility personnel (e.g., public works or facility engineering) before any intrusive work begins to inquire about (and comply with) property owner requirements. It is important to note that private property owners may require several days to several weeks advance notice prior to locating utilities.

4. If the work location is on public property, the state agency that performs utility clearances must be notified (see Attachment 1). State "one-call" services must be notified prior to commencing fieldwork per their requirements. Most one-call services require, by law, 48- to 72-hour advance notice prior to beginning any excavation. Such services typically assign a "ticket" number to the particular site. This ticket number must be recorded for future reference and is valid for a specific period of time, but may be extended by contacting the service again. The utility service will notify utility representatives who then mark their respective lines within the specified time frame. It should be noted that most military installations own their own utilities but may lease service and maintenance from area providers. Given this situation, "one call" systems may still be required to provide location services on military installations.

5. Utilities must be identified and their locations plainly marked using pin flags, spray paint, or other accepted means. The location of all utilities must be noted on a field sketch for future inclusion on project maps. Utility locations are to be identified using the following industry-standard color code scheme, unless the property owner or utility locator service uses a different color code:

white	excavation/subsurface investigation location
red	electrical
yellow	gas, oil, steam
orange	telephone, communications
blue	water, irrigation, slurry
green	sewer, drain

6. Where utility locations are not confirmed with a high degree of confidence through drawings, schematics, location services, etc., the work area must be thoroughly investigated prior to beginning the excavation. In these situations, utilities must be identified using safe and effective methods such as passive and intrusive surveys, or the use of non-conductive hand tools. Also, in situations where such hand tools are used, they should always be used in conjunction with suitable detection equipment, such as the items described in Section 6.0 of this SOP. Each method has advantages and disadvantages including complexity, applicability, and price. It also should be noted that in some states, initial excavation is required by hand to a specified depth.

7. At each location where trenching or excavating will occur using a backhoe or other heavy equipment, and where utility identifications and locations cannot be confirmed prior to groundbreaking, the soil must be probed using a device such as a tile probe which is made of non-conductive material such as fiberglass. If these efforts are not successful in clearing the excavation area of suspect utilities, hand shoveling must be performed for the perimeter of the intended excavation.

8. All utilities uncovered or undermined during excavation must be structurally supported to prevent potential damage. Unless necessary as an emergency corrective measure, TtNUS shall not make any repairs or modifications to existing utility lines without prior permission of the utility owner, property owner, and Corporate HSM. All repairs require that the line be locked-out/tagged-out prior to work.

5.2 Overhead Power Lines

If it is necessary to work within the minimum clearance distance of an overhead power line, the overhead line must be de-energized and grounded, or re-routed by the utility company or a registered electrician. If protective measures such as guarding, isolating, or insulating are provided, these precautions must be adequate to prevent employees from contacting such lines directly with any part of their body or indirectly though conductive materials, tools, or equipment.

The following table provides the required minimum clearances for working in proximity to overhead power lines.

Nominal Voltage	Minimum Clearance
0 -50 kV	10 feet, or one mast length; whichever is greater
50+ kV	10 feet plus 4 inches for every 10 kV over 50 kV or 1.5 mast lengths; whichever is greater

6.0 UNDERGROUND LOCATING TECHNIQUES

A variety of supplemental utility locating approaches are available and can be applied when additional assurance is needed. The selection of the appropriate method(s) to employ is site-specific and should be tailored to the anticipated conditions, site and project constraints, and personnel capabilities.

6.1 Geophysical Methods

Geophysical methods include electromagnetic induction, magnetics, and ground penetrating radar. Additional details concerning the design and implementation of electromagnetic induction, magnetics, and ground penetrating radar surveys can be found in one or more of the TtNUS SOPs included in the References (Section 8.0).

Electromagnetic Induction

Electromagnetic Induction (EMI) line locators operate either by locating a background signal or by locating a signal introduced into the utility line using a transmitter. A utility line acts like a radio antenna, producing electrons, which can be picked up with a radiofrequency receiver. Electrical current carrying conductors have a 60HZ signal associated with them. This signal occurs in all power lines regardless of voltage. Utilities in close proximity to power lines or used as grounds may also have a 60HZ signal, which can be picked up with an EM receiver. A typical example of this type of geophysical equipment is an EM-61.

EMI locators specifically designed for utility locating use a special signal that is either indirectly induced onto a utility line by placing the transmitter above the line or directly induced using an induction clamp. The clamp induces a signal on the specific utility and is the preferred method of tracing since there is little chance of the resulting signals being interfered with. A good example of this type of equipment is the Schonstedt® MAC-51B locator. The MAC-51B performs inductively traced surveys, simple magnetic locating, and traced nonmetallic surveys.

When access can be gained inside a conduit to be traced, a flexible insulated trace wire can be used. This is very useful for non-metallic conduits but is limited by the availability of gaining access inside the pipe.

Magnetics

Magnetic locators operate by detecting the relative amounts of buried ferrous metal. They are incapable of locating or identifying nonferrous utility lines but can be very useful for locating underground storage tanks (UST's), steel utility lines, and buried electrical lines. A typical example of this type of equipment is the Schonstedt® GA-52Cx locator. The GA-52Cx is capable of locating 4-inch steel pipe up to 8 feet deep.

Non-ferrous lines are often located by using a typical plumbing tool (snake) fed through the line. A signal is then introduced to the snake that is then traced.

Ground Penetrating Radar

Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture. In general, an object which is harder than the surrounding soil will reflect a stronger signal. Utilities, tunnels, UST's, and footings will reflect a stronger signal than the surrounding soil. Although this surface detection method may determine the location of a utility, this method does not specifically identify utilities (i.e., water vs. gas, electrical vs. telephone); hence, verification may be necessary using other methods. This method is somewhat limited when used in areas with clay soil types or with a high water table.

6.2 Passive Detection Surveys

Acoustic Surveys

Acoustic location methods are generally most applicable to waterlines or gas lines. A highly sensitive Acoustic Receiver listens for background sounds of water flowing (at joints, leaks, etc.) or to sounds introduced into the water main using a transducer. Acoustics may also be applicable to determine the location of plastic gas lines.

Thermal Imaging

Thermal (i.e., infrared) imaging is a passive method for detecting the heat emitted by an object. Electronics in the infrared camera convert subtle heat differentials into a visual image on the viewfinder or a monitor. The operator does not look for an exact temperature; rather they look for heat anomalies (either elevated or suppressed temperatures) characteristic of a potential utility line.

The thermal fingerprint of underground utilities results from differences in temperature between the atmosphere and the fluid present in a pipe or the heat generated by electrical resistance. In addition, infrared scanners may be capable of detecting differences in the compaction, temperature and moisture content of underground utility trenches. High-performance thermal imagery can detect temperature differences to hundredths of a degree.

6.3 Intrusive Detection Surveys

Vacuum Excavation

Vacuum excavation is used to physically expose utility services. The process involves removing the surface material over approximately a 1' x 1' area at the site location. The air-vacuum process proceeds with the simultaneous action of compressed air-jets to loosen soil and vacuum extraction of the resulting

~~debris. This process ensures the integrity of the utility line during the excavation process, as no hammers, blades, or heavy mechanical equipment comes into contact with the utility line, eliminating the risk of damage to utilities. The process continues until the utility is uncovered. Vacuum excavation can be used at the proposed site location to excavate below the "utility window" which is usually 8 feet.~~

Hand Excavation

When the identification and location of underground utilities cannot be positively confirmed through document reviews and/or other methods, borings and excavations may be cleared via the use of non-conductive hand tools. This should always be done in conjunction with the use of detection equipment. This would be required for all locations where there is a potential to impact buried utilities. The minimum hand-excavation depth that must be reached is to be determined considering the geographical location of the work site. This approach recognizes that the placement of buried utilities is influenced by frost line depths that vary by geographical region. Attachment 2 presents frost line depths for the regions of the contiguous United States. At a minimum, hand excavation depths must be at least to the frost line depth (see Attachment 2) plus two (2) feet, but never less than 4 feet below ground surface (bgs). For hand excavation, the hole created must be reamed large enough to be at least the diameter of the drill rig auger or bit prior to drilling. For soil gas surveys, the survey probe shall be placed as close as possible to the cleared hand excavation. It is important to note that a post-hole digger must not be used in this type of hand excavation activity.

Tile Probe Surveys

For some soil types, site conditions, and excavation requirements, non-conductive tile probes may be used. A tile probe is a "T"-handled rod of varying lengths that can be pushed into the soil to determine if any obstructions exist at that location. Tile probes constructed of fiberglass or other nonconductive material are readily-available from numerous vendors. Tile probes must be performed to the same depth requirements as previously specified. As with other types of hand excavating activities, the use of a non-conductive tile probe, should always be in conjunction with suitable utility locating detection equipment.

7.0 INTRUSIVE ACTIVITIES SUMMARY

The following list summarizes the activities that must be performed prior to beginning subsurface activities:

1. Map and mark all subsurface locations and excavation boundaries using white paint or markers specified by the client or property owner.

2. Notify the property owner and/or client that the locations are marked. At this point, drawings of locations or excavation boundaries shall be provided to the property owner and/or client so they may initiate (if applicable) utility clearance.

   Note: Drawings with confirmed locations should be provided to the property owner and/or client as soon as possible to reduce potential time delays.

3. Notify "One Call" service. If possible, arrange for an appointment to show the One Call representative the surface locations or excavation boundaries in person. This will provide a better location designation to the utilities they represent. You should have additional drawings should you need to provide plot plans to the One Call service.

4. Implement supplemental utility detection techniques as necessary and appropriate to conform utility locations or the absence thereof.

5. Complete Attachment 3, Utility Clearance Form. This form should be completed for each excavation location. In situations where multiple subsurface locations exist within the close proximity of one another, one form may be used for multiple locations provided those locations are noted on the Utility Clearance Form. Upon completion, the Utility Clearance Form and revised/annotated utility location map becomes part of the project file.

8.0 REFERENCES

OSHA Letter of Interpretation, Mr. Joseph Caldwell, Attachment 4
OSHA 29 CFR 1926(b)(2)
OSHA 29 CFR 1926(b)(3)
TtNUS Utility Locating and Clearance Policy
TtNUS SOP GH-3.1; Resistivity and Electromagnetic Induction
TtNUS SOP GH-3.2; Magnetic and Metal Detection Surveys
TtNUS SOP GH-3.4; Ground-penetrating Radar Surveys

ATTACHMENT 1
LISTING OF UNDERGROUND UTILITY CLEARANCE RESOURCES

American Public Works Association
2345 Grand Boulevard, Suite 500, Kansas City, MO 64108-2625
Phone (816) 472-6100 • Fax (816) 472-1610
Web www.apwa.net • E-mail apwa@apwa.net

ONE-CALL SYSTEMS INTERNATIONAL CONDENSED DIRECTORY

Alabama
Alabama One-Call
1-800-292-8525

Alaska
Locate Call Center of Alaska, Inc.
1-800-478-3121

Arizona
Arizona Blue Stake
1-800-782-5348

Arkansas
Arkansas One Call System, Inc.
1-800-482-8998

California
Underground Service Alert North
1-800-227-2600
Underground Service Alert of Southern California
1-800-227-2600

Colorado
Utility Notification Center of Colorado
1-800-922-1987

Connecticut
Call Before You Dig
1-800-922-4455

Delaware
Miss Utility of Delmarva
1-800-282-8555

Florida
Sunshine State One-Call of Florida, Inc.
1-800-432-4770

Georgia
Underground Protection Center, Inc.
1-800-282-7411

Hawaii
Underground Service Alert North
1-800-227-2600

Idaho
Dig Line Inc.
1-800-342-1585
Kootenai County One-Call
1-800-428-4950
Shoshone - Benewah One-Call
1-800-398-3285

Illinois
JULIE, Inc.
1-800-892-0123
Digger (Chicago Utility Alert Network)
312-744-7000

Indiana
Indiana Underground Plant Protection Service
1-800-382-5544

Iowa
Iowa One-Call
1-800-292-8989

Kansas
Kansas One-Call System, Inc.
1-800-344-7233

Kentucky
Kentucky Underground Protection Inc.
1-800-752-6007

Louisiana
Louisiana One Call System, Inc.
1-800-272-3020

Maine
Dig Safe System, Inc.
1-888-344-7233

Maryland
Miss Utility
1-800-257-7777
Miss Utility of Delmarva
1-800-282-8555

Massachusetts
Dig Safe System, Inc.
1-888-344-7233

Michigan
Miss Dig System, Inc.
1-800-482-7171

Minnesota
Gopher State One Call
1-800-252-1166

Mississippi
Mississippi One-Call System, Inc
1-800-227-6477

Missouri
Missouri One-Call System, Inc.
1-800-344-7483

Montana
Utilities Underground Protection Center
1-800-424-5555
Montana One Call Center
1-800-551-8344

Nebraska
Diggers Hotline of Nebraska
1-800-331-5666

Nevada
Underground Service Alert North
1-800-227-2600

New Hampshire
Dig Safe System, Inc.
1-888-344-7233

New Jersey
New Jersey One Call
1-800-272-1000

New Mexico
New Mexico One Call System, Inc.
1-800-321-2537
Las Cruces- Dona Ana Blue Stakes
1-888-526-0400

New York
Dig Safely New York
1-800-962-7962
New York City- Long Island One Call Center
1-800-272-4480

North Carolina
The North Carolina One-Call Center, Inc.
1-800-632-4949

North Dakota
North Dakota One-Call
1-800-795-0555

Ohio
Ohio Utilities Protection Service
1-800-362-2764
Oil & Gas Producers Underground Protect'n Svc
1-800-925-0988

Oklahoma
Call Okie
1-800-522-6543

Oregon
Oregon Utility Notification Center/One Call Concepts
1-800-332-2344

Pennsylvania
Pennsylvania One Call System, Inc.
1-800-242-1776

Rhode Island
Dig Safe System, Inc.
1-888-344-7233

South Carolina
Palmetto Utility Protection Service Inc.
1-888-721-7877

South Dakota
South Dakota One Call
1-800-781-7474

Tennessee
Tennessee One-Call System, Inc.
1-800-351-1111

ATTACHMENT 1 (Continued)

Texas
Texas One Call System
1-800-245-4545
Texas Excavation Safety System, Inc.
1-800-344-8377
Lone Star Notification Center
1-800-669-8344

Utah
Blue Stakes of Utah
1-800-662-4111

Vermont
Dig Safe System, Inc.
1-888-344-7233

Virginia
Miss Utility of Virginia
1-800-552-7001
Miss Utility (Northern Virginia)
1-800-257-7777

Washington
Utilities Underground Location Center
1-800-424-5555
Northwest Utility Notification Center
1-800-553-4344
Inland Empire Utility Coordinating Council
509-456-8000

West Virginia
Miss Utility of West Virginia, Inc.
1-800-245-4848

Wisconsin
Diggers Hotline, Inc.
1-800-242-8511

Wyoming
Wyoming One-Call System, Inc.
1-800-348-1030
Call Before You Dig of Wyoming
1-800-849-2476

District of Columbia
Miss Utility
1-800-257-7777

Alberta
Alberta One-Call Corporation
1-800-242-3447

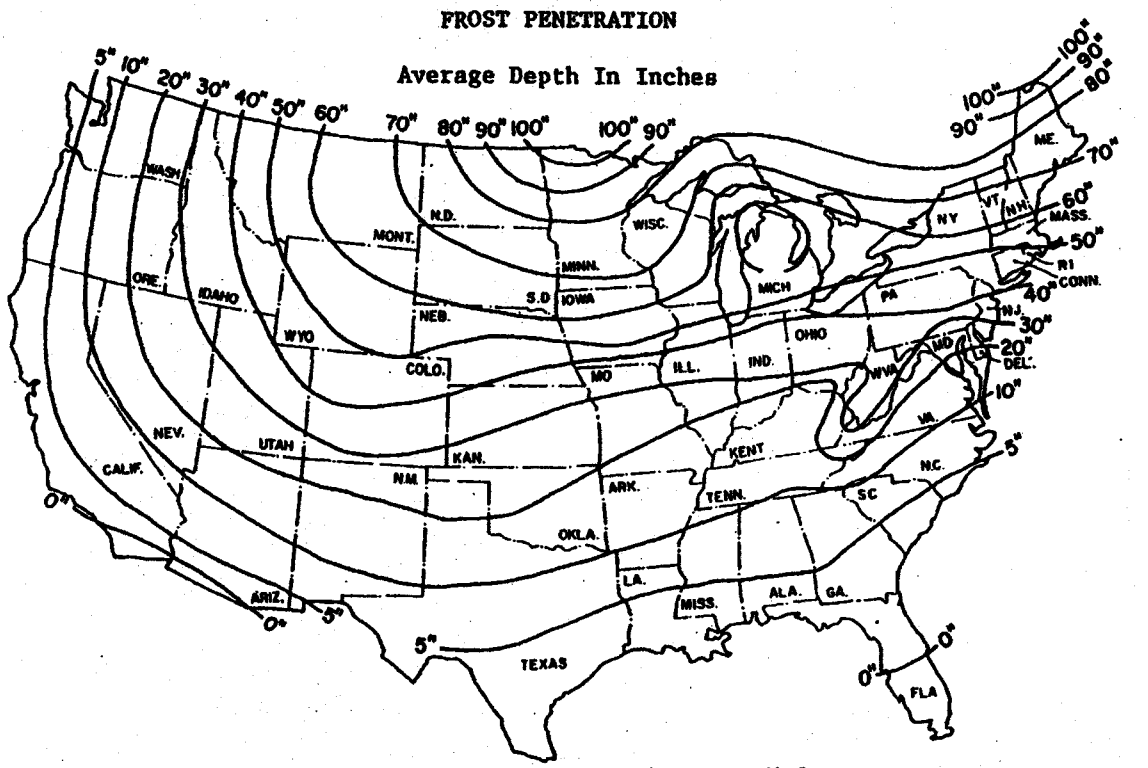
British Columbia
BC One Call
1-800-474-6886

Ontario
Ontario One-Call System
1-800-400-2255

Quebec
Info-Excavation
1-800-663-9228

ATTACHMENT 2

FROST LINE PENETRATION DEPTHS BY GEOGRAPHIC LOCATION



Courtesy U.S. Department Of Commerce

ATTACHMENT 3
UTILITY CLEARANCE FORM

Client: _____ Project Name: _____

Project No.: _____ Completed By: _____

Location Name: _____ Work Date: _____

Excavation Method/Overhead Equipment: _____

1. Underground Utilities — Circle One

a)	Review of existing maps?	yes no N/A
b)	Interview local personnel?	yes no N/A
c)	Site visit and inspection?	yes no N/A
d)	Excavation areas marked in the field?	yes no N/A
e)	Utilities located in the field?	yes no N/A
f)	Located utilities marked/added to site maps?	yes no N/A
g)	Client contact notified Name _____ Telephone: _____ Date: _____	yes no N/A
g)	State One-Call agency called? Caller: _____ Ticket Number: _____ Date: _____	yes no N/A
h)	Geophysical survey performed? Survey performed by: _____ Method: _____ Date: _____	yes no N/A
i)	Hand excavation performed (with concurrent use of utility detection device)? Completed by: _____ Total depth: _____ feet Date: _____	yes no N/A
j)	Trench/excavation probed? Probing completed by: _____ Depth/frequency: _____ Date: _____	yes no N/A

2. Overhead Utilities — Present Absent

a)	Determination of nominal voltage	yes no N/A
b)	Marked on site maps	yes no N/A
c)	Necessary to lockout/insulate/re-route	yes no N/A
d)	Document procedures used to lockout/insulate/re-route	yes no N/A
e)	Minimum acceptable clearance (SOP Section 5.2): _____	

3. Notes:

_____

_____

_____

Approval:

_____ _____

Site Manager/Field Operations Leader Date

c: PM/Project File
Program File

ATTACHMENT 4
OSHA LETTER OF INTERPRETATION

Mr. Joseph Caldwell
Consultant
Governmental Liaison
Pipeline Safety Regulations
211 Wilson Boulevard
Suite 700
Arlington, Virginia 22201

Re: Use of hydro-vacuum or non-conductive hand tools to locate underground utilities.

Dear Mr. Caldwell:

In a letter dated July 7, 2003, we responded to your inquiry of September 18, 2002, regarding the use of hydro-vacuum equipment to locate underground utilities by excavation. After our letter to you was posted on the OSHA website, we received numerous inquiries that make it apparent that aspects of our July 7 letter are being misunderstood. In addition, a number of industry stakeholders, including the National Utility Contractors Association (NUCA), have provided new information regarding equipment that is available for this work.

To clarify these issues, we are withdrawing our July 7 letter and issuing this replacement response to your inquiry.

Question: Section 1926.651 contains several requirements that relate to the safety of employees engaged in excavation work. Specifically, paragraphs (b)(2) and (b)(3) relate in part to the safety of the means used to locate underground utility installations that, if damaged during an uncovering operation, could pose serious hazards to employees.

Under these provisions, what constitutes an acceptable method of uncovering underground utility lines, and further, would the use of hydro-vacuum excavation be acceptable under the standard?

Answer

Background

Two sections of 29 CFR 1926 Subpart P (Excavations), 1926.651(Specific excavation requirements), govern methods for uncovering underground utility installations. Specifically, paragraph (b)(2) states:

> When utility companies or owners cannot respond to a request to locate underground utility installations within 24 hours * * * or cannot establish the exact location of these installations, the employer may proceed, provided the employer does so with caution, and provided detection equipment or other acceptable means to locate utility installations are used. (emphasis added).

Paragraph (b)(3) provides:

ATTACHMENT 4 (Continued)

> When excavation operations approach the estimated location of underground installations, the exact location of the installations shall be determined by safe and acceptable means. (emphasis added).

Therefore, "acceptable means" must be used where the location of the underground utilities have not been identified by the utility companies and detection equipment is not used.

Subpart P does not contain a definition of either "other acceptable means" or "safe and acceptable means." The preambles to both the proposed rule and the final rule discussed the rationale behind the wording at issue. For example, the preamble to the proposed rule, 52 Fed. Reg. 12301 (April 15, 1987), noted that a 1972 version of this standard contained language that specified "careful probing or hand digging" as the means to uncover utilities. The preamble then noted that an amendment to the 1972 standard later deleted that language "to allow other, *equally effective means* of locating such installations." The preamble continued that in the 1987 proposed rule, OSHA again proposed using language in section (b)(3) that would provide another example of an acceptable method of uncovering utilities that could be used *where the utilities have not been marked and detection equipment is not being used* – "probing with hand-held tools." This method was rejected in the final version of 29 CFR 1926. As OSHA explained in the preamble to the final rule, 54 Fed. Reg. 45916 (October 31, 1989):

> OSHA received two comments * * * and input from ACCSH [OSHA's Advisory Committee on Construction Safety and Health] * * * on this provision. All commenters recommended dropping 'such as probing with hand-held tools' from the proposed provision, because this could create a hazard to employees by damaging the installation or its insulation.

In other words, the commenters objected to the use of hand tools being used unless detection equipment was used in conjunction with them. OSHA then concluded its discussion relative to this provision by agreeing with the commentators and ultimately not including any examples of "acceptable means" in the final provision.

Non-conductive hand tools are permitted

This raises the question of whether the standard permits the use of hand tools alone -- without also using detection equipment. NUCA and other industry stakeholders have recently informed us that non-conductive hand tools that are appropriate to be used to locate underground utilities are now commonly available.

Such tools, such as a "shooter" (which has a non-conductive handle and a snub nose) and non-conductive or insulated probes were not discussed in the rulemaking. Since they were not considered at that time, they were not part of the class of equipment that was thought to be unsafe for this purpose. Therefore, we conclude that the use of these types of hand tools, when used with appropriate caution, is an "acceptable means" for locating underground utilities.

ATTACHMENT 4 (Continued)

Hydro-vacuum excavation

It is our understanding that some hydro-vacuum excavation equipment can be adjusted to use a minimum amount of water and suction pressure. When appropriately adjusted so that the equipment will not damage underground utilities (especially utilities that are particularly vulnerable to damage, such as electrical lines), use of such equipment would be considered a "acceptable means" of locating underground utilities. However, if the equipment cannot be sufficiently adjusted, then this method would not be acceptable under the standard.

Other technologies

We are not suggesting that these are the only devices that would be "acceptable means" under the standard. Industry stakeholders have informed us that there are other types of special excavation equipment designed for safely locating utilities as well.

We apologize for any confusion our July 7 letter may have caused. If you have further concerns or questions, please feel free to contact us again by fax at: U.S. Department of Labor, OSHA, Directorate of Construction, Office of Construction Standards and Compliance Assistance, fax # 202-693-1689. You can also contact us by mail at the above office, Room N3468, 200 Constitution Avenue, N.W., Washington, D.C. 20210, although there will be a delay in our receiving correspondence by mail.

Sincerely,

Russell B. Swanson, Director
Directorate of Construction

NOTE: OSHA requirements are set by statute, standards and regulations. Our interpretation letters explain these requirements and how they apply to particular circumstances, but they cannot create additional employer obligations. This letter constitutes OSHA=s interpretation of the requirements discussed. Note that our enforcement guidance may be affected by changes to OSHA rules. Also, from time to time we update our guidance in response to new information. To keep apprised of such developments, you can consult OSHA's website at http://www.osha.gov.

S2 SA-2.5 DIRECT PUSH TECHNOLOGY

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1.0 PURPOSE

The purpose of this procedure is to provide general reference information on Direct Push Technology (DPT). DPT is designed to collect soil, groundwater, and soil gas samples without using conventional drilling techniques. The advantage of using DPT over conventional drilling includes the generation of little or no drill cuttings, sampling in locations with difficult accessibility, reduced overhead clearance requirements, no fluid introduction during probing, and typical lower costs per sample than with conventional techniques. Disadvantages include a maximum penetration depth of approximately 15 to 40 feet in dense soils (although it may be as much as 60 to 80 feet in certain types of geological environments), reduced capability of obtaining accurate water-level measurements, and the inability to install permanent groundwater monitoring wells. The methods and equipment described herein are for collection of surface and subsurface soil samples and groundwater samples. Soil gas sampling is discussed in SOP SA-2.4.

2.0 SCOPE

This procedure provides information on proper sampling equipment and techniques for DPT. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require adjustments in methodology.

3.0 GLOSSARY

Direct Push Technology (DPT) - DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools. A primary advantage of DPT over conventional drilling techniques is that DPT results in the generation of little or no investigation derived waste.

Geoprobe® - Geoprobe® is a manufacturer of a hydraulically-powered, percussion/probing machines utilizing DPT to collect subsurface environmental samples. Geoprobe® relies on a relatively small amount of static weight (vehicle) combined with percussion as the energy for advancement of a tool string. The Geoprobe® equipment can be mounted in a multitude of vehicles for access to all types of environmental sites.

HydroPunch™ - HydroPunch™ is a manufacturer of stainless steel and Teflon® sampling tools that are capable of collecting representative groundwater and/or soil samples without requiring the installation of a groundwater monitoring well or conventional soil boring. HydroPunch™ is an example of DPT sampling equipment.

Flame Ionization Detector (FID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing a flame as the energizing source.

Photo Ionization Detector (PID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing ultraviolet radiation as the energizing source.

4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for selecting and/or reviewing the appropriate DPT drilling procedure required to support the project objectives.

Field Operations Leader (FOL)- The FOL is primarily responsible for performing the DPT in accordance with the project-specific plan.

5.0 SOIL SAMPLING PROCEDURES

5.1 General

The common methodology for the investigation of the vadose zone is soil boring drilling and soil sampling. However, drilling soil borings can be very expensive. Generally the advantage of DPT for subsurface soil sampling is the reduced cost of disposal of drilling cuttings and shorter sampling times.

5.2 Sampling Equipment

Equipment needed for conducting DPT drilling for subsurface soil sampling includes, but is not limited to, the following:

- Geoprobe® Sampling Kit
- Cut-resistant gloves
- 4-foot x 1.5-inch diameter macrocore sampler
- Probe sampling adapters
- Roto-hammer with 1.5-inch bit
- Disposable acetate liners for soil macrocore sampler
- Cast aluminum or steel drive points
- Geoprobe® AT-660 Series Large Bore Soil Sampler, or equivalent
- Standard decontamination equipment and solutions

For health and safety equipment and procedures, follow the direction provided in the Safe Work Permit in Attachment 1, or the more detailed directions provided in the project's Health and Safety Plan.

5.3 DPT Sampling Methodology

There are several methods for the collection of soil samples using DPT drilling. The most common method is discussed in the following section. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project-specific plan.

- Macrocore samplers fitted with detachable aluminum or steel drive points are driven into the ground using hydraulic pressure. If there is concrete or pavement over a sampling location, a Roto-hammer is used to drill a minimum 1.5-inch diameter hole through the surface material. A Roto-hammer may also be used if very dense soils are encountered.

- The sampler is advanced continuously in 4-foot intervals or less if desired. No soil cuttings are generated because the soil which is not collected in the sampler is displaced within the formation.

- The sampler is retracted from the hole, and the 4-foot continuous sample is removed from the outer coring tube. The sample is contained within an inner acetate liner.

- Attach the metal trough from the Geoprobe® Sampling Kit firmly to the tail gate of a vehicle. If a vehicle with a tail gate is not available, secure the trough on another suitable surface.

- Place the acetate liner containing the soils in the trough.

- While wearing cut-resistant gloves (constructed of leather or other suitable material), cut the acetate liner through its entire length using the double-bladed knife that accompanies the Geoprobe® Sampling Kit. Then remove the strip of acetate from the trough to gain access to the collected soils. Do not attempt to cut the acetate liner while holding it in your hand.

- Field screen the sample with an FID or PID, and observe/examine the sample (according to SOP GH-1.3). If appropriate, transfer the sample to sample bottles for laboratory analysis. If additional volume is required, push an additional boring adjacent to the first and composite/mix the same interval. Field compositing is usually not acceptable for sample requiring volatile organics analysis.

- Once sampling has been completed, the hole is backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch is used to cap holes through paved or concrete areas. All holes should be finished smooth to existing grade.

- In the event the direct push van/truck cannot be driven to a remote location or a sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric operated equipment (e.g., jack hammer).

- Sampling equipment is decontaminated prior to collecting the next sample.

6.0 GROUNDWATER SAMPLING PROCEDURES

6.1 General

The most common methodology for the investigation of groundwater is the installation and sampling of permanent monitoring wells. If only groundwater screening is required, the installation and sampling of temporary well points may be performed. The advantage of temporary well point installation using DPT is reduced cost due to no or minimal disposal of drilling cuttings and well construction materials, and shorter installation/times sampling.

Two disadvantages of DPT drilling for well point installation are:

- In aquifers with low yields, well points may have to be sampled without purging or development.
- If volume requirements are high, this method can be time consuming for low yield aquifers.

6.2 Sampling Equipment

Equipment needed for temporary well installation and sampling using DPT includes, but is not limited, to the following:

- 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point
- Connecting rods
- Roto-hammer with 1.5-inch bit
- Mechanical jack
- 1/4-inch OD polyethylene tubing
- 3/8-inch OD polyethylene tubing
- Peristaltic pump
- Standard decontamination equipment and solutions

6.3 DPT Temporary Well Point Installation and Sampling Methodology

There are several methods for the installation and sampling of temporary well points using DPT. The most common methodology is discussed below. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project specific plan.

- A 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point attached to connecting rods is driven into the ground to the desired depth using a rotary electric hammer or other direct push drill rig. If there is concrete or pavement over a sampling location, a Roto-hammer or electric coring machine is used to drill a hole through the surface material.

- The well point will be allowed to equilibrate for at least 15 minutes, after which a measurement of the static water level will be taken. The initial measurement of the water level will be used to assess the amount of water which is present in the well point and to determine the amount of silt and sand infiltration that may have occurred.

- The well point will be developed using a peristaltic pump and polyethylene tubing to remove silt and sand which may have entered the well point. The well point is developed by inserting polyethylene tubing to the bottom of the well point and lifting and lowering the tubing slightly while the pump is operating. The pump will be operated at a maximum rate of approximately 2 liters per minute. After removal of sediment from the bottom of the well point, the well point will be vigorously pumped at maximum capacity until discharge water is visibly clear and no further sediments are being generated. Measurements of pH, specific conductance, temperature, and turbidity shall be recorded every 5 to 10 minutes during the purging process. After two consistent readings of pH, specific conductance, temperature and turbidity (±10 percent), the well may be sampled.

- A sample will be collected using the peristaltic pump set at the same or reduced speed as during well development. Samples (with the exception of the samples to be analyzed for volatile organic compounds, VOCs) will be collected directly from the pump discharge. Sample containers for VOCs will be filled by (first shutting off the pump) crimping the discharge end of the sample tubing when filled, removing the inlet end of the sample tubing from the well, suspending the inlet tubing above the vial, and allowing water to fill each vial by gravity flow.

- Once the groundwater sample has been collected, the connecting rods and well point will be removed from the hole with the direct push rig hydraulics. The hole will be backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch will be used to cap holes through paved or concrete areas. All holes will be finished smooth to existing grade.

- In the event the direct push van/truck cannot be driven to a remote location or sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric-operated equipment (e.g., jack hammer).

- Decontaminate the equipment before moving to the next location.

7.0 RECORDS

A record of all field procedures, tests, and observations must be recorded in the field logbook, boring logs, and sample log sheets, as needed. Entries should include all pertinent data regarding the investigation. The use of sketches and field landmarks will help to supplement the investigation and evaluation.

ATTACHMENT 1
SAFE WORK PERMIT FOR DPT OPERATIONS

Permit No. _____ Date: _____ Time: From _____ to _____

SECTION I: General Job Scope

I. Work limited to the following (description, area, equipment used): **Monitoring well drilling and installation through direct push technology**

II. Required Monitoring Instruments: _____

III. Field Crew: _____

IV. On-site Inspection conducted ☐ Yes ☐ No Initials of Inspector _____ _____ TtNUS

SECTION II: General Safety Requirements (To be filled in by permit issuer)

V. Protective equipment required
Level D ☒ Level B ☐
Level C ☐ Level A ☐
Detailed on Reverse

Respiratory equipment required
Full face APR ☐ Escape Pack ☐
Half face APR ☐ SCBA ☐
SKA-PAC SAR ☐ Bottle Trailer ☐
Skid Rig ☐ None ☒

Level D Minimum Requirements: Sleeved shirt and long pants, safety footwear, and work gloves. Safety glasses, hard hats, and hearing protection will be worn when working near or sampling in the vicinity of the DPT rig.

Modifications/Exceptions.

VI. Chemicals of Concern _____ Action Level(s) _____ Response Measures _____

VII. Additional Safety Equipment/Procedures

Hard-hat	☒ Yes	☐ No	Hearing Protection (Plugs/Muffs)	☒ Yes	☐ No
Safety Glasses	☒ Yes	☐ No	Safety belt/harness	☐ Yes	☒ No
Chemical/splash goggles	☐ Yes	☒ No	Radio	☐ Yes	☒ No
Splash Shield	☐ Yes	☒ No	Barricades	☒ Yes	☐ No
Splash suits/coveralls	☐ Yes	☐ No	Gloves (Type - _____)	☐ Yes	☐ No
Steel toe Work shoes or boots	☒ Yes	☐ No	Work/warming regimen	☐ Yes	☐ No

Modifications/Exceptions: Reflective vests for high traffic areas.

VIII. Procedure review with permit acceptors

	Yes	NA		Yes	NA
Safety shower/eyewash (Location & Use)	☐	☒	Emergency alarms	☐	☐
Daily tail gate meetings	☒	☐	Evacuation routes	☐	☐
Contractor tools/equipment/PPE inspected	☐	☐	Assembly points	☐	☐

IX. Site Preparation

Utility Clearances obtained for areas of subsurface investigation	☐ Yes	☐ No
Physical hazards removed or blockaded	☐ Yes	☐ No
Site control boundaries demarcated/signage	☐ Yes	☐ No

X. Equipment Preparation

	Yes	NA
Equipment drained/depressurized	☐	☒
Equipment purged/cleaned	☐	☒
Isolation checklist completed	☐	☒
Electrical lockout required/field switch tested	☐	☒
Blinds/misalignments/blocks & bleeds in place	☐	☒
Hazardous materials on walls/behind liners considered	☐	☒

XI. Additional Permits required (Hot work, confined space entry). ☐ Yes ☐ No
If yes, complete permit required or contact Health Sciences, Pittsburgh Office

XII. Special instructions, precautions:

_____

Permit Issued by: _____ Permit Accepted by: _____

S3 SA-1.3 SOIL SAMPLING

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1.0 PURPOSE

This procedure discusses the methods used to collect surface, near surface, and subsurface soil samples. Additionally, it describes the method for sampling of test pits and trenches to determine subsurface soil and rock conditions, and recover small-volume or bulk samples.

2.0 SCOPE

This procedure is applicable to the collection of surface, near surface and subsurface soils for laboratory testing, which are exposed through hand digging, hand augering, drilling, or machine excavating at hazardous substance sites.

3.0 GLOSSARY

Composite Sample - A composite sample exists as a combination of more than one sample at various locations and/or depths and times, which is homogenized and treated as one sample. This type of sample is usually collected when determination of an average waste concentration for a specific area is required. Composite samples are not to be collected for volatile organics analysis.

Grab Sample - One sample collected at one location and at one specific time.

Non-Volatile Sample - A non-volatile sample includes all other chemical parameters (e.g., semivolatiles, pesticides/PCBs, metals, etc.) and those engineering parameters that do not require undisturbed soil for their analysis.

Hand Auger - A sampling device used to extract soil from the ground in a relatively undisturbed form.

Thin-Walled Tube Sampler - A thin-walled metal tube (also called a Shelby tube) used to recover relatively undisturbed soil samples. These tubes are available in various sizes, ranging from 2 to 5 inches outside diameter (OD) and from 18 to 54 inches in length.

Split-Barrel Sampler - A steel tube, split in half lengthwise, with the halves held together by threaded collars at either end of the tube. Also called a split-spoon sampler, this device can be driven into resistant materials using a drive weight mounted in the drilling string. A standard split-barrel sampler is typically available in two common lengths, providing either 20-inch or 26-inch longitudinal clearance for obtaining 18-inch or 24-inch-long samples, respectively. These split-barrel samplers commonly range in size from 2-inch OD to 3-1/2 inch OD. The larger sizes are commonly used when a larger volume of sample material is required.

Test Pit and Trench - Open, shallow excavations, typically rectangular (if a test pit) or longitudinal (if a trench), excavated to determine the shallow subsurface conditions for engineering, geological, and soil chemistry exploration and/or sampling purposes. These pits are excavated manually or by machine (e.g., backhoe, clamshell, trencher excavator, or bulldozer).

Confined Space - As stipulated in 29 CFR 1910.146, a confined space means a space that: 1) is large enough and so configured that an employee can bodily enter and perform assigned work; 2) has limited or restricted means for entry or exit (for example tanks, vessels, silos, storage bins, hoppers, vaults, pits, and excavations); and 3) is not designed for continuous employee occupancy. TtNUS considers all confined space as permit-required confined spaces.

4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for determining sampling objectives, as well as, the field procedures used in the collection of soil samples. Additionally, in consultation with other project personnel (geologist, hydrogeologist, etc.), the Project Manager establishes the need for test pits or trenches, and determines their approximate locations and dimensions.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan. This will include (but not be limited to) performing air quality monitoring during sampling, boring and excavation activities, and to ensure that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO/designee may also be required to advise the FOL on other safety-related matters regarding boring, excavation and sampling, such as mitigative measures to address potential hazards from unstable trench walls, puncturing of drums or other hazardous objects, etc.

Field Operations Leader (FOL) - The FOL is responsible for finalizing the location of surface, near surface, and subsurface (hand and machine borings, test pits/trenches) soil samples. He/she is ultimately responsible for the sampling and backfilling of boreholes, test pits and trenches, and for adherence to OSHA regulations during these operations.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of soil samples and the completion of all required paperwork (i.e., sample log sheets, field notebook, boring logs, test pit logs, container labels, custody seals, and chain-of-custody forms).

Competent Person - A Competent Person, as defined in 29 CFR 1929.650 of Subpart P - Excavations, means one who is capable of identifying existing and predictable hazards in the surroundings, or working conditions which are unsanitary, hazardous, or dangerous to employees, and who has authorization to take prompt corrective measures to eliminate them.

5.0 PROCEDURES

5.1 Overview

Soil sampling is an important adjunct to groundwater monitoring. Sampling of the soil horizons above the groundwater table can detect contaminants before they have migrated into the water table, and can establish the amount of contamination sorbed on aquifer solids that have the potential of contributing to groundwater contamination.

Soil types can vary considerably on a hazardous waste site. These variations, along with vegetation, can affect the rate of contaminant migration through the soil. It is important, therefore, that a detailed record be maintained during the sampling operations, particularly noting the location, depth, and such characteristics as grain size, color, and odor. Subsurface conditions are often stable on a daily basis and may demonstrate only slight seasonal variation especially with respect to temperature, available oxygen and light penetration. Changes in any of these conditions can radically alter the rate of chemical reactions or the associated microbiological community, thus further altering specific site conditions. As a result, samples must be kept at their at-depth temperature or lower, protected from direct light, sealed tightly in approved glass containers, and be analyzed as soon as possible.

The physical properties of the soil, its grain size, cohesiveness, associated moisture, and such factors as depth to bedrock and water table, will limit the depth from which samples can be collected and the method required to collect them. Often this information on soil properties can be obtained from published soil surveys available through the U.S. Geological Surveys and other government or farm agencies. It is the

intent of this procedure to present the most commonly employed soil sampling methods used at hazardous waste sites.

5.2 Soil Sample Collection

5.2.1 Procedure for Collecting Soil Samples for Volatile Organic Compounds

The above described traditional sampling techniques, used for the collection of soil samples for volatile organic analysis, have recently been evaluated by the scientific community and determined to be ineffective in producing accurate results (biased low) due to the loss of volatile organics in the sampling stages and microbial degradation of aromatic volatiles. One of the newly adopted sampling procedures for collecting soil samples includes the field preservation of samples with methanol or sodium bisulfate to minimize volatilization and biodegradation. These preservation methods may be performed either in the field or laboratory, depending on the sampling methodology employed.

Soil samples to be preserved by the laboratory are currently being performed using method SW-846, 5035. Laboratories are currently performing low level analyses (sodium bisulfate preservation) and high level analyses (methanol preservation) depending on the end users needs.

It should be noted that a major disadvantage of the methanol preservation method is that the laboratory reporting limits will be higher than conventional testing. The reporting levels using the new method for most analytes are 0.5 µg/g for GC/MS and 0.05 µg/g for GC methods.

The alternative preservation method for collecting soil samples is with sodium bisulfate. This method is more complex to perform in the field and therefore is not preferred for field crews. It should also be noted that currently, not all laboratories have the capabilities to perform this analysis. The advantage to this method is that the reporting limits (0.001 µg/g for GC/PID or GC/ELCD, or 0.010 for GC/MS) are lower than those described above.

The following procedures outline the necessary steps for collecting soil samples to be preserved at the laboratory, and for collecting soil samples to be preserved in the field with methanol or sodium bisulfate.

5.2.1.1 Soil Samples to be Preserved at the Laboratory

Soil samples collected for volatile organics that are to be preserved at the laboratory will be obtained using a hermetically sealed sample vial such as an EnCore™ sampler. Each sample will be obtained using a reusable sampling handle provided with the EnCore™ sampler. The sample is collected by pushing the EnCore™ sampler directly into the soil, ensuring that the sampler is packed tight with soil, leaving zero headspace. Using this type of sampling device eliminates the need for field preservation and the shipping restrictions associated with preservatives. A complete set of instructions is included with each Encore™ sampler shipment by the manufacturer.

Once the sample is collected, it should be placed on ice immediately and shipped to the laboratory within 48 hours (following the chain-of-custody and documentation procedures outlined in SOP SA-6.1). Samples must be preserved by the laboratory within 48 hours of sample collection.

If the lower detection limits are necessary, an option would be to collect several EnCore™ samplers at a given sample location. Send all samplers to the laboratory and the laboratory can perform the required preservation and analyses.

5.2.1.2 Soil Samples to be Preserved in the Field

Soil samples preserved in the field may be prepared for analyses using both the low-level (sodium bisulfate preservation) method and medium-level (methanol preservation) method.

Methanol Preservation (Medium Level):

Soil samples to be preserved in the field with methanol will utilize 40-60 mL glass vials with septum lids. Each sample bottle will be filled with 25 mL of demonstrated analyte-free purge and trap grade methanol. Bottles may be prespiked with methanol in the laboratory or prepared in the field.

Soil will be collected with the use of a decontaminated (or disposable), small-diameter coring device such as a disposable tube/plunger-type syringe with the tip cut off. The outside diameter of the coring device must be smaller than the inside diameter of the sample bottle neck.

A small electronic balance or manual scale will be necessary for measuring the volume of soil to be added to the methanol preserved sample bottle. Calibration of the scale should be performed prior to use and intermittently throughout the day according to the manufacturers requirements.

The sample should be collected by pulling the plunger back and inserting the syringe into the soil to be sampled. The top several inches of soil should be removed before collecting the sample. Approximately 10 grams ±2g (8-12 grams) of soil should be collected. The sample should be weighed and adjusted until obtaining the required amount of sample. The sample weight should be recorded to the nearest 0.01 gram in the field logbook and/or sample log sheet. The soil should then be extruded into the methanol preserved sample bottle taking care not to contact the sample container with the syringe. The threads of the bottle and cap must be free of soil particles.

After capping the bottle, swirl the sample (do not shake) in the methanol and break up the soil such that all of the soil is covered with methanol. Place the sample on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

Sodium Bisulfate Preservation (Low Level):

Samples to be preserved using the sodium bisulfate method are to be prepared as follows:

Add 1 gram of sodium bisulfate to 5 mL of laboratory grade deionized water in a 40-60 mL glass vial with septum lid. Bottles may be prespiked in the laboratory or prepared in the field. The soil sample should be collected in a manner as described above and added to the sample container. The sample should be weighed to the nearest 0.01 gram as described above and recorded in the field logbook or sample log sheet.

Care should be taken when adding the soil to the sodium bisulfate solution. A chemical reaction of soils containing carbonates (limestone) may cause the sample to effervesce or the vial to possibly explode.

When preparing samples using the sodium bisulfate preservation method, duplicate samples must be collected using the methanol preservation method on a one for one sample basis. The reason for this is because it is necessary for the laboratory to perform both the low level and medium level analyses. Place the sample on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

If the lower detection limits are necessary, an option to field preserving with sodium bisulfate would be to collect 3 EnCore™ samplers at a given sample location. Send all samplers to the laboratory and the laboratory can perform the required preservation and analyses.

5.2.2 Procedure for Collecting Non-Volatile Soil Samples

Non-volatile soil samples may be collected as either grab or composite samples. The non-volatile soil sample is thoroughly mixed in a stainless steel or disposable, inert plastic tray, using a stainless steel trowel or other approved tool, then transferred into the appropriate sample container(s). Head space is permitted in a non-volatile soil sample container to allow for sample expansion.

5.2.3 Procedure for Collecting Undisturbed Soil Samples (ASTM D1587-83)

When it is necessary to acquire undisturbed samples of soil for purposes of engineering parameter analysis (e.g., permeability), a thin-walled, seamless tube sampler (Shelby tube) will be employed. The following method will be used:

1. Remove all surface debris (e.g., vegetation, roots, twigs, etc.) from the specific sampling location and drill and clean out the borehole to the sampling depth, being careful to minimize the chance for disturbance of the material to be sampled. In saturated material, withdraw the drill bit slowly to prevent loosening of the soil around the borehole and to maintain the water level in the hole at or above groundwater level.

2. The use of bottom discharge bits or jetting through an open-tube sampler to clean out the borehole shall not be allowed. Use of any side-discharge bits is permitted.

3. A stationary piston-type sampler may be required to limit sample disturbance and aid in retaining the sample. Either the hydraulically operated or control rod activated-type of stationary piston sampler may be used. Prior to inserting the tube sampler into the borehole, check to ensure that the sampler head contains a check valve. The check valve is necessary to keep water in the rods from pushing the sample out the tube sampler during sample withdrawal and to maintain a suction within the tube to help retain the sample.

4. To minimize chemical reaction between the sample and the sampling tube, brass tubes may be required, especially if the tube is stored for an extended time prior to testing. While steel tubes coated with shellac are less expensive than brass, they're more reactive, and shall only be used when the sample will be tested within a few days after sampling or if chemical reaction is not anticipated. With the sampling tube resting on the bottom of the hole and the water level in the boring at groundwater level or above, push the tube into the soil by a continuous and rapid motion, without impacting or twisting. In no case shall the tube be pushed farther than the length provided for the soil sample. Allow about 3 inches in the tube for cuttings and sludge.

5. Upon removal of the sampling tube from the hole, measure the length of sample in the tube and also the length penetrated. Remove disturbed material in the upper end of the tube and measure the length of sample again. After removing at least an inch of soil from the lower end and after inserting an impervious disk, seal both ends of the tube with at least a 1/2-inch thickness of wax applied in a way that will prevent the wax from entering the sample. Clean filler must be placed in voids at either end of the tube prior to sealing with wax. Place plastic caps on the ends of the sample tube, tape the caps in place, and dip the ends in wax.

6. Affix label(s) to the tube as required and record sample number, depth, penetration, and recovery length on the label. Mark the "up" direction on the side of the tube with indelible ink, and mark the end of the sample. Complete Chain-of-Custody (see SOP SA-6.3) and other required forms (including Attachment A of this SOP). Do not allow tubes to freeze, and store the samples vertically with the same orientation they had in the ground, (i.e., top of sample is up) in a cool place out of the sun at all times. Ship samples protected with suitable resilient packing material to reduce shock, vibration, and disturbance.

~~Thin-walled undisturbed tube samplers are restricted in their usage by the consistency of the soil to be sampled. Often, very loose and/or wet samples cannot be retrieved by the samplers, and soils with a consistency in excess of very stiff cannot be penetrated by the sampler. Devices such as Dennison or Pitcher core samplers can be used to obtain undisturbed samples of stiff soils. Using these devices normally increases sampling costs, and therefore their use shall be weighed against the need for acquiring an undisturbed sample.~~

5.3 Surface Soil Sampling

The simplest, most direct method of collecting surface soil samples (most commonly collected to a depth of 6 inches) for subsequent analysis is by use of a stainless steel trowel. Surface soils are considered 0-12 inches bgs.

In general, the following equipment is necessary for obtaining surface soil samples:

- Stainless steel or pre-cleaned disposable trowel.
- Real-time air monitoring instrument (e.g., PID, FID, etc.).
- Latex gloves.
- Required Personal Protective Equipment (PPE).
- Required paperwork (see SOP SA-6.3 and Attachment A of this SOP).
- Required decontamination equipment.
- Required sample container(s).
- Wooden stakes or pin flags.
- Sealable polyethylene bags (i.e., Ziploc® baggies).
- Heavy duty cooler.
- Ice.
- Chain-of-custody records and custody seals.

When acquiring surface soil samples, the following procedure shall be used:

1. Carefully remove vegetation, roots, twigs, litter, etc., to expose an adequate soil surface area to accommodate sample volume requirements.

2. Using a decontaminated stainless steel trowel, follow the procedure cited in Section 5.2.1 for collecting a volatile soil sample. Surface soil samples for volatile organic analysis should be collected from 6-12 inches bgs only.

3. Thoroughly mix (in-situ) a sufficient amount of soil to fill the remaining sample containers and transfer the sample into those containers utilizing the same stainless steel trowel employed above. Cap and securely tighten all sample containers.

4. Affix a sample label to each container. Be sure to fill out each label carefully and clearly, addressing all the categories described in SOP SA-6.3.

5. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

5.4 Near-Surface Soil Sampling

Collection of samples from near the surface (depth of 6-18 inches) can be accomplished with tools such as shovels and stainless steel or pre-cleaned disposable trowels.

The following equipment is necessary to collect near surface soil samples:

- Clean shovel.
- The equipment listed under Section 5.3 of this procedure.
- Hand auger.

To obtain near-surface soil samples, the following protocol shall be observed:

1. With a clean shovel, make a series of vertical cuts to the depth required in the soil to form a square approximately 1 foot by 1 foot.

2. Lever out the formed plug and scrape the bottom of the freshly dug hole with a decontaminated stainless steel or pre-cleaned disposable trowel to remove any loose soil.

3. Follow steps 2 through 5 listed under Section 5.3 of this procedure.

5.5 Subsurface Soil Sampling With a Hand Auger

A hand augering system generally consists of a variety of all stainless steel bucket bits (i.e., cylinders 6-1/2" long, and 2-3/4", 3-1/4", and 4" in diameter), a series of extension rods (available in 2', 3', 4' and 5' lengths), and a cross handle. A larger diameter bucket bit is commonly used to bore a hole to the desired sampling depth and then withdrawn. In turn, the larger diameter bit is replaced with a smaller diameter bit, lowered down the hole, and slowly turned into the soil at the completion depth (approximately 6 inches). The apparatus is then withdrawn and the soil sample collected.

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

To accomplish soil sampling using a hand augering system, the following equipment is required:

- Complete hand auger assembly (variety of bucket bit sizes).
- Stainless steel mixing bowls.
- The equipment listed under Section 5.3 of this procedure.

To obtain soil samples using a hand auger, the following procedure shall be followed:

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

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

3. Begin augering (periodically removing accumulated soils from the bucket bit) and add additional rod extensions as necessary. Also, note (in a field notebook, boring log, and/or on standardized data sheets) any changes in the color, texture or odor of the soil.

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

5. Remove the soiled bucket bit from the rod extension and replace it with another properly decontaminated bucket bit. The bucket bit used for sampling is commonly smaller in diameter than the bucket bit employed to initiate the borehole.

6. Carefully lower the apparatus down the borehole. Care must be taken to avoid scraping the borehole sides.

7. Slowly turn the apparatus until the bucket bit is advanced approximately 6 inches.

8. Discard the top of the core (approximately 1"), which represents any loose material collected by the bucket bit before penetrating the sample material.

9. Fill volatile sample container(s), using a properly decontaminated stainless steel trowel, with sample material directly from the bucket bit. Refer to Section 5.2.1 of this procedure.

10. Utilizing the above trowel, remove the remaining sample material from the bucket bit and place into a properly decontaminated stainless steel mixing bowl and thoroughly homogenize the sample material prior to filling the remaining sample containers. Refer to Section 5.2.2 of this procedure.

11. Follow steps 4 and 5 listed under Section 5.3 of this procedure.

5.6 Subsurface Soil Sampling With a Split-Barrel Sampler (ASTM D1586-84)

Split-barrel (split-spoon) samplers consist of a heavy carbon steel or stainless steel sampling tube that can be split into two equal halves to reveal the soil sample (see Attachment B). A drive head is attached to the upper end of the tube and serves as a point of attachment for the drill rod. A removable tapered nosepiece/drive shoe attaches to the lower end of the tube and facilitates cutting. A basket-like sample retainer can be fitted to the lower end of the split tube to hold loose, dry soil samples in the tube when the sampler is removed from the drill hole. This split-barrel sampler is made to be attached to a drill rod and forced into the ground by means of a 140-lb. or larger casing driver.

Split-barrel samplers are used to collect soil samples from a wide variety of soil types and from depths greater than those attainable with other soil sampling equipment.

The following equipment is used for obtaining split-barrel samples:

- Drilling equipment (provided by subcontractor).
- Split-barrel samplers (O.D. 2 inches, I.D. 1-3/8 inches, either 20 inches or 26 inches long); Larger O.D. samplers are available if a larger volume of sample is needed.
- Drive weight assembly, 140-lb. weight, driving head and guide permitting free fall of 30 inches.
- Stainless steel mixing bowls.
- Equipment listed under Section 5.3 of this procedure.

The following steps shall be followed to obtain split-barrel samples:

1. Remove the drive head and nosepiece, and open the sampler to reveal the soil sample. Immediately scan the sample core with a real-time air monitoring instrument (e.g., FID, PID, etc.). Carefully separate the soil core, with a decontaminated stainless steel knife or trowel, at about 6-inch intervals while scanning the center of the core for elevated readings. Also scan stained soil, soil lenses, and anomalies (if present), and record readings.

2. Collect the volatile sample from the center of the core where elevated readings occurred. If no elevated readings where encountered the sample material should still be collected from the core's

center (this area represents the least disturbed area with minimal atmospheric contact). Refer to Section 5.2.1 of this procedure.

3. Using the same trowel, remove remaining sample material from the split-barrel sampler (except for the small portion of disturbed soil usually found at the top of the core sample) and place the soil into a decontaminated stainless steel mixing bowl. Thoroughly homogenize the sample material prior to filling the remaining sample containers. Refer to Section 5.2.2 of this procedure.

4. Follow steps 4 and 5 listed under Section 5.3 of this procedure.

5.7 Subsurface Sol Sampling Using Direct Push Technology

Subsurface soil samples can be collected to depths of 40+ feet using direct push technology (DPT). DPT equipment, responsibilities, and procedures are described in SOP SA-2.5.

5.8 Excavation and Sampling of Test Pits and Trenches

5.8.1 Applicability

This subsection presents routine test pit or trench excavation techniques and specialized techniques that are applicable under certain conditions.

During the excavation of trenches or pits at hazardous waste sites, several health and safety concerns arise which control the method of excavation. No personnel shall enter any test pit or excavation over 4 feet deep except as a last resort, and then only under direct supervision of a Competent Person (as defined in 29 CFR 1929.650 of Subpart P - Excavations). Whenever possible, all required chemical and lithological samples should be collected using the excavator bucket or other remote sampling apparatus. If entrance is still required, all test pits or excavations must be stabilized by bracing the pit sides using specifically designed wooden or steel support structures. Personnel entering the excavation may be exposed to toxic or explosive gases and oxygen-deficient environments. Any entry may constitute a Confined Space and must be done in conformance with all applicable regulations. In these cases, substantial air monitoring is required before entry, and appropriate respiratory gear and protective clothing is mandatory. There must be at least two persons present at the immediate site before entry by one of the investigators. The reader shall refer to OSHA regulations 29 CFR 1926, 29 CFR 1910.120, 29 CFR 1910.134, and 29 CFR 1910.146.

Excavations are generally not practical where a depth of more than about 15 feet is desired, and they are usually limited to a few feet below the water table. In some cases, a pumping system may be required to control water levels within the pit, providing that pumped water can be adequately stored or disposed. If data on soils at depths greater than 15 feet are required, the data are usually obtained through test borings instead of test pits.

In addition, hazardous wastes may be brought to the surface by excavation equipment. This material, whether removed from the site or returned to the subsurface, must be properly handled according to any and all applicable federal, state, and local regulations.

5.8.2 Test Pit and Trench Excavation

These procedures describe the methods for excavating and logging test pits and trenches excavated to determine subsurface soil and rock conditions. Test pit operations shall be logged and documented (see Attachment C).

Test pits and trenches may be excavated by hand or by power equipment to permit detailed description of the nature and contamination of the in-situ materials. The size of the excavation will depend primarily on the following:

- The purpose and extent of the exploration.
- The space required for efficient excavation.
- The chemicals of concern.
- The economics and efficiency of available equipment.

Test pits normally have a cross section that is 4 to 10 feet square; test trenches are usually 3 to 6 feet wide and may be extended for any length required to reveal conditions along a specific line. The following table, which is based on equipment efficiencies, gives a rough guide for design consideration:

Equipment	Typical Widths, in Feet
Trenching machine	2
Backhoe	2-6
Track dozer	10
Track loader	10
Excavator	10
Scraper	20

The lateral limits of excavation of trenches and the position of test pits shall be carefully marked on area base maps. If precise positioning is required to indicate the location of highly hazardous waste materials, nearby utilities, or dangerous conditions, the limits of the excavation shall be surveyed. Also, if precise determination of the depth of buried materials is needed for design or environmental assessment purposes, the elevation of the ground surface at the test pit or trench location shall also be determined by survey. If the test pit/trench will not be surveyed immediately, it shall be backfilled and its position identified with stakes placed in the ground at the margin of the excavation for later surveying.

The construction of test pits and trenches shall be planned and designed in advance as much as possible. However, field conditions may necessitate revisions to the initial plans. The final depth and construction method shall be determined by the field geologist. The actual layout of each test pit, temporary staging area, and spoils pile will be predicated based on site conditions and wind direction at the time the test pit is made. Prior to excavation, the area can be surveyed by magnetometer or metal detector to identify the presence of underground utilities or drums.

As mentioned previously, no personnel shall enter any test pit or excavation except as a last resort, and then only under direct supervision of a Competent Person. If entrance is still required, Occupational Safety and Health Administration (OSHA) requirements must be met (e.g., walls must be braced with wooden or steel braces, ladders must be in the hole at all times, and a temporary guardrail must be placed along the surface of the hole before entry). It is emphasized that the project data needs should be structured such that required samples can be collected without requiring entrance into the excavation. For example, samples of leachate, groundwater, or sidewall soils can be taken with telescoping poles, etc.

Dewatering may be required to assure the stability of the side walls, to prevent the bottom of the pit from heaving, and to keep the excavation dry. This is an important consideration for excavations in cohesionless material below the groundwater table. Liquids removed as a result of dewatering operations must be handled as potentially contaminated materials. Procedures for the collection and disposal of such materials should be discussed in the site-specific project plans.

5.8.3 Sampling in Test Pits and Trenches

5.8.3.1 General

Test pits and trenches are usually logged as they are excavated. Records of each test pit/trench will be made as presented in Attachment C. These records include plan and profile sketches of the test pit/trench showing materials encountered, their depth and distribution in the pit/trench, and sample locations. These records also include safety and sample screening information.

Entry of test pits by personnel is extremely dangerous, shall be avoided unless absolutely necessary, and can occur only after all applicable Health and Safety and OSHA requirements have been met.

The final depth and type of samples obtained from each test pit will be determined at the time the test pit is excavated. Sufficient samples are usually obtained and analyzed to quantify contaminant distribution as a function of depth for each test pit. Additional samples of each waste phase and any fluids encountered in each test pit may also be collected.

In some cases, samples of soil may be extracted from the test pit for reasons other than waste sampling and chemical analysis, for instance, to obtain geotechnical information. Such information would include soil types, stratigraphy, strength, etc., and could therefore entail the collection of disturbed (grab or bulk) or relatively undisturbed (hand-carved or pushed/driven) samples, which can be tested for geotechnical properties. The purposes of such explorations are very similar to those of shallow exploratory or test borings, but often test pits offer a faster, more cost-effective method of sampling than installing borings.

5.8.3.2 Sampling Equipment

The following equipment is needed for obtaining samples for chemical or geotechnical analysis from test pits and trenches:

- Backhoe or other excavating machinery.
- Shovels, picks, hand augers, and stainless steel trowels/disposable trowels.
- Sample container - bucket with locking lid for large samples; appropriate bottleware for chemical or geotechnical analysis samples.
- Polyethylene bags for enclosing sample containers; buckets.
- Remote sampler consisting of 10-foot sections of steel conduit (1-inch-diameter), hose clamps and right angle adapter for conduit (see Attachment D).

5.8.3.3 Sampling Methods

The methods discussed in this section refer to test pit sampling from grade level. If test pit entry is required, see Section 5.8.3.4.

- Excavate trench or pit in several depth increments. After each increment, the operator will wait while the sampler inspects the test pit from grade level to decide if conditions are appropriate for sampling. (Monitoring of volatiles by the SSO will also be used to evaluate the need for sampling.) Practical depth increments range from 2 to 4 feet.

- The backhoe operator, who will have the best view of the test pit, will immediately cease digging if:

  - Any fluid phase or groundwater seepage is encountered in the test pit.
  - Any drums, other potential waste containers, obstructions or utility lines are encountered.
  - Distinct changes of material are encountered.

This action is necessary to permit proper sampling of the test pit and to prevent a breach of safety protocol. Depending upon the conditions encountered, it may be required to excavate more slowly and carefully with the backhoe.

For obtaining test pit samples from grade level, the following procedure shall be followed:

- Remove loose material to the greatest extent possible with backhoe.

- Secure walls of pit if necessary. (There is seldom any need to enter a pit or trench which would justify the expense of shoring the walls. All observations and samples should be taken from the ground surface.)

- Samples of the test pit material are to be obtained either directly from the backhoe bucket or from the material once it has been deposited on the ground. The sampler or Field Operations Leader directs the backhoe operator to remove material from the selected depth or location within the test pit/trench. The bucket is brought to the surface and moved away from the pit. The sampler and/or SSO then approaches the bucket and monitors its contents with a photoionization or flame ionization detector. The sample is collected from the center of the bucket or pile and placed in sample containers using a decontaminated stainless steel trowel or disposable spatula.

- If a composite sample is desired, several depths or locations within the pit/trench are selected and a bucket is filled from each area. It is preferable to send individual sample bottles filled from each bucket to the laboratory for compositing under the more controlled laboratory conditions. However, if compositing in the field is required, each sample container shall be filled from materials that have been transferred into a mixing bucket and homogenized. Note that homogenization/compositing is not applicable for samples to be subjected to volatile organic analysis.

- Using the remote sampler shown in Attachment D, samples can be taken at the desired depth from the side wall or bottom of the pit. The face of the pit/trench shall first be scraped (using a long-handled shovel or hoe) to remove the smeared zone that has contacted the backhoe bucket. The sample shall then be collected directly into the sample jar, by scraping with the jar edge, eliminating the need to utilize samplers and minimizing the likelihood of cross-contamination. The sample jar is then capped, removed from the assembly, and packaged for shipment.

- Complete documentation as described in SOP SA-6.3 and Attachment C of this SOP.

5.8.3.4 <u>In-Pit Sampling</u>

Under rare conditions, personnel may be required to enter the test pit/trench. This is necessary only when soil conditions preclude obtaining suitable samples from the backhoe bucket (e.g., excessive mixing of soils or wastes within the test pit/trench) or when samples from relatively small discrete zones within the test pit are required. This approach may also be necessary to sample any seepage occurring at discrete levels or zones in the test pit that are not accessible with remote samplers.

In general, personnel shall sample and log pits and trenches from the ground surface, except as provided for by the following criteria:

- There is no practical alternative means of obtaining such data.
- The Site Safety Officer and Competent Person determines that such action can be accomplished without breaching site safety protocol. This determination will be based on actual monitoring of the pit/trench after it is dug (including, at a minimum, measurements of volatile organics, explosive gases and available oxygen).
- A Company-designated Competent Person determines that the pit/trench is stable or is made stable (by grading the sidewalls or using shoring) prior to entrance of any personnel. OSHA requirements must be strictly observed.

If these conditions are satisfied, one person will enter the pit/trench. On potentially hazardous waste sites, this individual will be dressed in safety gear as required by the conditions in the pit. He/she will be affixed to a safety rope and continuously monitored while in the pit.

A second individual will be fully dressed in protective clothing including a self-contained breathing device and on standby during all pit entry operations. The individual entering the pit will remain therein for as brief a period as practical, commensurate with performance of his/her work. After removing the smeared zone, samples shall be obtained with a decontaminated trowel or spoon. As an added precaution, it is advisable to keep the backhoe bucket in the test pit when personnel are working below grade. Such personnel can either stand in or near the bucket while performing sample operations. In the event of a cave-in they can either be lifted clear in the bucket, or at least climb up on the backhoe arm to reach safety.

5.8.3.5 Geotechnical Sampling

In addition to the equipment described in Section 5.8.3.2, the following equipment is needed for geotechnical sampling:

- Soil sampling equipment, similar to that used in shallow drilled boring (i.e., open tube samplers), which can be pushed or driven into the floor of the test pit.
- Suitable driving (i.e., a sledge hammer) or pushing (i.e., the backhoe bucket) equipment which is used to advance the sampler into the soil.
- Knives, spatulas, and other suitable devices for trimming hand-carved samples.
- Suitable containers (bags, jars, tubes, boxes, etc.), labels, wax, etc. for holding and safely transporting collected soil samples.
- Geotechnical equipment (pocket penetrometer, torvane, etc.) for field testing collected soil samples for classification and strength properties.

Disturbed grab or bulk geotechnical soil samples may be collected for most soils in the same manner as comparable soil samples for chemical analysis. These collected samples may be stored in jars or plastic-lined sacks (larger samples), which will preserve their moisture content. Smaller samples of this type are usually tested for their index properties to aid in soil identification and classification, while larger bulk samples are usually required to perform compaction tests.

Relatively undisturbed samples are usually extracted in cohesive soils using open tube samplers, and such samples are then tested in a geotechnical laboratory for their strength, permeability and/or compressibility. The techniques for extracting and preserving such samples are similar to those used in performing Shelby tube sampling in borings, except that the sampler is advanced by hand or backhoe,

rather than by a drill rig. Also, the sampler may be extracted from the test pit by excavation around the sampler when it is difficult to pull it out of the ground. If this excavation requires entry of the test pit, the requirements described in Section 5.8.3.4 of this procedure must be followed. The open tube sampler shall be pushed or driven vertically into the floor or steps excavated in the test pit at the desired sampling elevations. Extracting tube samples horizontally from the walls of the test pit is not appropriate, because the sample will not have the correct orientation.

A sledge hammer or the backhoe may be used to drive or push the sampler or tube into the ground. Place a piece of wood over the top of the sampler or sampling tube to prevent damage during driving/pushing of the sample. Pushing the sampler with a constant thrust is always preferable to driving it with repeated blows, thus minimizing disturbance to the sample. If the sample cannot be extracted by rotating it at least two revolutions (to shear off the sample at the bottom), hand-excavate to remove the soil from around the sides of the sampler. If hand-excavation requires entry of the test pit, the requirements in Section 5.8.3.4 of this procedure must be followed. Prepare, label, pack and transport the sample in the required manner, as described in SOP SA-6.3 and SA-6.1.

5.8.4 Backfilling of Trenches and Test Pits

All test pits and excavations must be either backfilled, covered, or otherwise protected at the end of each day. No excavations shall remain open during non-working hours unless adequately covered or otherwise protected.

Before backfilling, the onsite crew shall photograph all significant features exposed by the test pit and trench and shall include in the photograph a scale to show dimensions. Photographs of test pits shall be marked to include site number, test pit number, depth, description of feature, and date of photograph. In addition, a geologic description of each photograph shall be entered in the site logbook. All photographs shall be indexed and maintained as part of the project file for future reference.

After inspection, backfill material shall be returned to the pit under the direction of the FOL.

If a low permeability layer is penetrated (resulting in groundwater flow from an upper contaminated flow zone into a lower uncontaminated flow zone), backfill material must represent original conditions or be impermeable. Backfill could consist of a soil-bentonite mix prepared in a proportion specified by the FOL (representing a permeability equal to or less than original conditions). Backfill can be covered by "clean" soil and graded to the original land contour. Revegetation of the disturbed area may also be required.

5.9 Records

The appropriate sample log sheet (see Attachment A of this SOP) must be completed by the site geologist/sampler. All soil sampling locations should be documented by tying in the location of two or more nearby permanent landmarks (building, telephone pole, fence, etc.) or obtaining GPS coordinates; and shall be noted on the appropriate sample log sheet, site map, or field notebook. Surveying may also be necessary, depending on the project requirements.

Test pit logs (see Attachment C of this SOP) shall contain a sketch of pit conditions. In addition, at least one photograph with a scale for comparison shall be taken of each pit. Included in the photograph shall be a card showing the test pit number. Boreholes, test pits and trenches shall be logged by the field geologist in accordance with SOP GH-1.5.

Other data to be recorded in the field logbook include the following:

- Name and location of job.
- Date of boring and excavation.

- Approximate surface elevation.
- Total depth of boring and excavation.
- Dimensions of pit.
- Method of sample acquisition.
- Type and size of samples.
- Soil and rock descriptions.
- Photographs.
- Groundwater levels.
- Organic gas or methane levels.
- Other pertinent information, such as waste material encountered.

6.0 REFERENCES

American Society for Testing and Materials, 1987. ASTM Standards D1587-83 and D1586-84. ASTM Annual Book of Standards. ASTM. Philadelphia, Pennsylvania. Volume 4.08.

NUS Corporation, 1986. Hazardous Material Handling Training Manual.

NUS Corporation and CH2M Hill, August, 1987. Compendium of Field Operation Methods. Prepared for the U.S. EPA.

OSHA, Excavation, Trenching and Shoring 29 CFR 1926.650-653.

OSHA, Confined Space Entry 29 CFR 1910.146.

ATTACHMENT A
SOIL & SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

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

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

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

GRAB SAMPLE DATA

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

COMPOSITE SAMPLE DATA

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

SAMPLE COLLECTION INFORMATION

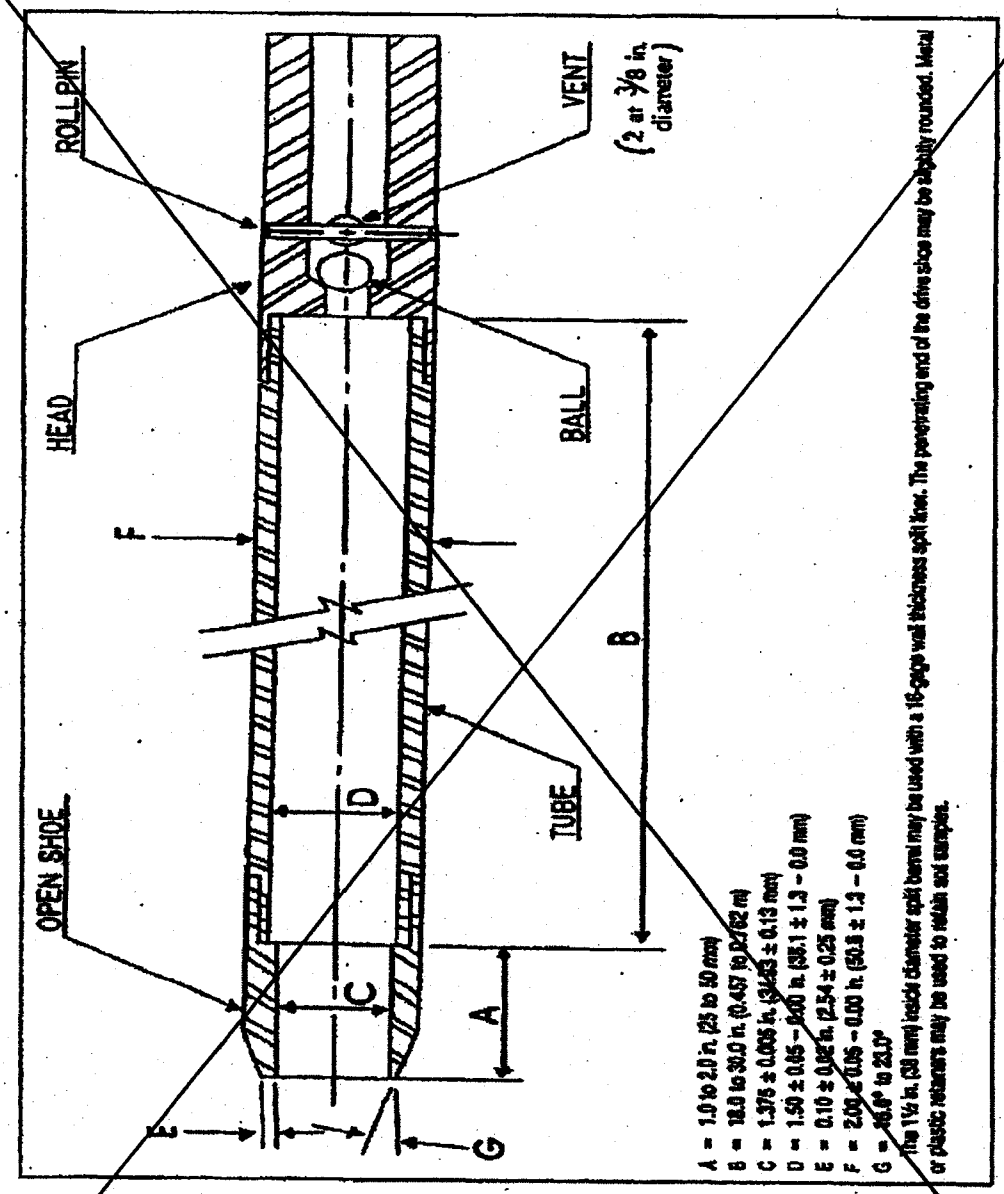
Analysis	Container Requirements	Collected	Other

OBSERVATIONS/NOTES

QA/QC INFORMATION

MS/MSD	Duplicate ID No.:	Signature(s):

ATTACHMENT B
SPLIT-SPOON SAMPLER



A = 1.0 to 2.0 in. (25 to 50 mm)
B = 18.0 to 30.0 in. (0.457 to 0.762 m)
C = 1.375 ± 0.005 in. (34.93 ± 0.13 mm)
D = 1.50 ± 0.05 – 0.00 in. (38.1 ± 1.3 – 0.0 mm)
E = 0.10 ± 0.02 in. (2.54 ± 0.25 mm)
F = 2.00 ± 0.05 – 0.00 in. (50.8 ± 1.3 – 0.0 mm)
G = 45.0° to 23.0°

The 1½ in. (38 mm) inside diameter split barrel may be used with a 16-gage wall thickness split liner. The penetrating end of the drive shoe may be slightly rounded. Metal or plastic retainers may be used to retain soil samples.

ATTACHMENT C
TEST PIT LOG

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TEST PIT LOG

Page ___ of ___

PROJECT NAME: _____ TEST PIT No.: _____
PROJECT NUMBER: _____ DATE: _____
LOCATION: _____ GEOLOGIST: _____

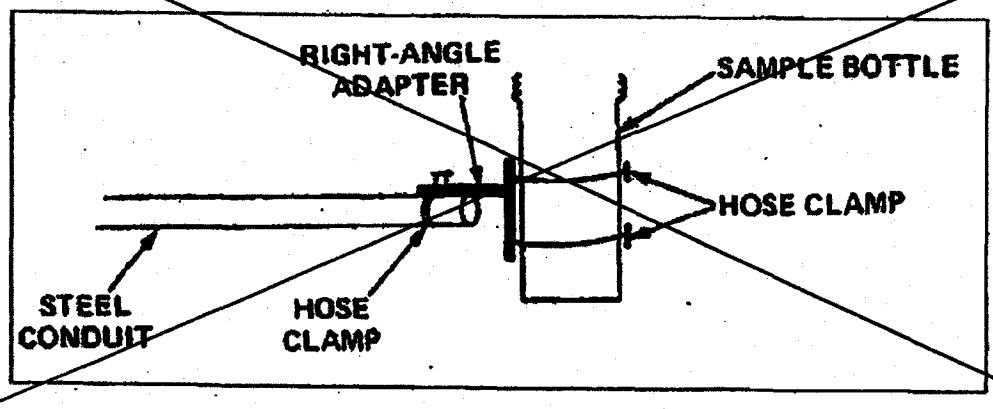
Depth (Ft.)	Lithology Change (Depth/Ft.)	MATERIAL DESCRIPTION Soil/waste characteristics (lithology, density, color, etc.)	USCS	Remarks	PID/FID READING Background	PID/FID READING Reading

TEST PIT CROSS SECTION AND / OR PLAN VIEW

REMARKS: _____

PHOTO LOG: _____

ATTACHMENT D
REMOTE SAMPLE HOLDER FOR TEST PIT/TRENCH SAMPLING



S4 GH-1.5 BOREHOLE AND SAMPLE LOGGING

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1.0 PURPOSE

The purpose of this document is to establish standard procedures and technical guidance on borehole and sample logging.

2.0 SCOPE

These procedures provide descriptions of the standard techniques for borehole and sample logging. These techniques shall be used for each boring logged to provide consistent descriptions of subsurface lithology. While experience is the only method to develop confidence and accuracy in the description of soil and rock, the field geologist/engineer can do a good job of classification by careful, thoughtful observation and by being consistent throughout the classification procedure.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Site Geologist. Responsible for supervising all boring activities and assuring that each borehole is completely logged. If more than one rig is being used on site, the Site Geologist must make sure that each field geologist is properly trained in logging procedures. A brief review or training session may be necessary prior to the start up of the field program and/or upon completion of the first boring.

5.0 PROCEDURES

The classification of soil and rocks is one of the most important jobs of the field geologist/engineer. To maintain a consistent flow of information, it is imperative that the field geologist/engineer understand and accurately use the field classification system described in this SOP. This identification is based on visual examination and manual tests.

5.1 Materials Needed

When logging soil and rock samples, the geologist or engineer may be equipped with the following:

- Rock hammer
- Knife
- Camera
- Dilute hydrochloric acid (HCl)
- Ruler (marked in tenths and hundredths of feet)
- Hand Lens

5.2 Classification of Soils

All data shall be written directly on the boring log (Figure 1) or in a field notebook if more space is needed. Details on filling out the boring log are discussed in Section 5.5.

FIGURE 1

BORING LOG (EXAMPLE)

BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING NUMBER: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	Color	Material Classification	USCS *	Remarks	PID/FID Reading (ppm): Sample	Sampler BZ	Borehole**	Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 5 foot intervals @ borehole. Increase reading frequency if elevated response read.

Remarks: _____

Drilling Area Background (ppm): [ ]

Converted to Well: Yes _____ No _____ Well I.D. #: _____

FIGURE 1 (CONTINUED)

SOIL TERMS

UNIFIED SOIL CLASSIFICATION (USCS)

COARSE-GRAINED SOILS More Than Half of Material is LARGER Than No. 200 Sieve Size					FINE-GRAINED SOILS More Than Half of Material is SMALLER Than No. 200 Sieve Size						
FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights)			GROUP SYMBOL	TYPICAL NAMES	FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights)					GROUP SYMBOL	TYPICAL NAMES
					Identification Procedures on Fraction Smaller Than No. 40 Sieve Size						
						DRY STRENGTH (Crushing Characteristics)	DILATANCY (Reaction to Shaking)	TOUGHNESS (Consistency Near Plastic Limit)			
GRAVELS (More Than Half of Coarse Fraction Is Larger Than No. 4 Sieve Size)	CLEAN GRAVELS (Less Than 5% Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	GW	Well graded gravels, gravel-sand mixtures, little or no fines.	SILTS AND CLAYS Liquid Limit Less Than 50	None to Slight	Quick to Slow	None		ML	Inorganic silts and very fine sands, rock flour, silty or clayey fine sands with slight plasticity.
		Predominately one size or a range of sizes with some intermediate sizes missing.	GP	Poorly graded gravels, gravel-sand mixtures, little or no fines.		Medium to High	None to Very Slow	Medium		CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays.
	GRAVELS WITH FINES (More Than 12% Fines)	Nonplastic fines (for identification procedures, see ML)	GM	Silty gravels, poorly graded gravel-sand-silt mixtures.		Slight to Medium	Slow	Slight		OL	Organic silts and organic silty clays of low plasticity.
		Plastic fines (for identification procedures, see CL)	GC	Clayey gravels, poorly graded gravel-sand-clay mixtures.	SILTS AND CLAYS Liquid Limit Greater Than 50	Slight to Medium	Slow to None	Slight to Medium		MH	Inorganic silts, micaceous or diatomaceous fine sands or silty soils, elastic silts.
SANDS (More Than Half of Coarse Fraction Is Smaller Than No. 4 Sieve Size)	CLEAN SANDS (Less Than 5% Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	SW	Well graded sands, gravelly sands, little or no fines.		High to Very High	None	High		CH	Inorganic clays of high plasticity, fat clays.
		Predominately one size or a range of sizes with some intermediate sizes missing.	SP	Poorly graded sands, gravelly sands, little or no fines.		Medium to High	None to Very Slow	Slight to Medium		OH	Organic clays of medium to high plasticity.
	SANDS WITH FINES (More Than 12% Fines)	Nonplastic fines (for identification procedures, see ML)	SM	Silty sands, poorly graded sand-silt mixtures.	HIGHLY ORGANIC SOILS	Readily identified by color, odor, spongy feel and frequently by fibrous texture.				Pt	Peat and other highly organic soils
		Plastic fines (for identification procedures, see CL)	SC	Clayey sands, poorly graded sand-clay mixtures.							

Boundary classifications: Soils possessing characteristics of two groups are designated by combining group symbols. For example, GW-GC, well graded gravel-sand mixture with clay binder.
All sieve sizes on this chart are U.S. Standard.

DENSITY OF GRANULAR SOILS

DESIGNATION	STANDARD PENETRATION RESISTANCE - BLOWS/FOOT
Very Loose	0-4
Loose	5-9
Medium Dense	10-29
Dense	30-49
Very Dense	Over 50

CONSISTENCY OF COHESIVE SOILS

CONSISTENCY	UNCONFINED STRENGTH (TONS/SQ.FT.)	STANDARD PENETRATION RESISTANCE - BLOWS/FOOT	FIELD IDENTIFICATION METHOD
Very Soft	Less than 0.25	0 to 1	Easily penetrated several inches by fist
Soft	0.25 to 0.5	2 to 4	Easily penetrated several inches by thumb.
Medium Stiff	0.5 to 1.0	5 to 8	Can be penetrated several inches by thumb.
Stiff	1.0 to 2.0	9 to 15	Readily indented by thumb.
Very Stiff	2.0 to 4.0	16 to 30	Readily indented by thumbnail.
Hard	More than 4.0	Over 30	Indented with difficulty by thumbnail.

ROCK TERMS

ROCK HARDNESS (FROM CORE SAMPLES)

Penetration Terms	Scratching and Effects	Hammer Effects
Soft	Easily chipped	Crushes when struck with hammer
Medium Soft	Can be chipped	Breakages along clearly edges
Medium Hard	Can be scratched	Breakages along sharp edges
Hard	Cannot be scratched	Breakages only beneath hammer along sharp edges

ROCK BROKENNESS

Description Terms	Abbreviation	Spacing
Very Broken	(V.B)	< 1"
Broken	(Br)	1"-3"
Blocky	(Bl)	3"-9"
Massive	(M)	> 9"

LEGEND :

SOIL SAMPLER - TYPES

2-4" Undisturbed Sample

0.75" O.D. Undisturbed Sample

0 - Other Sampling Equipment Or Methods

ROCK SAMPLER - TYPES

X-NQ (Diamond Core, 0.75" O.D.)

0-NQ (Diamond Core, 1.875" O.D.)

Z - Other Core Barrel, Specify Information

OTHER USES

S/P

S/P

Y.S.C. Water Level where a Depth

Y.S.C. Structural, and where a Depth

5.2.1 USCS Classification

Soils are to be classified according to the Unified Soil Classification System (USCS). This method of classification is detailed in Figure 1 (Continued).
This method of classification identifies soil types on the basis of grain size and cohesiveness.

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

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

5.2.2 Color

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

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

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

Soil Color Charts shall not be used unless specified by the project manager.

5.2.3 Relative Density and Consistency

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

The density of noncohesive, granular soils is classified according to standard penetration resistances obtained from split-barrel sampling performed according to the methods detailed in Standard Operating Procedures GH-1.3 and SA-1.3. Those designations are:

Designation	Standard Penetration Resistance (Blows per Foot)
Very loose	0 to 4
Loose	5 to 10
Medium dense	11 to 30
Dense	31 to 50
Very dense	Over 50

Standard penetration resistance is the number of blows required to drive a split-barrel sampler with a 2-inch outside diameter 12 inches into the material using a 140-pound hammer falling freely through 30 inches. The sampler is driven through an 18-inch sample interval, and the number of blows is recorded for each 6-inch increment. The density designation of granular soils is obtained by adding the number of blows required to penetrate the last 12 inches of each sample interval. It is important to note that if gravel or rock fragments are broken by the sampler or if rock fragments are lodged in the tip, the resulting blow count will be erroneously high, reflecting a higher density than actually exists. This shall be noted on the log and referenced to the sample number. Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

The consistency of cohesive soils is determined by performing field tests and identifying the consistency as shown in Figure 2.

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

The consistency of cohesive soils is determined either by blow counts, a pocket penetrometer (values listed in the table as Unconfined Compressive Strength), or by hand by determining the resistance to penetration by the thumb. The pocket penetrometer and thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample in the split-barrel sampler. The sample shall be broken in half and the thumb or penetrometer pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. Consistency shall not be determined solely by blow counts. One of the other methods shall be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in Figure 2.

5.2.4 Weight Percentages

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

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

FIGURE 2

CONSISTENCY FOR COHESIVE SOILS

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

Examples:

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

5.2.5 Moisture

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

Laboratory tests for water content shall be performed if the natural water content is important.

5.2.6 Stratification

Stratification can only be determined after the sample barrel is opened. The stratification or bedding thickness for soil and rock is depending on grain size and composition. The classification to be used for stratification description is shown in Figure 3.

5.2.7 Texture/Fabric/Bedding

The texture/fabric/bedding of the soil shall be described. Texture is described as the relative angularity of the particles: rounded, subrounded, subangular, and angular. Fabric shall be noted as to whether the particles are flat or bulky and whether there is a particular relation between particles (i.e., all the flat particles are parallel or there is some cementation). The bedding or structure shall also be noted (e.g., stratified, lensed, nonstratified, heterogeneous varved).

5.2.8 Summary of Soil Classification

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

- Density and/or consistency
- Color
- Plasticity (Optional)
- Soil types
- Moisture content
- Stratification
- Texture, fabric, bedding
- Other distinguishing features

FIGURE 3

BEDDING THICKNESS CLASSIFICATION

Thickness (metric)	Thickness (Approximate English Equivalent)	Classification
> 1.0 meter	> 3.3'	Massive
30 cm - 1 meter	1.0' - 3.3'	Thick Bedded
10 cm - 30 cm	4" - 1.0'	Medium Bedded
3 cm - 10 cm	1" - 4"	Thin Bedded
1 cm - 3 cm	2/5" - 1"	Very Thin Bedded
3 mm - 1 cm	1/8" - 2/5"	Laminated
1 mm - 3 mm	1/32" - 1/8"	Thinly Laminated
< 1 mm	<1/32"	Micro Laminated

(Weir, 1973 and Ingram, 1954)

5.3 Classification of Rocks

Rocks are grouped into three main divisions: sedimentary, igneous and metamorphic. Sedimentary rocks are by far the predominant type exposed at the earth's surface. The following basic names are applied to the types of rocks found in sedimentary sequences:

- Sandstone - Made up predominantly of granular materials ranging between 1/16 to 2 mm in diameter.
- Siltstone - Made up of granular materials less than 1/16 to 1/256 mm in diameter. Fractures irregularly. Medium thick to thick bedded.
- Claystone - Very fine-grained rock made up of clay and silt-size materials. Fractures irregularly. Very smooth to touch. Generally has irregularly spaced pitting on surface of drilled cores.
- Shale - A fissile very fine-grained rock. Fractures along bedding planes.
- Limestone - Rock made up predominantly of calcite ($CaCO_3$). Effervesces strongly upon the application of dilute hydrochloric acid.
- Coal - Rock consisting mainly of organic remains.
- Others - Numerous other sedimentary rock types are present in lesser amounts in the stratigraphic record. The local abundance of any of these rock types is dependent upon the depositional history of the area. Conglomerate, halite, gypsum, dolomite, anhydrite, lignite, etc. are some of the rock types found in lesser amounts.

In classifying a sedimentary rock the following hierarchy shall be noted:

- Rock type
- Color
- Bedding thickness
- Hardness
- Fracturing
- Weathering
- Other characteristics

5.3.1 Rock Type

As described above, there are numerous types of sedimentary rocks. In most cases, a rock will be a combination of several grain types, therefore, a modifier such as a sandy siltstone, or a silty sandstone can be used. The modifier indicates that a significant portion of the rock type is composed of the modifier. Other modifiers can include carbonaceous, calcareous, siliceous, etc.

Grain size is the basis for the classification of clastic sedimentary rocks. Figure 4 is the Udden-Wentworth classification that will be assigned to sedimentary rocks. The individual boundaries are slightly different than the USCS subdivision for soil classification. For field determination of grain sizes, a scale can be used for the coarse grained rocks. For example, the division between siltstone and claystone may not be measurable in the field. The boundary shall be determined by use of a hand lens. If the grains cannot be seen with the naked eye but are distinguishable with a hand lens, the rock is a siltstone. If the grains are not distinguishable with a hand lens, the rock is a claystone.

FIGURE 4

GRAIN SIZE CLASSIFICATION FOR ROCKS

Particle Name	Grain Size Diameter
Cobbles	> 64 mm
Pebbles	4 - 64 mm
Granules	2 - 4 mm
Very Coarse Sand	1 - 2 mm
Coarse Sand	0.5 - 1 mm
Medium Sand	0.25 - 0.5 mm
Fine Sand	0.125 - 0.25 mm
Very Fine Sand	0.0625 - 0.125 mm
Silt	0.0039 - 0.0625 mm

After Wentworth, 1922

5.3.2 Color

The color of a rock can be determined in a similar manner as for soil samples. Rock core samples shall be classified while wet, when possible, and air cored samples shall be scraped clean of cuttings prior to color classifications.

Rock color charts shall not be used unless specified by the Project Manager.

5.3.3 Bedding Thickness

The bedding thickness designations applied to soil classification (see Figure 3) will also be used for rock classification.

5.3.4 Hardness

The hardness of a rock is a function of the compaction, cementation, and mineralogical composition of the rock. A relative scale for sedimentary rock hardness is as follows:

- Soft - Weathered, considerable erosion of core, easily gouged by screwdriver, scratched by fingernail. Soft rock crushes or deforms under pressure of a pressed hammer. This term is always used for the hardness of the saprolite (decomposed rock which occupies the zone between the lowest soil horizon and firm bedrock).
- Medium soft - Slight erosion of core, slightly gouged by screwdriver, or breaks with crumbly edges from single hammer blow.
- Medium hard - No core erosion, easily scratched by screwdriver, or breaks with sharp edges from single hammer blow.
- Hard - Requires several hammer blows to break and has sharp conchoidal breaks. Cannot be scratched with screwdriver.

Note the difference in usage here of the works "scratch" and "gouge." A scratch shall be considered a slight depression in the rock (do not mistake the scraping off of rock flour from drilling with a scratch in the rock itself), while a gouge is much deeper.

5.3.5 Fracturing

The degree of fracturing or brokenness of a rock is described by measuring the fractures or joint spacing. After eliminating drilling breaks, the average spacing is calculated and the fracturing is described by the following terms:

- Very broken (V. BR.) - Less than 2-inch spacing between fractures
- Broken (BR.) - 2-inch to 1-foot spacing between fractures
- Blocky (BL.) - 1- to 3-foot spacing between fractures
- Massive (M.) - 3 to 10-foot spacing between fractures

The structural integrity of the rock can be approximated by calculating the Rock Quality Designation (RQD) of cores recovered. The RQD is determined by adding the total lengths of all pieces exceeding 4 inches and dividing by the total length of the coring run, to obtain a percentage.

Method of Calculating RQD
(After Deere, 1964)

$$RQD\ \% = r/l \times 100$$

r = Total length of all pieces of the lithologic unit being measured, which are greater than 4 inches length, and have resulted from natural breaks. Natural breaks include slickensides, joints, compaction slicks, bedding plane partings (not caused by drilling), friable zones, etc.

l = Total length of the coring run.

5.3.6 Weathering

The degree of weathering is a significant parameter that is important in determining weathering profiles and is also useful in engineering designs. The following terms can be applied to distinguish the degree of weathering:

- Fresh - Rock shows little or no weathering effect. Fractures or joints have little or no staining and rock has a bright appearance.
- Slight - Rock has some staining which may penetrate several centimeters into the rock. Clay filling of joints may occur. Feldspar grains may show some alteration.
- Moderate - Most of the rock, with exception of quartz grains, is stained. Rock is weakened due to weathering and can be easily broken with hammer.
- Severe - All rock including quartz grains is stained. Some of the rock is weathered to the extent of becoming a soil. Rock is very weak.

5.3.7 Other Characteristics

The following items shall be included in the rock description:

- Description of contact between two rock units. These can be sharp or gradational.
- Stratification (parallel, cross stratified).
- Description of any filled cavities or vugs.
- Cementation (calcareous, siliceous, hematitic).
- Description of any joints or open fractures.
- Observation of the presence of fossils.
- Notation of joints with depth, approximate angle to horizontal, any mineral filling or coating, and degree of weathering.

All information shown on the boring logs shall be neat to the point where it can be reproduced on a copy machine for report presentation. The data shall be kept current to provide control of the drilling program and to indicate various areas requiring special consideration and sampling.

5.3.8 Additional Terms Used in the Description of Rock

The following terms are used to further identify rocks:

- Seam - Thin (12 inches or less), probably continuous layer.
- Some - Indicates significant (15 to 40 percent) amounts of the accessory material. For example, rock composed of seams of sandstone (70 percent) and shale (30 percent) would be "sandstone -- some shale seams."
- Few - Indicates insignificant (0 to 15 percent) amounts of the accessory material. For example, rock composed of seam of sandstone (90 percent) and shale (10 percent) would be "sandstone -- few shale seams."
- Interbedded - Used to indicate thin or very thin alternating seams of material occurring in approximately equal amounts. For example, rock composed of thin alternating seams of sandstone (50 percent) and shale (50 percent) would be "interbedded sandstone and shale."
- Interlayered - Used to indicate thick alternating seams of material occurring in approximately equal amounts.

The preceding sections describe the classification of sedimentary rocks. The following are some basic names that are applied to igneous rocks:

- Basalt - A fine-grained extrusive rock composed primarily of calcic plagioclase and pyroxene.
- Rhyolite - A fine-grained volcanic rock containing abundant quartz and orthoclase. The fine-grained equivalent of a granite.
- Granite - A coarse-grained plutonic rock consisting essentially of alkali feldspar and quartz.
- Diorite - A coarse-grained plutonic rock consisting essentially of sodic plagioclase and hornblende.
- Gabbro - A coarse-grained plutonic rock consisting of calcic plagioclase and clinopyroxene. Loosely used for any coarse-grained dark igneous rock.

The following are some basic names that are applied to metamorphic rocks:

- Slate - A very fine-grained foliated rock possessing a well developed slaty cleavage. Contains predominantly chlorite, mica, quartz, and sericite.
- Phyllite - A fine-grained foliated rock that splits into thin flaky sheets with a silky sheen on cleavage surface.
- Schist - A medium to coarse-grained foliated rock with subparallel arrangement of the micaceous minerals which dominate its composition.
- Gneiss - A coarse-grained foliated rock with bands rich in granular and platy minerals.
- Quartzite - A fine- to coarse-grained nonfoliated rock breaking across grains, consisting essentially of quartz sand with silica cement.

5.4 Abbreviations

Abbreviations may be used in the description of a rock or soil. However, they shall be kept at a minimum. Following are some of the abbreviations that may be used:

C	-	Coarse	Lt	-	Light	Yl	-	Yellow
Med	-	Medium	BR	-	Broken	Or	-	Orange
F	-	Fine	BL	-	Blocky	SS	-	Sandstone
V	-	Very	M	-	Massive	Sh	-	Shale
Sl	-	Slight	Br	-	Brown	LS	-	Limestone
Occ	-	Occasional	Bl	-	Black	Fgr	-	Fine-grained
Tr	-	Trace						

5.5 Boring Logs and Documentation

This section describes in more detail the procedures to be used in completing boring logs in the field. Information obtained from the preceding sections shall be used to complete the logs. A sample boring log has been provided as Figure 5.

The field geologist/engineer shall use this example as a guide in completing each boring log. Each boring log shall be fully described by the geologist/engineer as the boring is being drilled. Every sheet contains space for 25 feet of log. Information regarding classification details is provided either on the back of the boring log or on a separate sheet, for field use.

5.5.1 Soil Classification

- Identify site name, boring number, job number, etc. Elevations and water level data to be entered when surveyed data is available.

- Enter sample number (from SPT) under appropriate column. Enter depth sample was taken from (1 block = 1 foot). Fractional footages, i.e., change of lithology at 13.7 feet, shall be lined off at the proportional location between the 13- and 14-foot marks. Enter blow counts (Standard Penetration Resistance) diagonally (as shown). Standard penetration resistance is covered in Section 5.2.3.

- Determine sample recovery/sample length as shown. Measure the total length of sample recovered from the split-spoon sampler, including material in the drive shoe. Do not include cuttings or wash material that may be in the upper portion of the sample tube.

- Indicate any change in lithology by drawing a line at the appropriate depth. For example, if clayey silt was encountered from 0 to 5.5 feet and shale from 5.5 to 6.0 feet, a line shall be drawn at this increment. This information is helpful in the construction of cross-sections. As an alternative, symbols may be used to identify each change in lithology.

- The density of granular soils is obtained by adding the number of blows for the last two increments. Refer to Density of Granular Soils Chart on back of log sheet. For consistency of cohesive soils refer also to the back of log sheet - Consistency of Cohesive Soils. Enter this information under the appropriate column. Refer to Section 5.2.3.

FIGURE 5
COMPLETED BORING LOG (EXAMPLE)

BORING LOG

Page 1 of 1

PROJECT NAME: NSB- SITE
PROJECT NUMBER: 9594
DRILLING COMPANY: SOILTEST CO.
DRILLING RIG: CME-55

BORING NUMBER: SB/MW 1
DATE: 3/8/96
GEOLOGIST: SJ CONTI
DRILLER: R. ROCK

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	Soil Density/ Consistency or Rock Hardness	Color	Material Classification	USCS*	Remarks	PID/FID Reading (ppm) Sample	Sampler BZ	Borehole**	Driller BZ**
S-1 @ 0800	0.0	7/6	1.5/2.0		M DENSE	BRN TO BLK	SILTY SAND - SOME	SM	MOIST SL. ORG. ODOR	5	0	0	0
	2.0	9/10					ROCK FR - TR BRICKS (FILL)		FILL TO 4'±				
	4.0			4.0									
S-2 @ 0810	6.0	5/7 9/8	2.0/2.0		M DENSE	BRN	SILTY SAND - TR FINE GRAVEL	SM	MOIST - W ODOR NAT. MATL. TOOK SAMPLE SB01-0406 FOR ANALYSIS	10	0	-	-
	• 8.0			7' ▽ 8.0									
S-3 @ 0820	10.0	6/8 17/16	1.9/2.0		DENSE	TAN BRN	FINE TO COARSE SAND TR. F. GRAVEL	SW	WET HIT WATER ≈ 7'±	0	0	0	0
	12.0			12.0									
S-4 @ 0830	14.0	7/6 5/8	1.6/2.0		STIFF	GRAY	SILTY CLAY	CL	MOIST → WET	0	.5	-	-
	15.0			15.0					AUGER REF @ 15'				
9/5	①		4.0/5.0	LIKELIHOOD 16	M HARD	BRN	SILTSTONE	VBR	WEATHERED LO % JNTS @ 15.5 WATER STAINS @ 16.5, 17.1, 17.5	0	0	0	0
				19'					LOSING SOME				
	20.0				HARD	GRAY	SANDSTONE - SOME SILTSTONE	BR	DRILL H2O @ 17'± SET TEMP 6" CAS TO 15.5				
4.8/5.0	②		5.0/5.0						SET 2" Φ PVC SCREEN 16-25	0	0	0	0
									SAND 14-25				
	25.0			25					PELLETS 12-14				

* When rock coring, enter rock brokeness.
** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.

• 1-20Z
1-80Z

Drilling Area Background (ppm): 0

Remarks: CME-55 R16, 4 1/4" ID HSA - 9" OD ±
2" SPLIT SPOONS - 140 LB HAMMER - 30" DROP
NX CORE IN BEDROCK RUN ① = 25 min. RUN ② = 15 min

Converted to Well: Yes ✓ No ____ Well I.D. #: MW-1

- Enter color of the material in the appropriate column.

- Describe material using the USCS. Limit this column for sample description only. The predominant material is described last. If the primary soil is silt but has fines (clay) - use clayey silt. Limit soil descriptors to the following:

  - Trace: 0 - 10 percent
  - Some: 11 - 30 percent
  - And/Or: 31 - 50 percent

- Also indicate under Material Classification if the material is fill or natural soils. Indicate roots, organic material, etc.

- Enter USCS symbol - use chart on back of boring log as a guide. If the soils fall into one of two basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example ML/CL or SM/SP.

- The following information shall be entered under the "Remarks" column and shall include, but is not limited by, the following:

  - Moisture - estimate moisture content using the following terms - dry, moist, wet and saturated. These terms are determined by the individual. Whatever method is used to determine moisture, be consistent throughout the log.

  - Angularity - describe angularity of coarse grained particles using the terms angular, subangular, subrounded, or rounded. Refer to ASTM D 2488 or Earth Manual for criteria for these terms.

  - Particle shape - flat, elongated, or flat and elongated.

  - Maximum particle size or dimension.

  - Water level observations.

  - Reaction with HCl - none, weak, or strong.

- Additional comments:

  - Indicate presence of mica, caving of hole, when water was encountered, difficulty in drilling, loss or gain of water.

  - Indicate odor and Photoionization Detector (PID) or Flame Ionization Detector (FID) reading if applicable.

  - Indicate any change in lithology by drawing a line through the lithology change column and indicate the depth. This will help when cross-sections are subsequently constructed.

  - At the bottom of the page indicate type of rig, drilling method, hammer size and drop, and any other useful information (i.e., borehole size, casing set, changes in drilling method).

- Vertical lines shall be drawn (as shown in Figure 5) in columns 6 to 8 from the bottom of each sample to the top of the next sample to indicate consistency of material from sample to sample, if the material is consistent. Horizontal lines shall be drawn if there is a change in lithology, then vertical lines drawn to that point.

- Indicate screened interval of well, as needed, in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.2 Rock Classification

- Indicate depth at which coring began by drawing a line at the appropriate depth. Indicate core run depths by drawing coring run lines (as shown) under the first and fourth columns on the log sheet. Indicate RQD, core run number, RQD percent, and core recovery under the appropriate columns.

- Indicate lithology change by drawing a line at the appropriate depth as explained in Section 5.5.1.

- Rock hardness is entered under designated column using terms as described on the back of the log or as explained earlier in this section.

- Enter color as determined while the core sample is wet; if the sample is cored by air, the core shall be scraped clean prior to describing color.

- Enter rock type based on sedimentary, igneous or metamorphic. For sedimentary rocks use terms as described in Section 5.3. Again, be consistent in classification. Use modifiers and additional terms as needed. For igneous and metamorphic rock types use terms as described in Sections 5.3.8.

- Enter brokenness of rock or degree of fracturing under the appropriate column using symbols VBR, BR, BL, or M as explained in Section 5.3.5 and as noted on the back of the Boring Log.

- The following information shall be entered under the remarks column. Items shall include but are not limited to the following:

  - Indicate depths of joints, fractures and breaks and also approximate to horizontal angle (such as high, low), i.e., 70° angle from horizontal, high angle.
  - Indicate calcareous zones, description of any cavities or vugs.
  - Indicate any loss or gain of drill water.
  - Indicate drop of drill tools or change in color of drill water.

- Remarks at the bottom of Boring Log shall include:

  - Type and size of core obtained.
  - Depth casing was set.
  - Type of rig used.

- As a final check the boring log shall include the following:

  - Vertical lines shall be drawn as explained for soil classification to indicate consistency of bedrock material.

  - If applicable, indicate screened interval in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.3 Classification of Soil and Rock from Drill Cuttings

The previous sections describe procedures for classifying soil and rock samples when cores are obtained. However, some drilling methods (air/mud rotary) may require classification and borehole logging based on identifying drill cuttings removed from the borehole. Such cuttings provide only general information on subsurface lithology. Some procedures that shall be followed when logging cuttings are:

- Obtain cutting samples at approximately 5-foot intervals, sieve the cuttings (if mud rotary drilling) to obtain a cleaner sample, place the sample into a small sample bottle or "zip lock" bag for future reference, and label the jar or bag (i.e. hole number, depth, date, etc.). Cuttings shall be closely examined to determine general lithology.
- Note any change in color of drilling fluid or cuttings, to estimate changes in lithology.
- Note drop or chattering of drilling tools or a change in the rate of drilling, to determine fracture locations or lithologic changes.
- Observe loss or gain of drilling fluids or air (if air rotary methods are used), to identify potential fracture zones.
- Record this and any other useful information onto the boring log as provided in Figure 1.

This logging provides a general description of subsurface lithology and adequate information can be obtained through careful observation of the drilling process. It is recommended that split-barrel and rock core sampling methods be used at selected boring locations during the field investigation to provide detailed information to supplement the less detailed data generated through borings drilled using air/mud rotary methods.

5.6 Review

Upon completion of the borings logs, copies shall be made and reviewed. Items to be reviewed include:

- Checking for consistency of all logs.
- Checking for conformance to the guideline.
- Checking to see that all information is entered in their respective columns and spaces.

6.0 REFERENCES

Unified Soil Classification System (USCS).

ASTM D2488, 1985.

Earth Manual, U.S. Department of the Interior, 1974.

7.0 RECORDS

Originals of the boring logs shall be retained in the project files.

S5 SA-1-1 GROUNDWATER SAMPLE ACQUISITION AND ON-SITE WATER QUALITY TESTING

TETRA TECH NUS, INC.	STANDARD OPERATING PROCEDURES	Number SA-1-1	Page 1 of 25
		Effective Date 09/03	Revision 5
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING		Approved D. Senovich	

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B GROUNDWATER SAMPLE LOG SHEET 24
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1.0 PURPOSE

The purpose of this procedure is to provide general reference information regarding the sampling of groundwater wells.

2.0 SCOPE

This procedure provides information on proper sampling equipment, onsite water quality testing, and techniques for groundwater sampling. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require modifications to methodology.

3.0 GLOSSARY

Conductivity – Conductivity is a numerical expression of the ability of an aqueous solution to carry an electric current. This ability depends on the presence of ions, their total concentration, mobility, valence, and relative concentrations, and on temperature of measure. Conductivity is highly dependent on temperature and should be reported at a particular temperature, i.e., 20.2 mS/cm at 14°C.

Dissolved Oxygen (DO) – DO levels in natural and wastewater depend on the physical, chemical, and biochemical activities in the water sample.

Oxidation-Reduction Potential (ORP) - A measure of the activity ratio of oxidizing and reducing species as determined by the electromotive force developed by a noble metal electrode, immersed in water, as referenced against a standard hydrogen electrode.

pH - The negative logarithm (base 10) of the hydrogen ion activity. The hydrogen ion activity is related to the hydrogen ion concentration, and, in a relatively weak solution, the two are nearly equal. Thus, for all practical purposes, pH is a measure of the hydrogen ion concentration.

pH Paper - Indicator paper that turns different colors depending on the pH of the solution to which it is exposed. Comparison with color standards supplied by the manufacturer will then give an indication of the solution's pH.

Salinity – The measurement of dissolved salts in a given mass of solution. Note: most field meters determined salinity automatically from conductivity and temperature. The displayed value will be displayed in either parts per thousand (ppt) or % (e.g., 35 ppt will equal 3.5%).

Turbidity – Turbidity in water is caused by suspended matter, such as clay, silt, fine organic and inorganic matter. Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample.

4.0 RESPONSIBILITIES

Project Hydrogeologist - Responsible for selecting and detailing the specific groundwater sampling techniques, onsite water quality testing (type, frequency, and location), and equipment to be used, and providing detailed input in this regard to the project plan documents. The project hydrogeologist is also responsible for properly briefing and overseeing the performance of the site sampling personnel.

Project Geologist/Field Sample Technician - is primarily responsible for the proper acquisition of the groundwater samples. He/she is also responsible for the actual analyses of onsite water quality samples, as well as instrument calibration, care, and maintenance. When appropriate, such responsibilities may be performed by other qualified personnel (e.g., field technicians).

5.0 PROCEDURES

5.1 General

To be useful and accurate, a groundwater sample must be representative of the particular zone of the water being sampled. The physical, chemical, and bacteriological integrity of the sample must be maintained from the time of sampling to the time of analysis in order to keep any changes in water quality parameters to a minimum.

Methods for withdrawing samples from completed wells include the use of pumps, compressed air, bailers, and various types of samplers. The primary considerations in obtaining a representative sample of the groundwater are to avoid collection of stagnant (standing) water in the well and to avoid physical or chemical alteration of the water due to sampling techniques. In a non-pumping well, there will be little or no vertical mixing of water in the well pipe or casing, and stratification will occur. The well water in the screened section will mix with the groundwater due to normal flow patterns, but the well water above the screened section will remain isolated and become stagnant. To safeguard against collecting non-representative stagnant water in a sample, the following approach shall be followed prior to sample acquisition:

1. All monitoring wells shall be purged prior to obtaining a sample. Evacuation of three to five volumes is recommended prior to sampling. In a high-yielding groundwater formation and where there is no stagnant water in the well above the screened section, extensive evacuation prior to sample withdrawal is not as critical.

2. For wells that can be purged dry, the well shall be evacuated and allowed to recover to 75% full capacity prior to sample acquisition. If the recovery rate is fairly rapid, evacuation of more than one volume of water is required.

3. For high-yielding monitoring wells which cannot be evacuated to dryness, there is no absolute safeguard against contaminating the sample with stagnant water. One of the following techniques shall be used to minimize this possibility:

   - A submersible pump or the intake line of a surface pump or bailer shall be placed just below the water surface when removing the stagnant water and lowered as the water level drops. Three to five volumes of water shall be removed to provide reasonable assurance that all stagnant water has been evacuated. Once this is accomplished, a bailer or other approved device may be used to collect the sample for analysis.

   - The intake line of the sampling pump (or the submersible pump itself) unless otherwise directed shall be placed near the center of the screened section, and approximately one casing volume of water shall be pumped from the well at a low purge rate, equal to the well's recovery rate (low flow sampling).

Stratification of contaminants may exist in the aquifer. Concentration gradients as a result of mixing and dispersion processes, layers of variable permeability, and the presence of separate-phase product (i.e.,

floating hydrocarbons) may cause stratification. Excessive pumping or improper sampling methods can dilute or increase the contaminant concentrations in the recovered sample compared to what is representative of the integrated water column as it naturally occurs at that point, thus the result is the collection of a non-representative sample.

5.2 Sampling, Monitoring, and Evacuation Equipment

Sample containers shall conform with the guidelines expressed in SOP SA-6.1.

The following equipment shall be on hand when sampling groundwater wells (reference SOPs SA-6.1 and SA-7.1):

- Sample packaging and shipping equipment - Coolers for sample shipping and cooling, chemical preservatives, appropriate sampling containers and filler, ice, labels and chain-of-custody documents.
- Field tools and instrumentation - Multi-parameters water quality meter capable of measuring ORP, pH, temperature, DO, specific conductance, turbidity and salinity or individual meters (as applicable), pH paper, camera and film (if appropriate), appropriate keys (for locked wells), water level indicator.
- Pumps
  - Shallow-well pumps: Centrifugal, bladder, suction, or peristaltic pumps with droplines, air-lift apparatus (compressor and tubing) where applicable.
  - Deep-well pumps: Submersible pump and electrical power-generating unit, or bladder pumps where applicable.
- Other sampling equipment - Bailers and inert line with tripod-pulley assembly (if necessary).
- Pails - Plastic, graduated.
- Decontamination solutions - Deionized water, potable water, laboratory detergents, 10% nitric acid solution (as required), and analytical-grade solvent (e.g., pesticide-grade isopropanol), as required.

Ideally, sample withdrawal equipment shall be completely inert, economical, easily cleaned, cleaned prior to use, reusable, able to operate at remote sites in the absence of power sources, and capable of delivering variable rates for well purging and sample collection.

5.3 Calculations of Well Volume

To insure that the proper volume of water has been removed from the well prior to sampling it is first necessary to know the volume of standing water in the well pipe. This volume can be easily calculated by the following method. Calculations shall be entered in the site logbook or field notebook or on a sample log sheet form (see SOP SA-6.3):

- Obtain all available information on well construction (location, casing, screens, etc.).
- Determine well or inner casing diameter.
- Measure and record static water level (depth below ground level or top of casing reference point).
- Determine depth of well by sounding using a clean, decontaminated, weighted tape measure.

- Calculate number of linear feet of static water (total depth or length of well pipe minus the depth to static water level).

- Calculate one static well volume in gallons $V = (0.163)(T)(r^2)1$

where:	V	=	Static volume of well in gallons.
	T=		Thickness of water table in the well measured in feet (i.e., linear feet of static water).
	r	=	Inside radius of well casing in inches.
	0.163	=	A constant conversion factor which compensates for the conversion of the casing radius from inches to feet, the conversion of cubic feet to gallons, and pi.

- Per evacuation volumes discussed above, determine the minimum amount to be evacuated before sampling.

5.4 Evacuation of Static Water (Purging)

5.4.1 General

The amount of purging a well shall receive prior to sample collection will depend on the intent of the monitoring program and the hydrogeologic conditions. Programs to determine overall quality of water resources may require long pumping periods to obtain a sample that is representative of a large volume of that aquifer. The pumped volume may be specified prior to sampling so that the sample can be a composite of a known volume of the aquifer. Alternately the well can be pumped until the parameters such as temperature, specific conductance, pH, and turbidity (as applicable), have stabilized. Onsite measurements of these parameters shall be recorded in the site logbook, field notebook, or on standardized data sheets.

5.4.2 Evacuation Devices

The following discussion is limited to those devices commonly used at hazardous waste sites. Attachment A provides guidance on the proper evacuation device to use for given sampling situations. Note that all of these techniques involve equipment which is portable and readily available.

~~Bailers~~

~~Bailers are the simplest evacuation devices used and have many advantages. They generally consist of a length of pipe with a sealed bottom (bucket-type bailer) or, as is more useful and favored, with a ball check-valve at the bottom. An inert line is used to lower the bailer and retrieve the sample.~~

~~Advantages of bailers include:~~

- ~~Few limitations on size and materials used for bailers.~~
- ~~No external power source needed.~~
- ~~Bailers are inexpensive, and can be dedicated and hung in a well to reduce the chances of cross-contamination.~~
- ~~Bailers are relatively easy to decontaminate.~~

~~Limitations on the use of bailers include the following:~~

- It is time consuming to remove stagnant water using a bailer.
- Transfer of sample may cause aeration.
- Use of bailers is physically demanding, especially in warm temperatures at protection levels above Level D.

Suction Pumps

There are many different types of inexpensive suction pumps including centrifugal, diaphragm, and peristaltic pumps. Centrifugal and diaphragm pumps can be used for well evacuation at a fast pumping rate and for sampling at a low pumping rate. The peristaltic pump is a low volume pump that uses rollers to squeeze a flexible tubing, thereby creating suction. This tubing can be dedicated to a well to prevent cross contamination.

These pumps are all portable, inexpensive and readily available. However, because they are based on suction, their use is restricted to areas with water levels within 20 to 25 feet of the ground surface. A significant limitation is that the vacuum created by these pumps can cause significant loss of dissolved gases and volatile organics.

Air-Lift Samplers

This group of pump samplers uses gas pressure either in the annulus of the well or in a venturi to force the water up a sampling tube. These pumps are also relatively inexpensive. Air (or gas)-lift samplers are more suitable for well development than for sampling because the samples may be aerated, leading to pH changes and subsequent trace metal precipitation, or loss of volatile organics.

Submersible Pumps

Submersible pumps take in water and push the sample up a sample tube to the surface. The power sources for these samplers may be compressed gas or electricity. The operation principles vary and the displacement of the sample can be by an inflatable bladder, sliding piston, gas bubble, or impeller. Pumps are available for 2-inch-diameter wells and larger. These pumps can lift water from considerable depths (several hundred feet).

Limitations of this class of pumps include:

- They may have low delivery rates.
- Many models of these pumps are expensive.
- Compressed gas or electric power is needed.
- Sediment in water may cause clogging of the valves or eroding the impellers with some of these pumps.
- Decontamination of internal components can be difficult and time-consuming.

5.5 Onsite Water Quality Testing

This section describes the procedures and equipment required to measure the following parameters of an aqueous sample in the field:

- pH
- Specific Conductance
- Temperature
- Dissolved Oxygen (DO)
- Oxidation-Reduction Potential (ORP)

- Turbidity
- Salinity

This section is applicable for use in an onsite groundwater quality monitoring program to be conducted at a hazardous or nonhazardous site. The procedures and equipment described are applicable to groundwater samples and are not, in general, subject to solution interferences from color, turbidity, and colloidal material or suspended matter.

This section provides general information for measuring the parameters listed above with instruments and techniques in common use. Since instruments from different manufacturers may vary, review of the manufacturer's literature pertaining to the use of a specific instrument is required before use. Most meters used to measure field parameters require calibration on a daily basis. Refer to SOP 6.3 for example equipment calibration log.

5.5.1 Measurement of pH

5.5.1.1 General

Measurement of pH is one of the most important and frequently used tests in water chemistry. Practically every phase of water supply and wastewater treatment such as acid-base neutralization, water softening, and corrosion control is pH dependent. Likewise, the pH of leachate can be correlated with other chemical analyses to determine the probable source of contamination. It is therefore important that reasonably accurate pH measurements be taken.

Two methods are given for pH measurement: the pH meter and pH indicator paper. The indicator paper is used when only a rough estimate of the pH is required, and the pH meter when a more accurate measurement is needed. The response of a pH meter can be affected to a slight degree by high levels of colloidal or suspended solids, but the effect is usually small and generally of little significance. Consequently, specific methods to overcome this interference are not described. The response of pH paper is unaffected by solution interferences from color, turbidity, colloidal or suspended materials unless extremely high levels capable of coating or masking the paper are encountered. In such cases, use of a pH meter is recommended.

5.5.1.2 Principles of Equipment Operation

Use of pH papers for pH measurement relies on a chemical reaction caused by the acidity or alkalinity of the solution created by the addition of the water sample reacting with the indicator compound on the paper. Various types of pH papers are available, including litmus (for general acidity or alkalinity determination) and specific pH range hydrion paper.

Use of a pH meter relies on the same principle as other ion-specific electrodes. Measurement relies on establishment of a potential difference across a glass or other type of membrane in response to (in this instance, hydrogen) ion concentration across that membrane. The membrane is conductive to ionic species and, in combination with a standard or reference electrode, a potential difference proportional to the ion concentration is generated and measured.

5.5.1.3 Equipment

The following equipment is needed for taking pH measurements:

- Stand-alone portable pH meter, or combination meter (e.g., Horiba U-10), or combination meters equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).

- Combination electrode with polymer body to fit the above meter (alternately a pH electrode and a reference electrode can be used if the pH meter is equipped with suitable electrode inputs).
- Buffer solutions, as specified by the manufacturer.
- pH indicator paper, to cover the pH range 2 through 12.
- Manufacturer's operation manual.

5.5.1.4 Measurement Techniques for Field Determination of pH

pH Meter

The following procedure is used for measuring pH with a pH meter (meter standardization is according to manufacturer's instructions):

- Inspect the instrument and batteries prior to initiation of the field effort.
- Check the integrity of the buffer solutions used for field calibration. Buffer solutions need to be changed often as a result of degradation upon exposure to the atmosphere.
- If applicable, make sure all electrolyte solutions within the electrode(s) are at their proper levels and that no air bubbles are present within the electrode(s).
- Calibrate on a daily use basis (or as recommended by manufacturer) following manufacturer's instructions. Record calibration data on an equipment calibration log sheet.
- Immerse the electrode(s) in the sample. Stabilization may take several seconds to minutes. If the pH continues to drift, the sample temperature may not be stable, a physical reaction (e.g., degassing) may be taking place in the sample, or the meter or electrode may be malfunctioning. This must be clearly noted in the logbook.
- Read and record the pH of the sample. pH shall be recorded to the nearest 0.01 pH unit. Also record the sample temperature.
- Rinse the electrode(s) with deionized water.
- Store the electrode(s) in an appropriate manner when not in use.

Any visual observation of conditions which may interfere with pH measurement, such as oily materials, or turbidity, shall be noted.

pH Paper

Use of pH paper is very simple and requires no sample preparation, standardization, etc. pH paper is available in several ranges, including wide-range (indicating approximately pH 1 to 12), mid-range (approximately pH 0 to 6, 6 to 9, 8 to 14) and narrow-range (many available, with ranges as narrow as 1.5 pH units). The appropriate range of pH paper shall be selected. If the pH is unknown the investigation shall start with wide-range paper and proceed with successively narrower range paper until the sample pH is adequately determined.

5.5.2 Measurement of Specific Conductance

5.5.2.1 General

Conductance provides a measure of dissolved ionic species in water and can be used to identify the direction and extent of migration of contaminants in groundwater or surface water. It can also be used as a measure of subsurface biodegradation or to indicate alternate sources of groundwater contamination.

Conductivity is a numerical expression of the ability of a water sample to carry an electric current. This value depends on the total concentration of the ionized substances dissolved in the water and the temperature at which the measurement is made. The mobility of each of the various dissolved ions, their valences, and their actual and relative concentrations affect conductivity.

It is important to obtain a specific conductance measurement soon after taking a sample, since temperature changes, precipitation reactions, and absorption of carbon dioxide from the air all affect the specific conductance. Most conductivity meters in use today display specific conductance (SC); units of milliSiemens per centimeter, which is the conductivity normalized to temperature @ 25°C. This format (SC) is the required units recorded on the groundwater sample log field form (Attachment B).

5.5.2.2 Principles of Equipment Operation

An aqueous system containing ions will conduct an electric current. In a direct-current field, the positive ions migrate toward the negative electrode, while the negatively charged ions migrate toward the positive electrode. Most inorganic acids, bases and salts (such as hydrochloric acid, sodium carbonate, or sodium chloride, respectively) are relatively good conductors. Conversely, organic compounds such as sucrose or benzene, which do not dissociate in aqueous solution, conduct a current very poorly, if at all.

A conductance cell and a Wheatstone Bridge (for the measurement of potential difference) may be used for measurement of electrical resistance. The ratio of current applied to voltage across the cell may also be used as a measure of conductance. The core element of the apparatus is the conductivity cell containing the solution of interest. Depending on ionic strength of the aqueous solution to be tested, a potential difference is developed across the cell which can be converted directly or indirectly (depending on instrument type) to a measurement of specific conductance.

5.5.2.3 Equipment

The following equipment is needed for taking specific conductance (SC) measurements:

- Stand alone portable conductivity meter, or combination meter (e.g., Horiba U-10), or combination meters equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Calibration solution, as specified by the manufacturer.
- Manufacturer's operation manual.

A variety of conductivity meters are available which may also be used to monitor salinity and temperature. Probe types and cable lengths vary, so equipment must be obtained to meet the specific requirement of the sampling program.

5.5.2.4 Measurement Techniques for Specific Conductance

The steps involved in taking specific conductance measurements are listed below (standardization is according to manufacturer's instructions):

- Check batteries and calibrate instrument before going into the field.
- Calibrate on a daily use basis (or as recommended by manufacturer), according to the manufacturer's instructions and record all pertinent information on an equipment calibration log sheet. Potassium chloride solutions with a SC closest to the values expected in the field shall be used for calibration.
- Rinse the cell with one or more portions of the sample to be tested or with deionized water.
- Immerse the electrode in the sample and measure the conductivity.
- Read and record the results in a field logbook or sample log sheet.
- Rinse the electrode with deionized water.

If the specific conductance measurements become erratic, recalibrate the instrument and see the manufacturer's instructions for details.

5.5.3 Measurement of Temperature

5.5.3.1 General

In combination with other parameters, temperature can be a useful indicator of the likelihood of biological action in a water sample. It can also be used to trace the flow direction of contaminated groundwater. Temperature measurements shall be taken in-situ, or as quickly as possible in the field. Collected water samples may rapidly equilibrate with the temperature of their surroundings.

5.5.3.2 Equipment

Temperature measurements may be taken with alcohol-toluene, mercury filled, dial-type thermometers or combination meters equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).. In addition, various meters such as specific conductance or dissolved oxygen meters, which have temperature measurement capabilities, may also be used. Using such instrumentation along with suitable probes and cables, in-situ measurements of temperature at great depths can be performed.

5.5.3.3 Measurement Techniques for Water Temperature

If a thermometer is used to determine the temperature for a water sample:

- Immerse the thermometer in the sample until temperature equilibrium is obtained (1-3 minutes). To avoid the possibility of cross-contamination, the thermometer shall not be inserted into samples which will undergo subsequent chemical analysis.
- Record values in a field logbook or sample log sheet.

If a temperature meter or probe is used, the instrument shall be calibrated according to manufacturer's recommendations.

5.5.4 Measurement of Dissolved Oxygen

5.5.4.1 General

Dissolved oxygen (DO) levels in natural water and wastewater depend on the physical, chemical and biochemical activities in the water body. Conversely, the growth of many aquatic organisms as well as the rate of corrosivity, are dependent on the dissolved oxygen concentration. Thus, analysis for dissolved oxygen is a key test in water pollution and waste treatment process control. If at all possible, DO measurements shall be taken in-situ, since concentration may show a large change in a short time if the sample is not adequately preserved.

The monitoring method discussed herein is limited to the use of dissolved oxygen meters only. Chemical methods of analysis (i.e., Winkler methods) are available, but require more equipment and greater sample manipulation. Furthermore, DO meters, using a membrane electrode, are suitable for highly polluted waters, because the probe is completely submersible, and is not susceptible to interference caused by color, turbidity, colloidal material or suspended matter.

5.5.4.2 Principles of Equipment Operation

Dissolved oxygen probes are normally electrochemical cells that have two solid metal electrodes of different nobility immersed in an electrolyte. The electrolyte is retained by an oxygen-permeable membrane. The metal of highest nobility (the cathode) is positioned at the membrane. When a suitable potential exists between the two metals, reduction of oxygen to hydroxide ion (OH^-) occurs at the cathode surface. An electrical current is developed that is directly proportional to the rate of arrival of oxygen molecules at the cathode.

Since the current produced in the probe is directly proportional to the rate of arrival of oxygen at the cathode, it is important that a fresh supply of sample always be in contact with the membrane. Otherwise, the oxygen in the aqueous layer along the membrane is quickly depleted and false low readings are obtained. It is therefore necessary to stir the sample (or the probe) constantly to maintain fresh solution near the membrane interface. Stirring, however, shall not be so vigorous that additional oxygen is introduced through the air-water interface at the sample surface. To avoid this possibility, some probes are equipped with stirrers to agitate the solution near the probe, while leaving the surface of the solution undisturbed.

Dissolved oxygen probes are relatively unaffected by interferences. Interferences that can occur are reactions with oxidizing gases (such as chlorine) or with gases such as hydrogen sulfide, which are not easily depolarized from the indicating electrode. If a gaseous interference is suspected, it shall be noted in the field log book and checked if possible. Temperature variations can also cause interference because probes exhibit temperature sensitivity. Automatic temperature compensation is normally provided by the manufacturer.

5.5.4.3 Equipment

The following equipment is needed to measure dissolved oxygen concentration:

- Stand alone portable dissolved oxygen meter, or combination meter (e.g., Horiba U-10), or combination meters equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Sufficient cable to allow the probe to contact the sample.
- Manufacturer's operation manual.

5.5.4.4 Measurement Techniques for Dissolved Oxygen Determination

Probes differ as to specifics of use. Follow the manufacturer's instructions to obtain an accurate reading. The following general steps shall be used to measure the dissolved oxygen concentration:

- The equipment shall be calibrated and have its batteries checked before going to the field.
- The probe shall be conditioned in a water sample for as long a period as practical before use in the field. Long periods of dry storage followed by short periods of use in the field may result in inaccurate readings.
- The instrument shall be calibrated in the field according to manufacturer's recommendations or in a freshly air-saturated water sample of known temperature.
- Record all pertinent information on an equipment calibration sheet.
- Rinse the probe with deionized water.
- Immerse the probe in the sample. Be sure to provide for sufficient flow past the membrane by stirring the sample. Probes without stirrers placed in wells can be moved up and down.
- Record the dissolved oxygen content and temperature of the sample in a field logbook or sample log sheet.
- Rinse the probe with deionized water.
- Recalibrate the probe when the membrane is replaced, or as needed. Follow the manufacturer's instructions.

Note that in-situ placement of the probe is preferable, since sample handling is not involved. This however, may not always be practical.

Special care shall be taken during sample collection to avoid turbulence which can lead to increased oxygen solubilization and positive test interferences.

5.5.5 Measurement of Oxidation-Reduction Potential

5.5.5.1 General

The oxidation-reduction potential (ORP) provides a measure of the tendency of organic or inorganic compounds to exist in an oxidized state. The ORP parameter therefore provides evidence of the likelihood of anaerobic degradation of biodegradable organics or the ratio of activities of oxidized to reduced species in the sample.

5.5.5.2 Principles of Equipment Operation

When an inert metal electrode, such as platinum, is immersed in a solution, a potential is developed at that electrode depending on the ions present in the solution. If a reference electrode is placed in the same solution, an ORP electrode pair is established. This electrode pair allows the potential difference between the two electrodes to be measured and is dependent on the concentration of the ions in solution. By this measurement, the ability to oxidize or reduce species in solution may be determined. Supplemental

measurements, such as dissolved oxygen, may be correlated with ORP to provide a knowledge of the quality of the solution, water, or wastewater.

5.5.5.3 Equipment

The following equipment is needed for measuring the oxidation-reduction potential of a solution:

- Combination meters with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Reference solution as specified by the manufacturer.
- Manufacturer's operation manual.

5.5.5.4 Measurement Techniques for Oxidation-Reduction Potential

The following procedure is used for measuring oxidation-reduction potential:

- The equipment shall be checked using the manufacturer's recommended reference solution and have its batteries checked before going to the field.
- Thoroughly rinse the electrode with deionized water.
- If the probe does not respond properly to the recommended reference solution, then verify the sensitivity of the electrodes by noting the change in millivolt reading when the pH of a test solution is altered. The ORP will increase when the pH of a test solution decreases, and the ORP will decrease if the test solution pH is increased. Place the sample in a clean container and agitate the sample. Insert the electrodes and note the ORP drops sharply when the caustic is added (i.e., pH is raised) thus indicating the electrodes are sensitive and operating properly. If the ORP increases sharply when the caustic is added, the polarity is reversed and must be corrected in accordance with the manufacturer's instructions or the probe should be replaced.
- Record all pertinent information on an equipment calibration log sheet.

5.5.6 Measurement of Turbidity

5.5.6.1 General

Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample. Turbidity in water is caused by suspended matter, such as clay, silt, finely divided organic and inorganic matter, soluble colored organic compounds, and microscopic organisms, including plankton.

It is important to obtain a turbidity reading immediately after taking a sample, since irreversible changes in turbidity may occur if the sample is stored too long.

5.5.6.2 Principles of Equipment Operation

Turbidity is measured by the Nephelometric Method. This method is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions. The higher the scattered light intensity, the higher the turbidity.

Formazin polymer is used as the reference turbidity standard suspension because of its ease of preparation combined with a higher reproducibility of its light-scattering properties than clay or turbid

natural water. The turbidity of a specified concentration of formazin suspension is defined as 40 nephelometric units. This same suspension has an approximate turbidity of 40 Jackson units when measured on the candle turbidmeter. Therefore, nephelometric turbidity units (NTU) based on the formazin preparation will approximate units derived from the candle turbidimeter but will not be identical to them.

5.5.6.3 Equipment

The following equipment is needed for turbidity measurement:

- Light meter (e.g., LaMotte 2020) which calibrates easily using test cells with standards of 0.0 NTUs, and 10 NTUs, or combination meter (e.g., Horiba U-10), or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Calibration solution, as specified by the manufacturer.
- Manufacturer's operation manual.

5.5.6.4 Measurement Techniques for Turbidity

The steps involved in taking turbidity measurements utilizing an electrode (e) or light meter (l) are listed below (standardization is according to manufacturer's instructions):

- Check batteries and calibrate instrument before going into the field.
- Check the expiration date (etc.) of the solutions used for field calibration.
- Calibrate on a daily use basis, according to the manufacturer's instructions and record all pertinent information on an equipment calibration log sheet.
- Rinse the electrode with one or more portions of the sample to be tested or with deionized water (applies to "e").
- Fill the light meters glass test cell with ~5 ml of sample, screw on cap, wipe off glass, place test cell in light meter and close the lid (applies to "l").
- Immerse the electrode in the sample and measure the turbidity (applies to "e").
- The reading must be taken immediately as suspended solids will settle over time resulting in a lower, inaccurate turbidity reading.
- Read and record the results in a field logbook or sample log sheet. Include a physical description of the sample, including color, qualitative estimate of turbidity, etc.
- Rinse the electrode or test cell with deionized water.

5.5.7 Measurement of Salinity

5.5.7.1 General

Salinity is a unitless property of industrial and natural waters. It is the measurement of dissolved salts in a given mass of solution. Note: Most field meters determined salinity automatically from conductivity and

temperature. The displayed value will be displayed in either parts per thousand (ppt) or % (e.g., 35 ppt will equal 3.5%).

5.5.7.2 Principles of Equipment Operation

Salinity is determined automatically from the meter's conductivity and temperature readings according to algorithms (found in *Standard methods for the Examination of Water and Wastewater*). Depending on the meter, the results are displayed in either ppt or %. The salinity measurements are carried out in reference to the conductivity of standard seawater (*corrected to S = 35*).

5.5.7.3 Equipment

The following equipment is needed for Salinity measurements:

- Multi-parameter water quality meter capable of measuring conductive, temperature and converting them to salinity (e.g., Horiba U-10 or YSI 600 series).
- Calibration Solution, as specified by the manufacturer.
- Manufacturer's operation manual.

5.5.7.4 Measurement Techniques for Salinity

The steps involved in taking Salinity measurements are listed below (standardization is according to manufacturer's instructions):

- Check batteries and calibrate before going into the field.
- Check the expiration date (etc.) of the solutions used for field calibration.
- Calibrate on a daily use basis, according to the manufacturer's instructions and record all pertinent information on an equipment calibration log sheet.
- Rinse the cell with the sample to be tested.
- Immerse the multi-probe in the sample and measure the salinity. Read and record the results in a field logbook or sample log sheet.
- Rinse the probes with deionized water.

5.6 Sampling

5.6.1 Sampling Plan

The sampling approach consisting of the following, shall be developed as part of the project plan documents which are approved prior to beginning work in the field:

- Background and objectives of sampling.
- Brief description of area and waste characterization.
- Identification of sampling locations, with map or sketch, and applicable well construction data (well size, depth, screened interval, reference elevation).

- Intended number, sequence volumes, and types of samples. If the relative degrees of contamination between wells is unknown or insignificant, a sampling sequence which facilitates sampling logistics may be followed. Where some wells are known or strongly suspected of being highly contaminated, these shall be sampled last to reduce the risk of cross-contamination between wells as a result of the sampling procedures.
- Sample preservation requirements.
- Work schedule.
- List of team members.
- List of observers and contacts.
- Other information, such as the necessity for a warrant or permission of entry, requirement for split samples, access problems, location of keys, etc.

5.6.2 Sampling Methods

The collection of a groundwater sample consists of the following steps:

1. The site Health & Safety Officer (or designee) will first open the well cap and use volatile organic detection equipment (PID or FID) on the escaping gases at the well head to determine the need for respiratory protection.
2. When proper respiratory protection has been donned, sound the well for total depth and water level (using clean equipment) and record these data on a groundwater sampling log sheet (see Attachment B); then calculate the fluid volume in the well pipe (as previously described in this SOP).
3. Calculate well volume to be removed as stated in Section 5.3.
4. Select the appropriate purging equipment (see Attachment A). If an electric submersible pump with packer is chosen, go to Step 10.
5. Lower the purging equipment or intake into the well to a short distance below the water level and begin water removal. Collect the purged water and dispose of it in an acceptable manner (as applicable). Lower the purging device, as required, to maintain submergence.
6. Measure the rate of discharge frequently. A graduated bucket or cylinder and stopwatch are most commonly used.
7. Observe the peristaltic pump intake for degassing "bubbles." If bubbles are abundant and the intake is fully submerged, this pump is not suitable for collecting samples for volatile organics.
8. Purge a minimum of three to five casing volumes before sampling. In low-permeability strata (i.e., if the well is pumped to dryness), one volume will suffice. Purged water shall be collected in a designated container and disposed in an acceptable manner.
9. If sampling using a pump, lower the pump intake to midscreen (or the middle of the open section in uncased wells) and collect the sample. If sampling with a bailer, lower the bailer to just below the water surface.

10. (For pump and packer assembly only). Lower the assembly into the well so that the packer is positioned just above the screen or open section. Inflate the packer. Purge a volume equal to at least twice the screened interval (or unscreened open section volume below the packer) before sampling. Packers shall always be tested in a casing section above ground to determine proper inflation pressures for good sealing.

11. In the event that recovery time of the well is very slow (e.g., 24 hours or greater), sample collection can be delayed until the following day. If the well has been purged early in the morning, sufficient water may be standing in the well by the day's end to permit sample collection. If the well is incapable of producing a sufficient volume of sample at any time, take the largest quantity available and record this occurrence in the site logbook.

12. Fill sample containers (preserve and label as described in SOP SA-6.1).

13. Replace the well cap and lock as appropriate. Make sure the well is readily identifiable as the source of the samples.

14. Process sample containers as described in SOP SA-6.1.

15. Decontaminate equipment as described in SOP SA-7.1.

5.7 Low Flow Purging and Sampling

5.7.1 Scope & Application

Low flow purging and sampling techniques are sometimes required for groundwater sampling activities. The purpose of low flow purging and sampling is to collect groundwater samples that contain "representative" amounts of mobile organic and inorganic constituents in the vicinity of the selected open well interval, at or near natural flow conditions. The minimum stress procedure emphasizes negligible water level drawdown and low pumping rates in order to collect samples with minimal alterations in water chemistry. This procedure is designed primarily to be used in wells with a casing diameter of 1 inch or more and a saturated screen, or open interval, length of ten feet or less. Samples obtained are suitable for analyses of common types of groundwater contaminants (volatile and semi-volatile organic compounds, pesticides, PCBs, metals and other inorganic ions [cyanide, chloride, sulfate, etc.]). This procedure is not designed to collect non-aqueous phase liquids samples from wells containing light or dense non-aqueous phase liquids (LNAPLs or DNAPLs), using the low flow pumps.

The procedure is flexible for various well construction types and groundwater yields. The goal of the procedure is to obtain a turbidity level of less than 10 NTU and to achieve a water level drawdown of less than 0.3 feet during purging and sampling. If these goals cannot be achieved, sample collection can take place provided the remaining criteria in this procedure are met.

5.7.2 Equipment

The following equipment is required (as applicable) for low flow purging and sampling:

- Adjustable rate, submersible pump (e.g., centrifugal or bladder pump constructed of stainless steel or Teflon).
- Disposable clear plastic bottom filling bailers may be used to check for and obtain samples of LNAPLs or DNAPLs.

- Tubing - Teflon, Teflon-lined polyethylene, polyethylene, PVC, Tygon, or stainless steel tubing can be used to collect samples for analysis, depending on the analyses to be performed and regulatory requirements.
- Water level measuring device, 0.01 foot accuracy, (electronic devices are preferred for tracking water level drawdown during all pumping operations).
- Interface probe, if needed.
- Flow measurement supplies.
- Power source (generator, nitrogen tank, etc.). If a gasoline generator is used, it must be located downwind and at a safe distance from the well so that the exhaust fumes do not contaminate the samples.
- Indicator parameter monitoring instruments - pH, turbidity, specific conductance, and temperature. Use of a flow-through cell is recommended. Optional Indicators - ORP, salinity, and dissolved oxygen, flow-through cell is required. Standards to perform field calibration of instruments.
- Decontamination supplies.
- Logbook(s), and other forms (see Attachments B and C).
- Sample Bottles.
- Sample preservation supplies (as required by the analytical methods).
- Sample tags and/or labels.
- Well construction data, location map, field data from last sampling event (if available).
- Field Sampling Plan.
- PID or FID instrument for measuring VOCs (volatile organic compounds).

5.7.3 Purging and Sampling Procedure

Open monitoring well, measure head space gases using PID/FID. If there is an indication of off gassing when opening the well, wait 3-5 minutes to permit water level an opportunity to reach equilibrium.

Measure and record the water level immediately prior to placing the pump in the well.

Lower pump or tubing slowly into the well so that the pump intake is located at the center of the saturated screen length of the well. If possible keep the pump intake at least two feet above the bottom of the well, to minimize mobilization of sediment that may be present in the bottom of the well. Collection of turbidity-free water samples may be difficult if there is three feet or less of standing water in the well.

Start with the initial pump rate set at approximately 0.1 liters/minute. Use a graduated cylinder and stopwatch to measure the pumping rate. Adjust pumping rates as necessary to prevent drawdown from exceeding 0.3 feet during purging. If no drawdown is noted, the pump rate may be increased (to a max of 0.4 liters/minute) to expedite the purging and sampling event. The pump rate will be reduced if turbidity is greater than 10 NTUs after all other field parameters have stabilized. If groundwater is drawn down below

the top of the well screen, purging will cease or the well will be pumped to dryness and the well will be allowed to recover before purging continues. Slow recovering wells will be identified and purged at the beginning of the workday. If possible, samples will be colleted from these wells within the same workday and no later than 24 hours after the start of purging.

Measure the well water level using the water level meter every 5 to 10 minutes. Record the well water level on the Low-Flow Purge Data Form (Attachment C).

Record on the Low-Flow Purge Data Form every 5 to 10 minutes the water quality parameters (pH, specific conductance, temperature, turbidity, oxidation-reduction potential, dissolved oxygen and salinity or as specified by the approved site specific work plan) measured by the water quality meter and turbidity meter. If the cell needs to be cleaned during purging operations, continue pumping (allow the pump to discharge into a container) and disconnect the cell. Rinse the cell with distilled/deionized water. After cleaning is completed, reconnect the flow-through cell and continue purging. Document the cell cleaning on the Low-Flow Purge Data Form.

Measure the flow rate using a graduated cylinder. Remeasure the flow rate any time the pump rate is adjusted.

During purging, check for the presence of bubbles in the flow-through cell. The presence of bubbles is an indication that connections are not tight. If bubbles are observed, check for loose connections.

After stabilization is achieved, sampling can begin when a minimum of two saturated screen volumes have been removed and three consecutive readings, taken at 5 to 10 minute intervals, are within the following limits:

- pH ~~±0.2~~ 0.1 standard units
- Specific conductance ~~±10%~~ 3
- Temperature ~~±10%~~
- Turbidity less than ~~10~~ 5 NTUs
- Dissolved oxygen ±10%

If the above conditions have still not been met after the well has been purged for 4 hours, purging will be considered complete and sampling can begin. Record the final well stabilization parameters from the Low-Flow Purge Data Form onto the Groundwater Sample Log Form.

VOC samples are preferably collected first, directly into pre-preserved sample containers. Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

If the water column in the pump tubing collapses (water does not completely fill the tubing) before exiting the tubing, use one of the following procedures to collect VOC samples: (1) Collect the non-VOCs samples first, then increase the flow rate incrementally until the water column completely fills the tubing, collect the sample and record the new flow rate; (2) reduce the diameter of the existing tubing until the water column fills the tubing either by adding a connector (Teflon or stainless steel), or clamp which should reduce the flow rate by constricting the end of the tubing; (3) insert a narrow diameter Teflon tube into the pump's tubing so that the end of the tubing is in the water column and the other end of the tubing protrudes beyond the pump's tubing, collect sample from the narrow diameter tubing.

Prepare samples for shipping as per SOP SA-6.1.

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ATTACHMENT A

PURGING EQUIPMENT SELECTION

Diameter Casing		Bailer	Peristaltic Pump	Vacuum Pump	Air-lift	Diaphragm "Trash" Pump	Submersible Diaphragm Pump	Submersible Electric Pump	Submersible Electric Pump w/Packer
1.25-Inch	Water level <25 feet	X	X	X	X	X			
	Water Level >25 feet	X			X				
2-Inch	Water level <25 feet	X	X	X	X	X	X		
	Water Level >25 feet	X			X		X		
4-Inch	Water level <25 feet	X	X	X	X	X	X	X	X
	Water Level >25 feet	X			X		X	X	X
6-Inch	Water level <25 feet				X	X		X	X
	Water Level >25 feet				X			X	X
8-Inch	Water level <25 feet				X	X		X	X
	Water Level >25 feet				X			X	X

ATTACHMENT A

PURGING EQUIPMENT SELECTION

PAGE 2

Manufacturer	Model Name/Number	Principle of Operation	Maximum Outside Diameter/Length (Inches)	Construction Materials (w/Lines and Tubing)	Lift Range (ft)	Delivery Rates or Volumes	1982 Price (Dollars)	Comments
BarCad Systems, Inc.	BarCad Sampler	Dedicated; gas drive (positive displacement)	1.5/16	PE, brass, nylon, aluminum oxide	0-150 with std. tubing	1 liter for each 10-15 feet of submergence	$220-350	Requires compressed gas; custom sizes and materials available; acts as piezometer.
Cole-Parmer Inst. Co.	Master Flex 7570 Portable Sampling Pump	Portable; peristaltic (suction)	<1.0/NA	(not submersible) Tygon®, silicone Viton®	0-30	670 mL/min with 7015-20 pump head	$500-600	AC/DC; variable speed control available; other models may have different flow rates.
ECO Pump Corp.	SAMPLifier	Portable; venturi	<1.5 or <2.0/NA	PP, PE, PVC, SS, Teflon®, Tefzel®	0-100	0-500 mL/min depending on lift	$400-700	AC, DC, or gasoline-driven motors available; must be primed.
Geltek Corp.	Bailer 219-4	Portable; grab (positive displacement)	1.66/38	Teflon®	No limit	1,075 mL	$120-135	Other sizes available.
GeoEngineering, Inc.	GEO-MONITOR	Dedicated; gas drive (positive displacement)	1.5/16	PE, PP, PVC, Viton®	Probably 0-150	Approximately 1 liter for each 10 feet of submergence	$185	Acts as piezometer; requires compressed gas.
Industrial and Environmental Analysts, Inc. (IEA)	Aquarius	Portable; bladder (positive displacement)	1.75/43	SS, Teflon®, Viton®	0-250	0-2,800 mL/min	$1,500-3,000	Requires compressed gas; other models available; AC, DC, manual operation possible.
IEA	Syringe Sampler	Portable; grab (positive displacement)	1.75/43	SS, Teflon®	No limit	850 mL sample volume	$1,100	Requires vacuum and/or pressure from hand pump.
Instrument Specialties Co. (ISCO)	Model 2600 Well Sampler	Portable; bladder (positive displacement)	1.75/50	PC, silicone, Teflon®, PP, PE, Detrin®, acetal	0-150	0-7,500 mL/min	$990	Requires compressed gas (40 psi minimum).
Keck Geophysical Instruments, Inc.	SP-81 Submersible Sampling Pump	Portable; helical rotor (positive displacement)	1.75/25	SS, Teflon®, PP, EPDM, Viton®	0-160	0-4,500 mL/min	$3,500	DC operated.
Leonard Mold and Die Works, Inc.	GeoFilter. Small Diameter Well Pump (#0500)	Portable; bladder (positive displacement)	1.75/38	SS, Teflon®, PC, Neoprene®	0-400	0-3,500 mL/min	$1,400-1,500	Requires compressed gas (55 psi minimum); pneumatic or AC/DC control module.
Oil Recovery Systems, Inc.	Surface Sampler	Portable; grab (positive displacement)	1.75/12	acrylic, Detrin®	No limit	Approximately 250 mL	$125-160	Other materials and models available; for measuring thickness of "floating" contaminants.
Q.E.D. Environmental Systems, Inc.	Well Wizard® Monitoring System (P-100)	Dedicated; bladder (positive displacement)	1.66/36	PVC	0-230	0-2,000 mL/min	$300-400	Requires compressed gas; piezometric level indicator; other materials available.

ATTACHMENT A

PURGING EQUIPMENT SELECTION

PAGE 3

Manufacturer	Model Name/Number	Principle of Operation	Maximum Outside Diameter/Length (Inches)	Construction Materials (w/Lines and Tubing)	Lift Range (ft)	Delivery Rates or Volumes	1982 Price (Dollars)	Comments
Randolph Austin Co.	Model 500 Vari-Flow Pump	Portable; peristaltic (suction)	<0.5/NA	(Not submersible) Rubber, Tygon®, or Neoprene®	0-30	See comments	$1,200-1,300	Flow rate dependent on motor and tubing selected; AC operated; other models available.
Robert Bennett Co.	Model 180	Portable; piston (positive displacement)	1.8/22	SS, Teflon®, Delrin® PP, Viton®, acrylic, PE	0-500	0-1,800 mL/min	$2,600-2,700	Requires compressed gas; water level indicator and flow meter; custom models available.
Slope Indicator Co. (SINCO)	Model 514124 Pneumatic Water Sampler	Portable; gas drive (positive displacement)	1.9/18	PVC, nylon	0-1,100	250 mL/flushing cycle	$250-350	Requires compressed gas; SS available; piezometer model available; dedicated model available.
Solinst Canada Ltd.	5W Water Sampler	Portable; grab (positive displacement)	1.9/27	PVC, brass, nylon, Neoprene®	0-330	500 mL	$1,300-1,800	Requires compressed gas; custom models available.
TIMCO Mfg. Co., Inc.	Std. Bailer	Portable; grab (positive displacement)	1.66/Custom	PVC, PP	No limit	250 mL/ft of bailer	$20-60	Other sizes, materials, models available; optional bottom-emptying device available; no solvents used.
TIMCO	Air or Gas Lift Sampler	Portable; gas drive (positive displacement)	1.66/30	PVC, Tygon®, Teflon®	0-150	350 mL/flushing cycle	$100-200	Requires compressed gas; other sizes, materials, models available; no solvents used.
Tole Devices Co.	Sampling Pump	Portable; bladder (positive displacement)	1.38/48	SS, silicone, Delrin®, Tygon®	0-125	0-4,000 mL/min	$800-1,000	Compressed gas required; DC control module; custom built.

Construction Material Abbreviations:

- PE Polyethylene
- PP Polypropylene
- PVC Polyvinyl chloride
- SS Stainless steel
- PC Polycarbonate
- EPDM Ethylene-propylene diene (synthetic rubber)

Other Abbreviations:

- NA Not applicable
- AC Alternating current
- DC Direct current

NOTE: Other manufacturers market pumping devices which could be used for groundwater sampling, though not expressly designed for this purpose. The list is not meant to be all-inclusive and listing does not constitute endorsement for use. Information in the table is from sales literature and/or personal communication. No skimmer, scavenger-type, or high-capacity pumps are included.

Source: Barcelona et al., 1983.

ATTACHMENT B
GROUNDWATER SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

GROUNDWATER SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[ ] Domestic Well Data
[x] Monitoring Well Data
[ ] Other Well Type: _____
[ ] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____
Type of Sample:
[X] Low Concentration
[ ] High Concentration

SAMPLING DATA

Date:	Color	pH	S.C.	Temp.	Turbidity	DO	ORP	Other
Time:	Visual	Standard	mS/cm	Degrees C	NTU	mg/l	mV	NA
Method:								

PURGING DATA

Date:	Volume	pH	S.C.	Temp. (C)	Turbidity	DO	ORP	Other
Method:								
Monitor Reading (ppm):			SEE LOW FLOW PURGE DATA SHEET					
Well Casing Diameter & Material								
Type:								
Total Well Depth (TD):								
Static Water Level (WL):								
One Casing Volume(gal/L):								
Start Purge (hrs):								
End Purge (hrs):								
Total Purge Time (min):								
Total Vol. Purged (gal/L):								

SAMPLE COLLECTION INFORMATION

Analysis	Preservative	Container Requirements	Collected

OBSERVATION NOTES

QC Samples Collected

MS/MSD	Duplicate ID No.:	Signature(s):

ATTACHMENT C
LOW FLOW PURGE DATA SHEET

LOW FLOW PURGE DATA SHEET

PROJECT SITE NAME: _____ WELL ID.: _____
PROJECT NUMBER: _____ DATE: _____

Time (Hrs.)	Water Level (Ft. below TOC)	Flow (mL/Min.)	pH (S.U.)	S. Cond. (mS/cm)	Turb. (NTU)	DO (mg/L)	Temp. (Celcius)	ORP mV	Salinity % or ppt	Comments

SIGNATURE(S): _____ PAGE___OF___

S6 GH-1.2 EVALUATION OF EXISTING MONITORING WELLS AND WATER-LEVEL MEASUREMENTS

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1.0 PURPOSE

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

2.0 SCOPE

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

3.0 GLOSSARY

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

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

4.0 RESPONSIBILITIES

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

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

5.0 PROCEDURES

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

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

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

5.1 Preliminary Evaluation

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

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

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

5.2 Field Inspection

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

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

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

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

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

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

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

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

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

See Attachment A, Monitoring Well Inspection Sheet.

5.3 Water Level (Hydraulic Head) Measurements

5.3.1 General

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

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

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

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

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

5.3.2 Water Level Measuring Techniques

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

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

5.3.3 Methods

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

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

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

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

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

5.3.4 Water Level Measuring Devices

Electric Water Level Indicators

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

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

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

Popper or Bell Sounder

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

~~**Pressure Transducer**~~

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

~~**Borehole Geophysics**~~

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

5.3.5 Data Recording

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

5.3.6 Specific Quality Control Procedures for Water Level Measuring Devices

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

5.4 Equipment Decontamination

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

5.5 Health and Safety Considerations

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

6.0 RECORDS

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

ATTACHMENT A
MONITORING WELL INSPECTION SHEET

Monitoring Well Inspection Sheet

Project Name: _____ Date: _____

Location: _____ Time: _____

Tidally Influenced: Y / N Personnel: _____

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

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

Check List:

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

Condition of Well:

Protective Case:
Riser:
Well Pad:
Other:

Presence/Evidence of:

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

Comments:

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

ATTACHMENT B
GROUNDWATER LEVEL MESUREMENT SHEET

Tetra Tech NUS, Inc.

GROUNDWATER LEVEL MEASUREMENT SHEET

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

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

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

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S7 GH-2.9 WELL ABANDONMENT

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1.0 PURPOSE

Well abandonment is that procedure by which any monitoring well is permanently closed. Abandonment procedures are designed to prevent fluids from entering or migrating within the monitoring well. Therefore, an abandoned monitoring well must be sealed in such a manner that it can not act as a conduit for migration of contaminants from the ground surface to the water table or between aquifers.

It is important that the appropriate state or local agency be notified of monitoring well abandonment. The application of and adherence to this SOP must be tailored to applicable state, local, and Federal regulatory requirements.

2.0 SCOPE

The methods described in this procedure shall be used for all projects requiring well abandonment where specific state, local, or Federal regulations are unavailable. An abandoned well shall be filled and sealed so that it will not act as a pathway for the interchange of water between the surface and subsurface or present a hazard to the environment.

3.0 GLOSSARY

Well - Any constructed access point to an aquifer, confined or unconfined, including, but not limited to, test borings, hydropunch holes, monitoring points, and production wells.

Abandon - To permanently discontinue the use of a well. Any well shall require abandonment when it is no longer serving as a monitoring point or is in such a state of disrepair that continued use for the purpose of obtaining groundwater is impracticable, or when it has been permanently disconnected from any water supply system or irrigation system.

4.0 RESPONSIBILITIES

Project Manager - It shall be the responsibility of the Project Manager and/or Project Hydrogeologist to determine the applicability of well abandonment, based on the established scope and objective of the project and program-specific requirements. It shall be the responsibility of the Project Manager (or designee) to ensure that the procedures established for well abandonment are thoroughly specified and/or referenced in the relevant project planning documents. It shall be the responsibility of the Project Manager to ensure that the Field Operations Leader is familiar with the proper procedures for well abandonment and confirm the supervising project geologist or the subcontractor performing the well abandonment are qualified to perform such activities.

Field Operations Leader (FOL) - It shall be the responsibility of the Field Operations Leader to ensure that all field technicians and/or drilling personnel are thoroughly familiar with this Standard Operating Procedure. It shall be the responsibility of the FOL to ensure that the procedures identified in this SOP are used during well abandonment.

5.0 PROCEDURES

5.1 General

Well abandonment is warranted when the project team has reason to believe, on the basis of local conditions, that the well is causing or is a potential source of pollution to an aquifer; is a production well that is producing water that is polluted; or does not have a certificate of potability, if required. Wells may

also be abandoned once their designed purposes have been fulfilled and are determined to no longer be of use.

Well abandonment is conducted to eliminate physical hazards, prevent groundwater contamination, prevent intermixing of aquifer waters, and conserve aquifer yield and hydrostatic head.

Please note Federal, state, and local regulations concerning this activity may vary. Therefore, applicable regulatory requirements should be reviewed to determine the need for Licensed/Certified Well Drillers to complete/oversight this activity.

5.2 Material for Sealing

Acceptable sealing materials include concrete, portland cement grout, sodium-base bentonite clay, or combinations of these materials. These materials are defined as follows:

- Concrete may be used for filling the upper part of a well or water bearing formation, or plugging short sections of casing and filling large diameter wells.
- Portland cement grout is superior for sealing small openings, penetrating any annular space outside the casing, and for filling voids in the surrounding formation. Portland cement grout shall be composed of one bag of Type I cement per 6 to 8 gallons of water. Two parts sand to one part cement may be added.
- Bentonite clay, when applied as a heavy mud-laden fluid under pressure, has most of the advantages of cement grout, but under some conditions may be carried away into the surrounding formation. A bentonite clay mixture shall be composed of not less than 2 pounds of clay per gallon of water. Bentonite clay may not be used where it will come in contact with water of a pH below 5.0 or total dissolved solids (TDS) content greater than 1,000 mg/L or both. Bentonite may also be added to cement grout to add flexibility.

Fill materials include clay, silt, sand, gravel, crushed stone, or a mixtures of these materials may be used as a filler in sealing a well when used in conjunction with the sealing materials described above. Organic material may not be used and fill material may be required to be disinfected or certified clean prior to use. Spent drilling muds or drill cuttings are not to be used to seal a well.

5.3 Procedures for Sealing Wells

5.3.1 Preliminary Considerations

Several factors should be considered to determine the appropriate well abandonment method. These factors include:

- Conditions of the well.
- Details of well construction, including casing material, diameter of casing, depth of well, and well plumbness.
- Obstructions within the well that may interfere with filling or sealing.
- Hydrogeologic setting.
- Level of contamination and the zone or zones where it occurs.
- Regulatory requirements.

Degraded wells may not permit casing removal by pulling. Also, the casing material may dictate whether a casing can be removed intact. Stainless steel will have a higher tensile strength than PVC and may hold

together while pulling the casing; PVC well casing may break under pulling and may need to be overdrilled to remove it. The depth of the well and well plumbness may limit casing removal depending on whether a casing is pulled or overdrilled. In some cases, casings can be left in-place if they are properly filled with appropriate backfill.

The formation lithology influences the selection of casing removal. Unconsolidated materials can be drilled with hollow-stem augering techniques whereas consolidated materials cannot. Unconsolidated materials may also cave-in during well casing removal.

5.3.2 Filling and Sealing Procedures

Drilled wells (all wells not dug) shall be filled with sealing material or a combination of sealing material and fill material.

In some cases, well casing removal is necessary for well abandonment. If the borehole is unstable and may cave-in, sealing material will be emplaced simultaneously during casing removal. If the well is not grouted, casing may be pulled with hydraulic jacks or a drilling rig. It may also be pulled by sandlocking. Sandlocking consists of lowering a pipe wrapped with burlap approximately 2/3 of the well depth and filling the burlap wrap with sand. The pipe is slowly lifted and locks the sand, pulling the casing. Well casings can also be removed by overdrilling. Wells can be overdrilled with larger diameter hollow steam or solid stem augers or direct rotary techniques, using air or mud. Augers used for overdrilling should be at least 2 inches larger in diameter than the diameter of the well casing.

If well casing is in poor condition or is grouted in place, the casing may be ripped or perforated and filled and pressure grouted in place.

Abandoned wells shall be filled with the appropriate filling and sealing material placed from the bottom of the well upward. When Portland cement grout or concrete is used, it shall be placed in continuous operation using a tremie pipe. Sealing material shall be placed in the interval or intervals to be sealed by methods that prevent free fall, dilution, and/or separation of aggregates from cementing material.

A well constructed in unconsolidated material in an unconfined groundwater zone shall be filled and sealed by placing fill material in the well to the level of the water table, and filling the remainder of the well with sealing material. If the water table is at a depth greater than 40 feet, a minimum of 40 feet of sealing material shall be required.

A well which penetrates several aquifers or formations shall be filled and sealed in such a way as to prevent the vertical movement of water from one aquifer or formation to another. If the casing has been removed, sealing material shall be placed opposite the confining formations and from the surface down to the first confining formation. Sand and other suitable fill material may be placed opposite the producing aquifer. Ideally, the entire well can be filled with sealing material. If the casing has not been removed, the entire well shall be fill with sealing material.

A well penetrating creviced or cavernous rock shall be filled using coarse fill material opposite the cavernous or creviced rock portions of the well. Sealing material shall extend from the top of the unfractured rock portion of the well or base of the casing, whichever is deeper, to the surface. The minimum depth of sealing material may not be less than 10 feet.

In the case where wells penetrate specific aquifers where conditions necessitate the sealing of specific aquifers or formations, the annular space in the area of the specific aquifer or formation shall be sealed during the abandonment of the well.

A dug well exceeding 24 inches in diameter shall be filled and sealed by placing fill material (excluding clay or silt) in the well to a level approximately 5 feet below the land surface, and placing a 3 foot plug of sealing material above the fill. The remainder of the well shall be back filled with soil material.

6.0 REFERENCES

Maryland Department of the Environment (MDE Regulations); Title 26, Subtitle 04; Regulation of Water Supply, Sewage Disposal, and Solid Waste; Chapter 4--Well Construction.

U.S. EPA, February 1990. Handbook of Suggested Practices for the Design and Installation of Ground-Water Monitoring Wells.

S8 SA-7.1 DECONTAMINATION OF FIELD EQUIPMENT AND WASTE HANDLING

TETRA TECH NUS, INC.	STANDARD OPERATING PROCEDURES	Number SA-7.1	Page 1 of 8
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1.0 PURPOSE

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The objective/purpose of this SOP is intended to protect site personnel, general public, and the sample integrity through the prevention of cross contamination onto unaffected persons or areas. It is further intended through this procedure to provide guidelines regarding the appropriate procedures to be followed when decontaminating drilling equipment, monitoring well materials, chemical sampling equipment and field analytical equipment.

2.0 SCOPE

This procedure applies to all equipment including drilling equipment, heavy equipment, monitoring well materials, as well as chemical sampling and field analytical equipment decontamination that may be used to provide access/acquire environmental samples. Where technologically and economically feasible, single use sealed disposable equipment will be employed to minimize the potential for cross contamination. This procedure also provides general reference information on the control of contaminated materials.

3.0 GLOSSARY

Acid - For decontamination of equipment when sampling for trace levels of inorganics, a 10% solution of nitric acid in deionized water should be used. Due to the leaching ability of nitric acid, it should not be used on stainless steel.

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

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

Deionized Water (DI) - Deionized water is tap water that has been treated by passing through a standard deionizing resin column. This water may also pass through additional filtering media to attain various levels of analyte-free status. The DI water should meet CAP and NCCLS specifications for reagent grade, Type I water.

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

Pressure Washing - Employs high pressure pumps and nozzle configuration to create a high pressure spray of potable water. High pressure spray is employed to remove solids.

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

Steam Pressure Washing - This method employs a high pressure spray of heated potable water. This method through the application of heat provides for the removal of various organic/inorganic compounds.

4.0 RESPONSIBILITIES

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

Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved Standards Operating Procedures or as otherwise dictated by the approved project plan(s).

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

5.0 PROCEDURES

The process of decontamination is accomplished through the removal of contaminants, neutralization of contaminants, or the isolation of contaminants. In order to accomplish this activity a level of preparation is required. This includes site preparation, equipment selection, and evaluation of the process. Site contaminant types, concentrations, media types, are primary drivers in the selection of the types of decontamination as well as where it will be conducted. For purposes of this SOP discussion will be provided concerning general environmental investigation procedures.

The decontamination processes are typically employed at:

- Temporary Decontamination Pads/Facilities
- Sample Locations
- Centralized Decontamination Pad/Facilities
- Combination of some or all of the above

The following discussion represents recommended site preparation in support of the decontamination process.

5.1 Decontamination Design/Constructions Considerations

5.1.1 Temporary Decontamination Pads

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

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

- Site Location – The site selected should be within a reasonable distance from the work site but should avoid:

  - Pedestrian/Vehicle thoroughfares
  - Areas where control/custody cannot be maintained
  - Areas where a potential releases may be compounded through access to storm water transport systems, streams or other potentially sensitive areas.
  - Areas potentially contaminated.

- Pad – The pad should be constructed to provide the following characteristics

  - Size – The size of the pad should be sufficient to accept the equipment to be decontaminated as well as permitting free movement around the equipment by the personnel conducting the decontamination.

  - Slope – An adequate slope will be constructed to permit the collection of the water and potentially contaminated soils within a trough or sump constructed at one end. The collection point for wash waters should be of adequate distance that the decontamination workers do not have to walk through the wash waters while completing their tasks.

  - Sidewalls – The sidewalls should be a minimum of 6-inches in height to provide adequate containment for wash waters and soils. If splash represents a potential problem, splash guards should be constructed to control overspray. Sidewalls maybe constructed of wood, inflatables, sand bags, etc. to permit containment.

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

  - Wash/drying Racks – Auger flights, drill/drive rods require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process. A minimum ground clearance of 2-feet is recommended.

  - Maintenance – The work area should be periodically cleared of standing water, soils, and debris. This action will aid in eliminating slip, trip, and fall hazards. In addition, these articles will reduce potential backsplash and cross contamination. Hoses should be gathered when not in use to eliminate potential tripping hazards.

5.1.2 Decontamination Activities at Drill Rigs/DPT Units

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

Buckets shall be placed within mortar tubs or similar secondary containment tubs to prevent splash and spills from reaching unprotected media. Drying racks will be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/re-use.

5.1.3 Decontamination Activities at Remote Sample Locations

When sampling at remote locations sampling devices such as trowels, pumps/tubing should be evacuated of potentially contaminated media to the extent possible. This equipment should be wrapped in plastic for transport to the temporary/centralized decontamination location for final cleaning and disposition.

5.2 Equipment Decontamination Procedures

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

5.2.1 Monitoring Well Sampling Equipment

5.2.1.1 Groundwater sampling pumps – This includes pumps inserted into the monitoring well such as Bladder pumps, Whale pumps, Redi-Flo, reusable bailers, etc.

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

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

3) Insert the pump and tubing into a clean container of soapy water. Pump a sufficient amount of soapy water through the pump to flush any residual purge water. Once flushed, circulate soapy water through the pump to ensure the internal components are thoroughly flushed.

4) Remove the pump and tubing from the container, rinse external components using tap water. Insert the pump and tubing into a clean container of tap water. Pump a sufficient amount of tap water through the pump to evacuate all of the soapy water (until clear).

5) Rinse equipment with pesticide grade isopropanol

6) Repeat item #4 using deionized water through the hose to flush out the tap water and solvent residue as applicable.

7) Drain residual deionized water to the extent possible, allow components to air dry.

8) Wrap pump in aluminum foil or a clear clean plastic bag for storage.

5.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

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

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* - The solvent should be employed when samples contain oil, grease, PAHs, PCBs, and other hard to remove materials. If these are not of primary concern, the solvent step may be omitted. In addition, do not rinse PE, PVC, and associated tubing with solvents.

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

Note: In situations where oil, grease, free product, other hard to remove materials are encountered probes and exposed tapes should be washed in hot soapy water.

5.2.1.3 Miscellaneous Equipment

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

Coolers/Shipping Containers employed to ship samples are received from the lab in a variety of conditions from marginal to extremely poor. Coolers should be evaluated prior to use for

- Structural integrity – Coolers missing handles or having breaks within the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples will not be attempted and request a replacement unit.

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

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

If these measures fail to clean the cooler to an acceptable level, remove the unit from use as a shipping container and notify the laboratory to provide a replacement unit.

5.2.2 Down-Hole Drilling Equipment

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

1) Remove all soils to the extent possible using shovels, scrapers, etc. to remove loose soils.
2) Through a combination of scrubbing using soap and water and/or steam cleaning remove visible dirt/soils.
3) Rinse with tap water.
4) Rinse equipment with pesticide grade isopropanol
5) To the extent possible allow components to air dry.
6) Wrap or cover equipment in clear plastic until it is time to be used.

5.2.3 Soil/Sediment Sampling Equipment

This consists of soil sampling equipment including but not limited to hand augers, stainless steel trowels/spoons, bowls, dredges, scoops, split spoons, Macro Core samplers, etc.

1) Remove all soils to the extent possible.

2) Through a combination of scrubbing using soap and water and/or steam cleaning remove visible dirt/soils.

3) Rinse with tap water.

4) Rinse equipment with pesticide grade isopropanol

5) Rinse with deionized water

6) To the extent possible allow components to air dry.

7) If the device is to be used immediately, screen with a PID/FID to insure all solvents (if they were used) and trace contaminants have been adequately removed.

8) Once these devices have been dried wrap in aluminum foil for storage until it is time to be used.

5.3 Contact Waste/Materials

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

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

5.3.1 Decontamination Solutions

All waste decontamination solutions and rinses must be assumed to contain the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. The waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.

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

5.4 Decontamination Evaluation

Determining the effectiveness of the decontamination process will be accomplished in the following manner

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

- Instrument Screening – A PID and/or an FID should be used to evaluate the presence of the contaminants or solvents used in the cleaning process. The air intake of the instrument should be passed over the article to be evaluated. A positive detection requires a repeat the decontamination process. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instruments capabilities.

- Rinsate Blanks – It is recommended that Rinsate samples be collected to
  - Evaluate the decontamination procedure representing different equipment applications (pumps versus drilling equipment) and different decontamination applications.
  - Single use disposable equipment – The number of samples should represent different types of equipment as well as different Lot Numbers of single use articles.

The collection and the frequency of collection of rinsate samples are as follows:

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

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and in an effort to avoid using a contaminated batch of single use articles. It is recommended that a follow up sample be collected during the execution of the project to insure those conditions do not change. Lastly, rinsate samples collection may be driven by types of and/or contaminant levels. Hard to remove contaminants, oils/greases, some PAHs/PCBs, etc. may also support the collection of additional rinsates due to the obvious challenges to the decontamination process. This is a field consideration to be determined by the FOL.

S9 SA-6.3 FIELD DOCUMENTATION

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1.0 PURPOSE

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

2.0 SCOPE

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

3.0 GLOSSARY

None

4.0 RESPONSIBILITIES

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

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

5.0 PROCEDURES

5.1 Site Logbook

5.1.1 General

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

- All field personnel present
- Arrival/departure of site visitors
- Time and date of H&S training
- Arrival/departure of equipment
- Time and date of equipment calibration
- Start and/or completion of borehole, trench, monitoring well installation, etc.
- Daily onsite activities performed each day
- Sample pickup information
- Health and Safety issues (level of protection observed, etc.)
- Weather conditions

A site logbook shall be maintained for each project. The site logbook shall be initiated at the start of the first onsite activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day

that onsite activities take place which involve Tetra Tech NUS or subcontractor personnel. Upon completion of the fieldwork, the site logbook must become part of the project's central file.

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

- Project name
- Tetra Tech NUS project number
- Sequential book number
- Start date
- End date

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

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

All logbook, notebook, and log sheet entries shall be made in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, the entry shall be crossed out with a single strike mark, and initialed and dated. At the completion of entries by any individual, the logbook pages used must be signed and dated. The site logbook must also be signed by the Field Operations Leader at the end of each day.

5.1.2 Photographs

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

5.2 Field Notebooks

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

5.3 Field Forms

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

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

5.3.1.1 Sample Log Sheet

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

5.3.1.2 Sample Label

A typical sample label is illustrated in Attachment B. Adhesive labels must be completed and applied to every sample container. Sample labels can usually be obtained from the appropriate Program source electronically generated in-house, or are supplied from the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record Form

The Chain-of-Custody (COC) Record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site. One carbonless copy of the completed COC form is retained by the field crew, one copy is sent to the Project Manager (or designee), while the original is sent to the laboratory. The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing vials for VOC analysis or the cooler with the air bill attached. The air bill should then state how many coolers are included with that shipment. An example of a Chain-of-Custody Record form is provided as Attachment C. Once the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed COC form (any discrepancies between the sample labels and COC form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the Tetra Tech NUS Project Manager). The COC form is signed and copied. The laboratory will retain the copy while the original becomes part of the samples' corresponding analytical data package.

5.3.1.4 Chain-of-Custody Seal

Attachment D is an example of a custody seal. The Custody seal is an adhesive-backed label. It is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. The COC seals are signed and dated by the sampler(s) and affixed across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). COC seals may be available from the laboratory; these seals may also be purchased from a supplier.

5.3.1.5 Geochemical Parameters Log Sheets

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

5.3.2 Hydrogeological and Geotechnical Forms

5.3.2.1 Groundwater Level Measurement Sheet

A Groundwater Level Measurement Sheet must be filled out for each round of water level measurements made at a site.

~~5.3.2.2 Data Sheet for Pumping Test~~

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

~~5.3.2.3 Packer Test Report Form~~

~~A Packer Test Report Form must be completed for each well upon which a packer test is conducted.~~

5.3.2.4 Boring Log

During the progress of each boring, a log of the materials encountered, operation and driving of casing, and location of samples must be kept. The Summary Log of Boring, or Boring Log is used for this purpose and must be completed for each soil boring performed. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a PID or FID), these readings must be entered on the boring log at the appropriate depth. The "Remarks" column can be used to subsequently enter the laboratory sample number, the concentration of key analytical results, or other pertinent information. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 Monitoring Well Construction Details Form

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

~~5.3.2.6 Test Pit Log~~

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

5.3.2.7 Miscellaneous Monitoring Well Forms

Monitoring Well Materials Certificate of Conformance should be used as the project directs to document all materials utilized during each monitoring well installation.

The Monitoring Well Development Record should be used as the project directs to document all well development activities.

5.3.2.8 Miscellaneous Field Forms - QA and Checklists

Container Sample and Inspection Sheet should be used as the project directs each time a container (drum, tank, etc.) is sampled and/or inspected.

QA Sample Log Sheet should be used at the project directs each time a QA sample is colleted, such as Rinsate Blank, Source Blank, etc.

Field Task Modification Request (FTMR) will be prepared for all deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Copies of all FTMRs will be maintained with the onsite planning documents and originals will be placed in the final evidence file.

The Field Project Daily Activities Check List and Field Project Pre-Mobilization Checklist should be used during both the planning and field effort to assure that all necessary tasks are planned for and completed. These two forms are not a requirement but a useful tool for most field work.

5.3.3 Equipment Calibration and Maintenance Form

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

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

5.4 Field Reports

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

5.4.1 Daily Activities Report

To provide timely oversight of onsite contractors, Daily Activities Reports are completed and submitted as described below.

5.4.1.1 Description

The Daily Activities Report (DAR) documents the activities and progress for each day's field work. This report must be filled out on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring which involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

5.4.1.2 Responsibilities

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

5.4.1.3 Submittal and Approval

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

5.4.2 Weekly Status Reports

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

It should be noted that in addition to summaries described herein, other summary reports may also be contractually required.

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

6.0 LISTING OF TETRA TECH NUS FIELD FORMS FOUND ON THE TTNUS INTRANET SITE. HTTP://INTRANET.TTNUS.COM CLICK ON FIELD LOG SHEETS

Groundwater Sample Log Sheet
Surface Water Sample Log Sheet
Soil/Sediment Sample Log Sheet
Container Sample and Inspection Sheet
Geochemical Parameters (Natural Attenuation)
Groundwater Level Measurement Sheet
Pumping Test Data Sheet
Packer Test Report Form
Boring Log
Monitoring Well Construction Bedrock Flush Mount
Monitoring Well Construction Bedrock Open Hole
Monitoring Well Construction Bedrock Stick Up
Monitoring Well Construction Confining Layer
Monitoring Well Construction Overburden Flush Mount
Monitoring Well Construction Overburden Stick Up
Test Pit Log
Monitoring Well Materials Certificate of Conformance
Monitoring Well Development Record

Daily Activities Record
Field Task Modification Request
Hydraulic Conductivity Test Data Sheet
Low Flow Purge Data Sheet
QA Sample Log Sheet
Equipment Calibration Log
Field Project Daily Activities Checklist
Field Project Pre-Mobilization Checklist

ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: _____ DATE: _____

SITE LEADER: _____

PERSONNEL:

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

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

ACTIVITIES:

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

_____

Field Operations Leader

ATTACHMENT B

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

ATTACHMENT C

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

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

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

CONTAINER TYPE PLASTIC (P) or GLASS (G)
PRESERVATIVE USED
TYPE OF ANALYSIS

DATE YEAR	TIME	SAMPLE ID	LOCATION ID	TOP DEPTH (FT)	BOTTOM DEPTH (FT)	MATRIX (GW, SO, SW, SD, QC, ETC.)	COLLECTION METHOD GRAP (G) COMP (C)	No. OF CONTAINERS									COMMENTS

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

COMMENTS

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

4/02R
FORM NO. TtNUS-001

ATTACHMENT D

CHAIN-OF-CUSTODY SEAL

CUSTODY SEAL	**CUSTODY SEAL**
Date	**Date**
Signature	**Signature**

S10 USEPA REGION 1 LOW-FLOW PURGING AND SAMPLING METHODS

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION I

LOW STRESS (low flow) PURGING AND SAMPLING PROCEDURE FOR THE COLLECTION OF GROUND WATER SAMPLES FROM MONITORING WELLS

July 30, 1996
Revision 2

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION I

LOW STRESS (low flow) PURGING AND SAMPLING PROCEDURE FOR THE COLLECTION OF GROUND WATER SAMPLES FROM MONITORING WELLS

I. SCOPE & APPLICATION

This standard operating procedure (SOP) provides a general framework for collecting ground water samples that are indicative of mobile organic and inorganic loads at ambient flow conditions (both the dissolved fraction and the fraction associated with mobile particulates). The SOP emphasizes the need to minimize stress by low water-level drawdowns, and low pumping rates (usually less than 1 liter/min) in order to collect samples with minimal alterations to water chemistry. This SOP is aimed primarily at sampling monitoring wells that can accept a submersible pump and have a screen, or open interval length of 10 feet or less (this is the most common situation). However, this procedure is flexible and can be used in a variety of well construction and ground-water yield situations. Samples thus obtained are suitable for analyses of ground water contaminants (volatile and semi-volatile organic analytes, pesticides, PCBs, metals and other inorganics), or other naturally occurring analytes.

This procedure does not address the collection of samples from wells containing light or dense non-aqueous phase liquids (LNAPLs and DNAPLs). For this the reader may wish to check: Cohen, R.M. and J.W. Mercer, 1993, DNAPL Site Evaluation; C.K. Smoley (CRC Press), Boca Raton, Florida and U.S. Environmental Protection Agency, 1992, RCRA Ground-Water Monitoring: Draft Technical Guidance; Washington, DC (EPA/530-R-93-001).

The screen, or open interval of the monitoring well should be optimally located (both laterally and vertically) to intercept existing contaminant plume(s) or along flowpaths of potential contaminant releases. It is presumed that the analytes of interest move (or potentially move) primarily through the more permeable zones within the screen, or open interval.

Use of trademark names does not imply endorsement by U.S.EPA but is intended only to assist in identification of a specific type of device.

Proper well construction and development cannot be overemphasized, since the use of installation techniques that are appropriate to the hydrogeologic setting often prevents "problem well" situations from occurring. It is also recommended that as part of development or redevelopment the well should be tested to determine the appropriate pumping rate to obtain stabilization of field indicator parameters with minimal drawdown in shortest amount of time. With this information field crews can then conduct purging and sampling in a more expeditious manner.

The mid-point of the saturated screen length (which should not exceed 10 feet) is used by convention as the location of the pump intake. However, significant chemical or permeability contrast(s) within the screen may require additional field work to determine the optimum vertical location(s) for the intake, and appropriate pumping rate(s) for purging and sampling more localized target zone(s). Primary flow zones (high(er) permealability and/or high(er) chemical concentrations) should be identified in wells with screen lengths longer than 10 feet, or in wells with open boreholes in bedrock. Targeting these zones for water sampling will help insure that the low stress procedure will not underestimate contaminant concentrations. The Sampling and Analysis Plan must provide clear instructions on how the pump intake depth(s) will be selected, and reason(s) for the depth(s) selected.

Stabilization of indicator field parameters is used to indicate that conditions are suitable for sampling to begin. Achievement of turbidity levels of less than 5 NTU and stable drawdowns of less than 0.3 feet, while desirable, are not mandatory. Sample collection may still take place provided the remaining criteria in this procedure are met. If after 4 hours of purging indicator field parameters have not stabilized, one of 3 optional courses of action may be taken: a) continue purging until stabilization is achieved, b) discontinue purging, do not collect any samples, and record in log book that stabilization could not be achieved (documentation must describe attempts to achieve stabilization) c) discontinue purging, collect samples and provide full explanation of attempts to achieve stabilization (note: there is a risk that the analytical data obtained, especially metals and strongly hydrophobic organic analytes, may not meet the sampling objectives).

2

Changes to this SOP should be proposed and discussed when the site Sampling and Analysis Plan is submitted for approval. Subsequent requests for modifications of an approved plan must include adequate technical justification for proposed changes. All changes and modifications must be approved before implementation in field.

II. EQUIPMENT

A. Extraction device

Adjustable rate, submersible pumps are preferred (for example, centrifugal or bladder pump constructed of stainless steel or

Teflon).

Adjustable rate, peristaltic pumps (suction) may be used with caution. Note that EPA guidance states: "Suction pumps are not recommended because they may cause degassing, pH modification, and loss of volatile compounds" (EPA/540/P-87/001, 1987, page 8.5-11).

The use of inertial pumps is discouraged. These devices frequently cause greater disturbance during purging and sampling and are less easily controlled than the pumps listed above. This can lead to sampling results that are adversely affected by purging and sampling operations, and a higher degree of data variability.

B. Tubing

Teflon or Teflon lined polyethylene tubing are preferred when sampling is to include VOCs, SVOCs, pesticides, PCBs and inorganics.

PVC, polypropylene or polyethylene tubing may be used when collecting samples for inorganics analyses. However, these materials should be used with caution when sampling for organics. If these materials are used, the equipment blank (which includes the tubing) data must show that these materials do not add contaminants to the sample.

Stainless steel tubing may be used when sampling for VOCs, SVOCs, pesticides, and PCBs. However, it should be used with caution when sampling for metals.

The use of 1/4 inch or 3/8 inch (inner diameter) tubing is preferred. This will help ensure the tubing remains liquid filled when operating at very low pumping rates.

Pharmaceutical grade (Pharmed) tubing should be used for the section around the rotor head of a peristaltic pump, to minimize gaseous diffusion.

C. Water level measuring device(s), capable of measuring to 0.01 foot accuracy (electronic "tape", pressure transducer). Recording pressure transducers, mounted above the pump, are especially helpful in tracking water levels during pumping operations, but their use must include check measurements with a water level "tape" at the start and end of each record.

D. Flow measurement supplies (e.g., graduated cylinder and stop watch).

E. Interface probe, if needed.

F. Power source (generator, nitrogen tank, etc.). If a gasoline generator is used, it must be located downwind and at least 30 feet from the well so that the exhaust fumes do not contaminate the samples.

G. Indicator field parameter monitoring instruments - pH, Eh, dissolved oxygen (DO), turbidity, specific conductance, and temperature. Use of a flow-through-cell is required when measuring all listed parameters, except turbidity. Standards to perform field calibration of instruments. Analytical methods are listed in 40 CFR 136, 40 CFR 141, and SW-846. For Eh measurements, follow manufacturer's instructions.

H. Decontamination supplies (for example, non-phosphate detergent, distilled/deionized water, isopropyl alcohol, etc.).

I. Logbook(s), and other forms (for example, well purging forms).

J. Sample Bottles.

K. Sample preservation supplies (as required by the analytical methods).

L. Sample tags or labels.

M. Well construction data, location map, field data from last sampling event.

N. Well keys.

O. Site specific Sample and Analysis Plan/Quality Assurance Project Plan.

P. PID or FID instrument (if appropriate) to detect VOCs for health and safety purposes, and provide qualitative field evaluations.

III. PRELIMINARY SITE ACTIVITIES

Check well for security damage or evidence of tampering, record pertinent observations.

Lay out sheet of clean polyethylene for monitoring and sampling equipment.

Remove well cap and immediately measure VOCs at the rim of the well with a PID or FID instrument and record the reading in the field logbook.

If the well casing does not have a reference point (usually a V-cut or indelible mark in the well casing), make one. Describe its location and record the date of the mark in the logbook.

A synoptic water level measurement round should be performed (in the shortest possible time) before any purging and sampling activities begin. It is recommended that water level depth (to 0.01 ft.) and

total well depth (to 0.1 ft.) be measured the day before, in order to allow for re-settlement of any particulates in the water column. If measurement of total well depth is not made the day before, it should not be measured until after sampling of the well is complete. All measurements must be taken from the established referenced point. Care should be taken to minimize water column disturbance.

Check newly constructed wells for the presence of LNAPLs or DNAPLs before the initial sampling round. If none are encountered, subsequent check measurements with an interface probe are usually not needed unless analytical data or field head space information signal a worsening situation. Note: procedures for collection of LNAPL and DNAPL samples are not addressed in this SOP.

IV. PURGING AND SAMPLING PROCEDURE

Sampling wells in order of increasing chemical concentrations (known or anticipated) is preferred.

1. Install Pump

Lower pump, safety cable, tubing and electrical lines slowly (to minimize disturbance) into the well to the midpoint of the zone to be sampled. The Sampling and Analysis Plan should specify the sampling depth, or provide criteria for selection of intake depth for each well (see Section I). If possible keep the pump intake at least two feet above the bottom of the well, to minimize mobilization of particulates present in the bottom of the well. Collection of turbid free water samples may be especially difficult if there is two feet or less of standing water in the well.

2. Measure Water Level

Before starting pump, measure water level. If recording pressure transducer is used-initialize starting condition.

3. Purge Well

3a. Initial Low Stress Sampling Event

Start the pump at its lowest speed setting and slowly increase the speed until discharge occurs. Check water level. Adjust pump speed until there is little or no water level drawdown (less than 0.3 feet). If the minimal drawdown that can be achieved exceeds 0.3 feet but remains stable, continue purging until indicator field parameters stabilize.

Monitor and record water level and pumping rate every three to five minutes (or as appropriate) during purging. Record any pumping rate adjustments (both time and flow rate). Pumping rates should, as needed, be reduced to the minimum capabilities of the pump (for example, 0.1 - 0.4 l/min) to ensure stabilization of indicator

parameters. Adjustments are best made in the first fifteen minutes of pumping in order to help minimize purging time. During pump start-up, drawdown may exceed the 0.3 feet target and then "recover" as pump flow adjustments are made. Purge volume calculations should utilize stabilized drawdown value, not the initial drawdown. Do not allow the water level to fall to the intake level (if the static water level is above the well screen, avoid lowering the water level into the screen). The final purge volume must be greater than the stabilized drawdown volume plus the extraction tubing volume.

Wells with low recharge rates may require the use of special pumps capable of attaining very low pumping rates (bladder, peristaltic), and/or the use of dedicated equipment. If the recharge rate of the well is lower than extraction rate capabilities of currently manufactured pumps and the well is essentially dewatered during purging, then the well should be sampled as soon as the water level has recovered sufficiently to collect the appropriate volume needed for all anticipated samples (ideally the intake should not be moved during this recovery period). Samples may then be collected even though the indicator field parameters have not stabilized.

3b. Subsequent Low Stress Sampling Events

After synoptic water level measurement round, check intake depth and drawdown information from previous sampling event(s) for each well. Duplicate, to the extent practicable, the intake depth and extraction rate (use final pump dial setting information) from previous event(s). Perform purging operations as above.

4. Monitor Indicator Field Parameters

During well purging, monitor indicator field parameters (turbidity, temperature, specific conductance, pH, Eh, DO) every three to five minutes (or less frequently, if appropriate). Note: during the early phase of purging emphasis should be put on minimizing and stabilizing pumping stress, and recording those adjustments. Purging is considered complete and sampling may begin when all the above indicator field parameters have stabilized. Stabilization is considered to be achieved when three consecutive readings, taken at three (3) to five (5) minute intervals, are within the following limits:

turbidity (10% for values greater than 1 NTU),
DO (10%),
specific conductance (3%),
temperature (3%),
pH (± 0.1 unit),
ORP/Eh (± 10 millivolts).

All measurements, except turbidity, must be obtained using a flow-through-cell. Transparent flow-through-cells are preferred, because they allow field personnel to watch for particulate build-up within the cell. This build-up may affect indicator field parameter values

measured within the cell and may also cause an underestimation of turbidity values measured after the cell. If the cell needs to be cleaned during purging operations, continue pumping and disconnect cell for cleaning, then reconnect after cleaning and continue monitoring activities.

The flow-through-cell must be designed in a way that prevents air bubble entrapment in the cell. When the pump is turned off or cycling on/off (when using a bladder pump), water in the cell must not drain out. Monitoring probes must be submerged in water at all times. If two flow-through-cells are used in series, the one containing the dissolved oxygen probe should come first (this parameter is most susceptible to error if air leaks into the system).

5. Collect Water Samples

Water samples for laboratory analyses must be collected before water has passed through the flow-through-cell (use a by-pass assembly or disconnect cell to obtain sample).

VOC samples should be collected first and directly into pre-preserved sample containers. Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

During purging and sampling, the tubing should remain filled with water so as to minimize possible changes in water chemistry upon contact with the atmosphere. It is recommended that 1/4 inch or 3/8 inch (inside diameter) tubing be used to help insure that the sample tubing remains water filled. If the pump tubing is not completely filled to the sampling point, use one of the following procedures to collect samples: (1) add clamp, connector (Teflon or stainless steel) or valve to constrict sampling end of tubing; (2) insert small diameter Teflon tubing into water filled portion of pump tubing allowing the end to protrude beyond the end of the pump tubing, collect sample from small diameter tubing; (3) collect non-VOC samples first, then increase flow rate slightly until the water completely fills the tubing, collect sample and record new drawdown, flow rate and new indicator field parameter values.

Add preservative, as required by analytical methods, to samples immediately after they are collected if the sample containers are not pre-preserved. Check analytical methods (e.g. EPA SW-846, water supply, etc.) for additional information on preservation. Check pH for all samples requiring pH adjustment to assure proper pH value. For VOC samples, this will require that a test sample be collected during purging to determine the amount of preservative that needs to be added to the sample containers prior to sampling.

If determination of filtered metal concentrations is a sampling objective, collect filtered water samples using the same low flow procedures. The use of an in-line filter is required, and the filter

size (0.45 um is commonly used) should be based on the sampling objective. Pre-rinse the filter with approximately 25 - 50 ml of ground water prior to sample collection. Preserve filtered water sample immediately. Note: filtered water samples are not an acceptable substitute for unfiltered samples when the monitoring objective is to obtain chemical concentrations of total mobile contaminants in ground water for human health risk calculations.

Label each sample as collected. Samples requiring cooling (volatile organics, cyanide, etc.) will be placed into a cooler with ice or refrigerant for delivery to the laboratory. Metal samples after acidification to a pH less than 2 do not need to be cooled.

6. Post Sampling Activities

If recording pressure transducer is used, remeasure water level with tape.

After collection of the samples, the pump tubing may either be dedicated to the well for resampling (by hanging the tubing inside the well), decontaminated, or properly discarded.

Before securing the well, measure and record the well depth (to 0.1 ft.), if not measured the day before purging began. Note: measurement of total well depth is optional after the initial low stress sampling event. However, it is recommended if the well has a "silting" problem or if confirmation of well identity is needed.

Secure the well.

V. DECONTAMINATION

Decontaminate sampling equipment prior to use in the first well and following sampling of each subsequent well. Pumps will not be removed between purging and sampling operations. The pump and tubing (including support cable and electrical wires which are in contact with the well) will be decontaminated by one of the procedures listed below.

Procedure 1

The decontaminating solutions can be pumped from either buckets or short PVC casing sections through the pump or the pump can be disassembled and flushed with the decontaminating solutions. It is recommended that detergent and isopropyl alcohol be used sparingly in the decontamination process and water flushing steps be extended to ensure that any sediment trapped in the pump is removed. The pump exterior and electrical wires must be rinsed with the decontaminating solutions, as well. The procedure is as follows:

Flush the equipment/pump with potable water.

Flush with non-phosphate detergent solution. If the solution is recycled, the solution must be changed periodically.

Flush with potable or distilled/deionized water to remove all of the detergent solution. If the water is recycled, the water must be changed periodically.

Flush with isopropyl alcohol (pesticide grade). If equipment blank data from the previous sampling event show that the level of contaminants is insignificant, then this step may be skipped.

Flush with distilled/deionized water. The final water rinse must not be recycled.

Procedure 2

Steam clean the outside of the submersible pump.

Pump hot potable water from the steam cleaner through the inside of the pump. This can be accomplished by placing the pump inside a three or four inch diameter PVC pipe with end cap. Hot water from the steam cleaner jet will be directed inside the PVC pipe and the pump exterior will be cleaned. The hot water from the steam cleaner will then be pumped from the PVC pipe through the pump and collected into another container. Note: additives or solutions should not be added to the steam cleaner.

Pump non-phosphate detergent solution through the inside of the pump. If the solution is recycled, the solution must be changed periodically.

Pump potable water through the inside of the pump to remove all of the detergent solution. If the solution is recycled, the solution must be changed periodically.

Pump distilled/deionized water through the pump. The final water rinse must not be recycled.

VI. FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not compromised the quality of the ground water samples. All field quality control samples must be prepared the same as regular investigation samples with regard to sample volume, containers, and preservation. The following quality control samples shall be collected for each batch of samples (a batch may not exceed 20 samples). Trip blanks are required for the VOC samples at a frequency of one set per VOC sample cooler.

Field duplicate.

Matrix spike.

Matrix spike duplicate.

Equipment blank.

Trip blank (VOCs).

Temperature blank (one per sample cooler).

Equipment blank shall include the pump and the pump's tubing. If tubing is dedicated to the well, the equipment blank will only include the pump in subsequent sampling rounds.

Collect samples in order from wells with lowest contaminant concentration to highest concentration. Collect equipment blanks after sampling from contaminated wells and not after background wells.

Field duplicates are collected to determine precision of sampling procedure. For this procedure, collect duplicate for each analyte group in consecutive order (VOC original, VOC duplicate, SVOC original, SVOC duplicate, etc.).

If split samples are to be collected, collect split for each analyte group in consecutive order (VOC original, VOC split, etc.). Split sample should be as identical as possible to original sample.

All monitoring instrumentation shall be operated in accordance with EPA analytical methods and manufacturer's operating instructions. EPA analytical methods are listed in 40 CFR 136, 40 CFR 141, and SW-846 with exception of Eh, for which the manufacturer's instructions are to be followed. Instruments shall be calibrated at the beginning of each day. If a measurement falls outside the calibration range, the instrument should be re-calibrated so that all measurements fall within the calibration range. At the end of each day, check calibration to verify that instruments remained in calibration. Temperature measuring equipment, thermometers and thermistors, need not be calibrated to the above frequency. They should be checked for accuracy prior to field use according to EPA Methods and the manufacturer's instructions.

VII. FIELD LOGBOOK

A field log shall be kept to document all ground water field monitoring activities (see attached example matrix), and record all of the following:

Well identification.

Well depth, and measurement technique.

Static water level depth, date, time and measurement technique.

Presence and thickness of immiscible liquid (NAPL) layers and

detection method.

Pumping rate, drawdown, indicator parameters values, and clock time, at the appropriate time intervals; calculated or measured total volume pumped.

Well sampling sequence and time of each sample collection.

Types of sample bottles used and sample identification numbers.

Preservatives used.

Parameters requested for analysis.

Field observations during sampling event.

Name of sample collector(s).

Weather conditions.

QA/QC data for field instruments.

Any problems encountered should be highlighted.

Description of all sampling equipment used, including trade names, model number, diameters, material composition, etc.

VIII. DATA REPORT

Data reports are to include laboratory analytical results, QA/QC information, and whatever field logbook information is needed to allow for a full evaluation of data useability.

EXAMPLE (Minimum Requirements)

Page____of____

Well PURGING-FIELD WATER QUALITY MEASUREMENTS FORM

Location (Site/Facility Name)_____ Depth to _____/_____ of screen
Well Number_____ Date_____ (below MP) top bottom
Field Personnel_____ Pump Intake at (ft. below MP)_____
Sampling Organization_____ Purging Device; (pump type)_____
Identify MP

Clock Time 24 HR	Water Depth below MP ft	Pump Dial[1]	Purge Rate ml/min	Cum. Volume Purged liters	Temp. °C	Spec. Cond.[2] µS/cm	pH	ORP/ Eh[3] mv	DO mg/L	Turb-idity NTU	Comments

1. Pump dial setting (for example: hertz, cycles/min, etc).
2. µSiemens per cm(same as µmhos/cm)at 25 °C.
3. Oxidation reduction potential (stand in for Eh).

S11 USEPA REGION 1 DRAFT CALIBRATION OF FIELD INSTRUMENTS

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION 1

DRAFT CALIBRATION OF FIELD INSTRUMENTS
(temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction potential [ORP], and turbidity)

I. SCOPE & APPLICATION

The purpose of this standard operating procedure (SOP) is to provide a framework for calibrating field instruments used to measure water quality parameters for ground water and surface water. Water quality parameters include temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction potential [ORP], and turbidity. This SOP supplements, but does not replace, EPA analytical methods listed in 40 CFR 136 and 40 CFR 141 for temperature, dissolved oxygen, conductivity/specific conductance, pH and turbidity.

This SOP is written for instruments that utilize multiple probes (temperature, pH, dissolved oxygen, conductivity/specific conductance and/or oxidation/reduction potential [ORP]) and the probe readings for pH, dissolved oxygen, and specific conductance are automatically corrected for temperature. Communications to the instrument (programming and displaying the measurement values) are performed using a display/logger or a computer. Information sent to the instrument is entered through the keypad on the display/logger or computer. It is desirable that the display/logger or computer has data storage capabilities. If the instrument does not have a keypad, follow the manufacturer's instructions for entering information into the instrument.

For ground water monitoring, the instrument must be equipped with a flow-through-cell, and the display/logger or computer display screen needs to be large enough to simultaneously contain the readouts of each probe in the instrument. Turbidity is measured using a separate instrument because turbidity cannot be measured in a flow-through-cell. This procedure is applicable for use with the EPA Region 1 Low Stress (low flow) Purging and Sampling Procedure for the Collection of Ground Water Samples from Monitoring Wells.

II. GENERAL

All monitoring instruments must be calibrated before they are used to measure environmental samples. Part of the calibration is performed prior to the field event. For instrument probes that rely on the temperature sensor (pH, dissolved oxygen, conductivity/specific conductance, and

oxidation/reduction potential [ORP]), each temperature sensor needs to be checked for accuracy against a thermometer that is traceable to the National Institute of Standards and Technology (NIST) . Before any instrument is calibrated or used to perform environmental measurements, the instrument must stabilize (warm-up) according to manufacturer's instructions.

Most instruments will require at least two standards to bracket the expected measurement range, that is, one standard less than the expected value and one above. Calibration must be performed at the beginning of each sampling day prior to sample collection. To determine if the instruments have remained in calibration during transport to the sampling location, use one of the previously used standards as a check standard at the sampling location. If the check measurement does not agree with the initial calibration or to within the specifications of the instrument, then the instrument must be re-calibrated. When an environmental sample measurement falls outside the calibration range, the instrument must be recalibrated to bracket the new range before continuing measurements.

This SOP requires that the manufacturer's instruction manual (including the instrument specifications) accompany the instrument into the field.

III. CALIBRATION PROCEDURES

Prior to calibration, all instrument probes must be cleaned according to the manufacturer's instructions. Failure to perform maintenance (proper maintenance) can lead to erratic measurements.

Program the calibration instrument so that the following parameters to be measured will be displayed: temperature, percent dissolved oxygen, mg/l dissolved oxygen, conductivity, specific conductance, and pH.

The volume of the calibration solutions must be sufficient to cover both the probe and temperature sensor (see manufacturer's instructions for additional information).

When calibrating or measuring, make sure there are no air bubbles lodged between the probe and the solution.

TEMPERATURE

Most instrument manuals state there is no calibration of the temperature sensor, but the temperature sensor must be checked to determine its accuracy. This accuracy check is performed at least once per year and the accuracy check date/information is kept with the

instrument. If the accuracy check date/information is not included with the instrument or the last check was over a year, the temperature sensor accuracy needs to be checked at the beginning of the sampling event. If the instrument contains multiple temperature sensors, each sensor must be checked.

Verification Procedure

1. Allow a container filled with water to come to room temperature.

2. Place a thermometer that is traceable to the National Institute of Standards and Technology (NIST), and the instrument's temperature sensor into the water and wait for both temperature readings to stabilize.

3. Compare the two measurements. The instrument's temperature sensor must agree with the reference thermometer measurement within the accuracy of the sensor (usually ±0.15°C). If the measurements do not agree, the instrument may not be working properly and the manufacturer needs to be consulted.

pH (electrometric)

The pH of water is determined electrometrically using a glass electrode.

Choose the appropriate buffered standards that will bracket the expected values at the sampling locations. For groundwater samples the pH will usually be close to seven. Three standards are needed for the calibration: one at least seven, one at least two pH units below seven and the other at least two pH units above seven. For those instruments that will not accept three standards, the instrument will need to be recalibrated if the water sample's pH is outside the initial calibration range described by the two standards.

Calibration Procedure

1. Allow the buffered standards to equilibrate to the ambient temperature.

2. Fill calibration containers with the buffered standards so each standard will cover the pH probe and temperature sensor.

3. Remove probe from its storage container, rinse with distilled water, blot dry with soft tissue.

draft

4. Select monitoring/run mode. Immerse probe into the initial standard (e.g., pH 7).

5. Stir the standard until the readings stabilize. If the readings do not change within 30 seconds, select calibration mode and then select "[illegible]". Enter the [illegible] standard value into instrument. Select monitoring/run mode. The readings should [illegible] within manufacturer's specifications; if they change, re-calibrate. If readings [illegible] change after re-calibration, consult manufacturer.

6. Remove probe from the initial standard, rinse with distilled water, and blot dry.

7. Immerse probe into the second standard (e.g., pH 4). Repeat step 5.

8. Remove probe from the second standard, rinse with distilled water, and blot dry. If instrument only accepts two standards, the calibration is complete. Go to step 11. Otherwise continue.

9. Immerse probe in third buffered standard (e.g., pH 9) and repeat step 5.

10. Remove probe from the third standard, rinse with distilled water, and blot dry.

11. Select monitoring/run mode, if not already selected. To ensure that the initial calibration standard (pH 7) has not changed, immerse the probe into the initial standard [illegible] for the [illegible] to stabilize. The reading should read the initial standard value within [illegible] manufacturer specifications. If not, re-calibrate the instrument. If re-calibration [illegible] help, the calibration range may be too great. Reduce calibration range by using [illegible] that are closer together.

12. The calibration is complete. Place pH probe in its storage container.

DISSOLVED OXYGEN

Dissolved oxygen (DO) content in water is measured using a membrane electrode. The DO probe's membrane and electrolyte solution should be replaced prior to the sampling period. Failure to perform this step may lead to erratic measurements.

<u>Calibration Procedure</u>

1. Gently dry the temperature sensor according to manufacturer's instructions.

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2. Place a wet sponge or a wet paper towel on the bottom of the DO calibration container.

3. Place the DO probe into the container without the probe coming in contact with the wet sponge or paper towel. The probe must fit tightly into the container to prevent the escape of moisture evaporating from the sponge or towel.

4. Allow the confined air to become saturated with water vapor (saturation occurs in approximately 10 to 15 minutes). During this time, turn the instrument to allow the DO probe to warm-up. Select monitoring/run mode to view temperature readings. Readings must stabilize before continuing to the next step.

5. Select calibration mode; then select "DO".

6. Enter the local barometric pressure (usually in inches of mercury) for the sampling location into the instrument. This measurement must be determined from an on-site barometer. Do not use barometric pressure obtained from the local weather services unless the pressure is corrected for the elevation of the sampling location. [Note: inches of mercury times 25.4 mm/inch equals mm of mercury or consult Oxygen Solubility at Indicated Pressure chart attached to the SOP for conversion at selected pressures.]

7. The instrument should indicate that the calibration is in progress. The instrument will take approximately one minute to calibrate. After calibration, the instrument should display 100% saturation.

8. Select monitoring/run mode. Compare the DO mg/l reading to the Oxygen Solubility at Indicated Pressure chart attached to the SOP. The numbers should agree. If they do not agree to the accuracy of the instrument (usually ± 0.2 mg/L), repeat calibration. If this does not work, change the membrane and electrolyte solution.

9. Remove the probe from the container and place it into a 0.0 mg/L DO standard (see note). The standard must be filled to the top of its container and the DO probe must fit tightly into the standard's container (no head space). Check temperature readings. They must stabilize before continuing.

10. Wait until the "mg/l DO" readings have stabilized. The instrument should read 0.0 mg/L or to the accuracy of the instrument (usually ± 0.2 mg/L). If the instrument cannot reach these values, it will be necessary to clean the probe, and change the membrane and electrolyte solution. If this does not work, prepare a new 0.0 mg/L DO standard. If these measures do not work, contact manufacturer.

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~~Note: To prepare a zero mg/L DO standard follow the procedure stated in Standard Methods (Method 4500-O G). The method basically states to add excess sodium sulfite (until no more dissolves) and a trace amount of cobalt chloride to water. The standard container must be completely filled (no head space). This solution must be prepared prior to the sampling event. If some of the solution is lost during instrument calibration, add more water to the container so that the standard is stored with no head space.~~

SPECIFIC CONDUCTANCE

Conductivity is used to measure the ability of an aqueous solution to carry an electrical current. Specific conductance is the conductivity value corrected to 25°C.

Most instruments are calibrated against a single standard that is near, but below the specific conductance of the environmental samples. A second standard which is above the environmental sample specific conductance is used to check the linearity of the instrument in the range of measurements.

<u>Calibration Procedure</u>

1. Allow the calibration standard to equilibrate to the ambient temperature.

2. Remove the probe from the storage container, rinse the probe with a small amount of the conductivity specific conductance standard (discard the rinsate), and place the probe into the conductivity specific conductance standard.

3. Select monitoring/run mode. Wait until the probe temperature has stabilized.

4. Look up the conductivity value at this temperature from the conductivity versus temperature correction table usually found on the standard bottle or on the standard instruction sheet. You may need to interpolate the conductivity value between temperatures. Select calibration mode, then conductivity. Enter the temperature corrected conductivity value into the instrument.

5. Select monitoring/run mode. The reading should remain within manufacturer's specifications. If it does not, re-calibrate. If readings continue to change after re-calibration, consult manufacturer.

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6. Read the specific conductance on the instrument and compare the value to the specific conductance value on the standard. The instrument value should agree with the standard within the manufacturer's specifications. If not, re-calibrate. If the calibration does not correct the problem, the probe may need to be cleaned or serviced by the instrument manufacturer.

7. Remove probe from the standard, rinse the probe with a small amount of the second conductivity/specific conductance standard (discard the rinsate), and place the probe into the second conductivity/specific conductance standard. The second standard will serve to verify the linearity of the instrument. Read the specific conductance value from the instrument and compare the value to the specific conductance of the standard. The two values should agree within the specifications of the instrument. If they do not agree, re-calibrate. If readings do not compare, then the second standard may be outside the linear range of the instrument. Use a standard that is closer but above the first standard and repeat the verification. If values still do not compare, clean the probe or consult the manufacturer.

8. When monitoring ground water or surface water, use the specific conductance readings.

OXIDATION/REDUCTION POTENTIAL (ORP)

The oxidation/reduction potential is the electrometric difference measured in a solution between an inert indicator electrode and a suitable reference electrode. The electrometric difference is measured in millivolts and is temperature dependent.

<u>Calibration or Verification Procedure</u>

1. Allow the calibration standard (a Zobell solution) to equilibrate to ambient temperature.

2. Remove the probe from its storage container, and place it into the standard.

3. Select monitoring/run mode.

4. While stirring the standard, wait for the probe temperature to stabilize, then read the temperature.

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5. Look up the millivolt (mv) value at this temperature from the millivolt versus temperature correction table usually found on the standard bottle or the standard instruction sheet. You may need to interpolate millivolt values between temperatures. Select "calibration mode", then "ORP". Enter the temperature-corrected ORP value into the instrument.

6. Select monitoring/run mode. The readings should then run unchanged within manufacturer's specifications. If they change, re-calibrate. If readings continue to change after re-calibration, consult manufacturer.

7. If the instrument instruction manual states that the instrument is factory calibrated, then verify the factory calibration against the standard. If they do not agree within the specifications of the instrument, the instrument will need to be re-calibrated by the manufacturer.

TURBIDITY

The turbidity method is based upon a comparison of intensity of light scattered by a sample under defined conditions with the intensity of light scattered by a standard reference suspension. A turbidimeter is a nephelometer equipped with a visible light source for illuminating the sample and one or more photoelectric detectors positioned ninety degrees to the path of the light source.

Some instruments are only accepted standard. For these instruments, the standards will serve as check points.

<u>Calibration Procedures</u>

1. Allow the calibration standards to equilibrate at the ambient temperature. The use of commercially available polymer primary standards (AMCO-AEPA-1) is preferred, however, the standards can be prepared using Formazin according to the EPA analytical Method 180.1.

2. If the standard cuvette is not sealed, rinse a cuvette with deionized water. Shake the cuvette to remove as much water as possible. Do not wipe dry the inside of the cuvette because lint from the wipe may remain in the cuvette. Add the standard to the cuvette.

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3. Before performing the calibration procedure, make sure the cuvettes are not scratched and the outside surfaces are dry, free from fingerprints and dust. If the cuvette is scratched or dirty, discard or clean the cuvette respectively.

4. Zero the instrument by using either a zero or 0.02 NTU standard. A zero standard (approximately 0 NTU) can be prepared by passing distilled water through a 0.45 micron pore size membrane filter.

5. Using a standard in the range of 5 - 20 NTUs, calibrate according to manufacturer's instructions or verify calibration if instrument will not accept second standard. If verifying, the instrument should read standard value to within the specifications of the instrument. If the instrument has range of scales, check each range that will be used during the sampling event with a standard within that range.

7. Using a standard between 20 and 100 NTUs, calibrate according to manufacturer's instructions or verify calibration if instrument does not accept a third standard. If verifying, the instrument should read standard value to within the specifications of the instrument. If the instrument has range of scales, check each range that will be used with the proper standard for that scale.

IV. DATA MANAGEMENT AND RECORDS MANAGEMENT

All calibration records must be documented in the project's log book. At a minimum, include the instrument manufacturer, model number, instrument identification number, standards used to calibrate the instrument (including source), calibration date, and the instrument readings.

References

Standard Methods for the Examination of Water and Wastewater, 19th edition, 1995.

Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised March 1983.

Turbidity - Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100, August 1993.

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Oxygen Solubility at Indicated Pressure

Temp.	Pressure (Hg)							
	760	755	750	745	740	735	730	mm
°C	29.92	29.72	29.53	29.33	29.13	28.94	28.74	in
0	14.57	14.47	14.38	14.28	14.18	14.09	13.99	mg/l
1	14.17	14.08	13.98	13.89	13.79	13.70	13.61	
2	13.79	13.70	13.61	13.52	13.42	13.33	13.24	
3	13.43	13.34	13.25	13.16	13.07	12-98	12.90	
4	13.08	12.99	12.91	12.82	12.73	12.65	12.56	
5	12.74	12.66	12.57	12.49	12.40	12.32	12.23	
6	12.42	12.34	12.26	12.17	12.09	12.01	11.93	
7	12.11	12.03	11.95	11.87	11.79	11.71	[illegible]	
8	11.81	11.73	11.65	11.57	11.50	11.42	[illegible]	
9	11.53	11.45	11.38	11.30	11.22	[illegible]	11.07	
10	11.28	11.19	11.11	11.04	10.96	[illegible]	10.81	
11	10.99	10.92	10.84	10.77	10.70	[illegible]	10.55	
12	10.74	10.67	10.60	10.53	10.45	[illegible]	[illegible]	
13	10.50	10.43	10.36	10.29	10.22	[illegible]	[illegible]	
14	10.27	10.20	10.13	10.06	10.00	9.93	[illegible]	
15	10.05	9.98	9.92	9.85	9.78	[illegible]	[illegible]	
16	9.83	9.76	9.70	9.63	9.57	[illegible]	9.43	
17	9.63	9.57	9.50	9.44	9.37	[illegible]	9.24	
18	9.43	9.37	9.30	9.24	[illegible]	[illegible]	9.05	
19	9.24	9.18	9.12	9.05	[illegible]	[illegible]	[illegible]	
20	9.06	9.00	8.94	8.88	[illegible]	[illegible]	[illegible]	
21	8.88	8.82	8.76	8.70	8.64	[illegible]	[illegible]	
22	8.71	8.65	8.59	[illegible]	8.47	8.42	[illegible]	
23	8.55	8.49	8.43	[illegible]	[illegible]	8.26	[illegible]	
24	8.39	8.33	8.28	[illegible]	[illegible]	8.11	8.05	
25	8.24	8.18	[illegible]	8.07	[illegible]	7.96	7.90	
26	8.09	8.03	[illegible]	7.92	[illegible]	[illegible]	7.76	
27	7.95	7.90	[illegible]	7.79	[illegible]	[illegible]	7.62	
28	[illegible]	[illegible]	[illegible]	7.65	[illegible]	7.54	7.49	
[illegible]	[illegible]	[illegible]	[illegible]	7.52	[illegible]	7.42	7.36	
30	[illegible]	7.50	[illegible]	[illegible]	7.34	7.29	7.24	
31	[illegible]	[illegible]	[illegible]	[illegible]	7.22	7.16	7.11	
32	[illegible]	[illegible]	[illegible]	[illegible]	7.10	7.05	7.00	
33	[illegible]	[illegible]	7.08	[illegible]	6.98	6.93	6.88	
34	7.07	[illegible]	[illegible]	6.92	6.87	6.82	6.78	
[illegible]	6.95	[illegible]	[illegible]	6.80	6.76	6.71	6.66	
[illegible]	6.84	[illegible]	[illegible]	6.70	6.65	6.60	6.55	
37	6.73	[illegible]	6.64	6.59	6.54	6.49	6.45	
38	6.63	[illegible]	6.54	6.49	6.44	6.40	6.35	
39	6.52	[illegible]	6.43	6.38	6.35	6.29	6.24	
40	6.42	[illegible]	6.33	6.28	6.24	6.19	6.15	
41	[illegible]	6.27	6.23	6.18	6.14	6.09	6.05	
[illegible]	[illegible]	6.18	6.13	6.09	6.04	6.00	5.95	
[illegible]	[illegible]	6.09	6.04	6.00	5.95	5.91	5.87	
[illegible]	6.03	5.99	5.94	5.90	5.86	5.81	5.77	
45	5.94	5.90	5.85	5.81	5.77	5.72	5.68	

(Continued)

Source: Draft EPA Handbook of Methods for Acid Deposition Studies, Field Operations for Surface Water Chemistry, EPA/600/4-89/020, August 1989.

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Oxygen Solubility at Indicated Pressure (continued)

Temp.	Pressure (Hg)								
	725	720	715	710	705	700	695	690	mm
°C	28.54	28.35	28.15	27.95	27.76	27.56	27.36	27.17	in
0	13.89	13.80	13.70	13.61	13.51	13.41	13.32	13.22	mg/l
1	13.51	13.42	13.33	13.23	13.14	13.04	12.95	12.86	
2	13.15	13.06	12.07	12.88	12.79	12.69	12.60	12.51	
3	12.81	12.72	12.63	12.54	12.45	12.36	12.27	12.18	
4	12.47	12.39	12.30	12.21	12.13	12.04	11.95	11.87	
5	12.15	12.06	11.98	11.89	11.81	11.73	11.64	11.56	
6	11.84	11.73	11.68	11.60	11.51	11.43	11.35	11.27	
7	11.55	11.47	11.39	11.31	11.22	11.14	11.06	[illegible]	
8	11.26	11.18	11.10	11.02	10.95	10.87	[illegible]	[illegible]	
9	10.99	10.92	10.84	10.76	10.69	10.61	[illegible]	[illegible]	
10	10.74	10.66	10.59	10.51	10.44	[illegible]	10.29	[illegible]	
11	10.48	10.40	10.33	10.28	10.18	[illegible]	10.04	[illegible]	
12	10.24	10.17	10.10	10.02	9.95	[illegible]	9.81	9.46	
13	10.01	9.94	9.87	9.80	9.73	[illegible]	[illegible]	9.52	
14	9.79	9.72	9.65	9.68	9.51	[illegible]	[illegible]	9.31	
15	9.58	9.51	9.44	9.58	9.31	9.24	[illegible]	[illegible]	
16	9.37	9.30	9.24	9.17	9.11	[illegible]	[illegible]	[illegible]	
17	9.18	9.11	9.05	8.98	8.92	[illegible]	8.79	[illegible]	
18	8.99	8.92	8.86	8.80	8.73	[illegible]	8.61	[illegible]	
19	8.81	8.74	8.68	8.62	[illegible]	[illegible]	8.43	[illegible]	
20	8.63	8.57	8.51	8.45	[illegible]	[illegible]	8.27	[illegible]	
21	8.46	8.40	8.34	8.28	[illegible]	[illegible]	[illegible]	[illegible]	
22	8.30	8.24	8.18	8.12	8.06	[illegible]	[illegible]	[illegible]	
23	8.15	8.09	8.03	[illegible]	7.91	7.85	[illegible]	7.74	
24	7.99	7.94	7.88	[illegible]	[illegible]	7.71	[illegible]	7.59	
25	7.85	7.79	7.74	[illegible]	[illegible]	7.57	7.51	7.46	
26	7.70	7.65	[illegible]	7.54	[illegible]	7.43	7.37	7.32	
27	7.57	7.52	[illegible]	7.41	[illegible]	[illegible]	7.25	7.19	
28	7.44	7.38	[illegible]	7.28	[illegible]	[illegible]	7.12	7.06	
29	[illegible]	[illegible]	[illegible]	7.15	[illegible]	7.05	7.00	6.94	
[illegible]	[illegible]	[illegible]	[illegible]	7.03	[illegible]	6.93	6.88	6.82	
31	[illegible]	7.01	[illegible]	6.91	6.86	6.81	6.76	6.70	
32	[illegible]	6.90	[illegible]	[illegible]	6.70	6.70	6.64	6.59	
33	[illegible]	[illegible]	[illegible]	[illegible]	6.83	6.58	6.53	6.48	
34	[illegible]	[illegible]	6.63	[illegible]	6.53	6.48	6.43	6.38	
35	6.61	[illegible]	[illegible]	6.47	6.42	6.37	6.36	6.27	
[illegible]	6.51	[illegible]	[illegible]	6.36	6.31	6.27	6.22	6.17	
[illegible]	6.40	[illegible]	[illegible]	6.26	6.21	6.16	6.12	6.07	
38	6.30	[illegible]	6.21	6.16	6.12	6.07	6.02	5.98	
39	6.26	[illegible]	6.11	6.06	6.01	5.97	5.92	5.87	
40	6.10	[illegible]	6.01	5.96	5.92	5.86	5.83	5.78	
41	6.00	[illegible]	5.91	5.87	5.82	5.78	5.73	5.69	
42	5.91	5.86	5.82	5.77	5.73	5.69	5.64	5.60	
[illegible]	[illegible]	5.78	5.73	5.69	5.65	5.60	5.56	5.51	
[illegible]	[illegible]	5.68	5.64	5.59	5.55	5.51	5.46	5.42	
[illegible]	5.64	5.59	5.55	5.51	5.47	5.42	5.38	5.34	

Source: Draft EPA Handbook of Methods for Acid Deposition Studies, Field Operations for Surface Water Chemistry, EPA/600/4-89/020, August 1989.

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S12 SA-1.2 SURFACE WATER AND SEDIMENT SAMPLING

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1.0 PURPOSE

This Standard Operating Procedure (SOP) describes procedures and equipment commonly used for collecting environmental samples of surface water and aquatic sediment for either onsite examination and chemical testing or for offsite laboratory analysis.

2.0 SCOPE

The information presented in this document is applicable to all environmental sampling of surface waters (Section 5.3) and aquatic sediments (Section 5.5), except where the analyte(s) may interact with the sampling equipment. The collection of concentrated sludges or hazardous waste samples from disposal or process lagoons often requires methods, precautions, and equipment different from those described herein.

3.0 GLOSSARY

Analyte – Chemical or radiochemical material whose concentration, activity, or mass is measured.

Composite Sample – A sample representing a physical average of grab samples.

Environmental Sample – A quantity of material collected in support of an environmental investigation that does not require special handling or transport considerations as detailed in SOP SA-6.1.

Grab Sample – A portion of material collected to represent material or conditions present at a single unit of space and time.

Hazardous Waste Sample – A sample containing (or suspected to contain) concentrations of contaminants that are high enough to require special handling and/or transport considerations per SOP SA-6.1.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, initial sampling locations, and field procedures used in the collection of soil samples. The Project Manager also has the overall responsibility for seeing that all surface water and sediment sampling activities are properly conducted by appropriately trained personnel in accordance with applicable planning documents.

Field Operations Leader - This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that

custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is responsible for finalizing the locations for collection of surface water and sediment samples. The FOL is ultimately responsible for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan (HASP). This includes but is not be limited to performing air quality monitoring during sampling and boring and excavation activities, and ensuring that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO or SSO designee may also be required to advise the FOL on other safety-related matters regarding boring and sampling, such as mitigative measures to address potential hazards from hazardous objects or conditions.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP and other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, , container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

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

5.0 HEALTH AND SAFETY

Precautions to preserve the health and safety of field personnel implementing this SOP are distributed throughout. The following general hazards may also exist during field activities, and the means of avoiding them must be used to preserve the health and safety of field personnel:

Bridge/Boat Sampling – Potential hazards associated with this activity include:

- Traffic – one of the primary concerns as samplers move across a bridge because free space of travel is not often provided. Control measures should include:
  - When sampling from a bridge, if the samplers do not have at least 6 feet of free travel space or physical barriers separating them and the traffic patterns, the HASP will include a Traffic Control Plan.
  - The use of warning signs and high-visibility vests are required to warn oncoming traffic and to increase the visibility of sample personnel.
- Slips, trips, and falls from elevated surfaces are a primary concern. Fall protection shall be worn when or if samplers must lean over a rail to obtain sample material. A Fall Protection Competent

Person (in accordance with Occupational safety and Health Administration [OSHA] fall protection standards) must be assigned to ensure that fall protection is appropriately and effectively employed

- Water hazards/drowning – if someone enters the water from an elevated surface (such as a bridge or dock) and when sampling from a boat. To minimize this potential, personnel shall wear United states Coast Guard (USCG)-approved floatation devices, and the sampling crew must also have on hand a Type IV Throwable Personal Floatation Device with at least 90 feet of 3/8-inch rope. See Section 5.5.2 of this SOP.

- Within the HASP, provisions will also be provided concerning the requirement of a Safe Vessel Certification or the necessity to conduct a boat inspection prior to use. In addition, the HASP shall also specify requirements as to whether the operator must be certified as a commercial boat operator and whether members of the sampling team must have a state-specific safe boating certification.

Entering Water to Collect Samples – Several hazards are associated with this activity and can be mitigated as follows:

- Personnel must wear a USCG-approved Floatation Device (selected and identified in the HASP). The SSO shall ensure that the device selected is in acceptable condition and suitable for the individual using it. This includes consideration of the weight of the individual.

- Lifelines shall be employed from a point on the shore. This activity will always be conducted with a Buddy. See Section 6.5.2.

- Personnel shall carry a probe to monitor the bottom ahead of them for drop offs or other associated hazards.

- The person in the water shall exercise caution concerning the path traveled so that the lifeline does not become entangled in underwater obstructions such as logs, branches, stumps, etc., thereby restricting its effectiveness in extracting the person from the water.

- Personnel shall not enter waters on foot in situations where natural hazards including alligators, snakes, as well as sharks, gars, and other predators within inland waterways may exist.

- In all cases, working along and/or entering the water during high currents or flood conditions shall be prohibited.

- Personnel shall not enter bodies of water where known debris exists that could result in injuries from cuts and lacerations.

Sampling in marshes or tidal areas in some instances can be accomplished using an all-terrain vehicle (ATV). This is not the primary recommended approach because the vehicle may become disabled, or weather conditions or tidal changes could result in environmental damage as well as loss of the vehicle. The primary approach is recommended to be on foot where minimal disturbance would occur. The same precautions specified above with regard to sediment disturbance apply as well as the previously described safety concerns associated with natural hazards. The natural hazards include alligators, bees (nests in dead falls and tree trunks), snakes, etc. In addition, moving through and over this terrain is difficult and could result in muscle strain and slips, trips, and falls. Common sense dictates that the sampler selects the most open accessible route over moderate terrain. Move slowly and deliberately through challenging terrain to minimize falls. Mud boots or other supportive PPE should be considered and specified in the HASP to permit samplers to move over soft terrain with the least amount of effort. In these situations, it is also recommended, as the terrain allows, that supplies be loaded and transported in a sled over the soft ground.

Working in these areas, also recognize the following hazards and means of protection against them:

Insects are also a primary concern. These include mosquitoes, ticks, spiders, bees, ants, etc. The HASP will identify those particular to your area. Typical preventative measures include:

- Use insect repellant. Approval of various repellants should be approved by the Project Chemist or Project Manager.
- Wearing light-colored clothing to control heat load due to excessive temperatures. In addition, it makes it easier to detect crawling insects on your clothing.
- Taping pants to boots to deny access. Again, this is recommended to control access to the skin by crawling insects. Consultation with the Project Health and Safety Officer SSO/Health and Safety Manager is recommended under extreme heat loads because this will create conditions of heat stress.
- Performing a body check to remove insects. The quicker you remove ticks, the less likely they will become attached and transfer bacteria to your bloodstream. Have your Buddy check areas inaccessible to yourself. This includes areas such as the upper back and between shoulder blades where it is difficult for you to examine and even more difficult for you to remove.

Safety Reminder

If you are allergic to bee or ant stings, it is especially critical that you carry your doctor-recommended antidote with you in these remote sampling locations due to the extended time required to extract incapacitated individuals as well as the effort required to extract them. In these scenarios, instruct your Buddy in the proper administration of the antidote. In all cases, if you have received a sting, administer the antidote regardless of the immediate reaction, evacuate, and seek medical attention as necessary. The FOL and/or SSO will determine when and if you may return to the field based on the extent of the immune response and hazards or potential hazards identified in these locations. To the FOL and SSO, this is a serious decision you have to make as to whether to take someone vulnerable to these hazards into a remote location where you may not be able to carry them out. Consider it wisely.

Poisonous Plants – To minimize the potential of encountering poisonous plants in the field, at least one member of the field team needs to have basic knowledge of what these plants look like so that they can be recognized, pointed out to other field personnel, and avoided if at all possible. If the field team cannot avoid contact and must move through an area where these plants exist, the level of personal protective equipment (PPE) shall include Tyvek coveralls and enhanced decontamination procedures for the removal of oils from the tooling and/or equipment.

Temperature-Related Stress – Excessively cold temperatures may result in cold stress, especially when entering the water either intentionally or by accident. Provisions for combating this hazard should be maintained at the sample location during this activity. Excessively hot temperatures may result in heat stress especially in scenarios where equipment is packed through the marsh.

Because all of these activities are conducted outside, electrical storms are a significant concern. The following measures will be incorporated to minimize this hazard:

- Where possible, utilize commercial warning systems and weather alerts to detect storms moving into the area.
- If on or in the water, get out of the water. Move to vehicles or preferably into enclosed buildings with plumbing and wiring.
- Where warning systems are not available, follow the 30/30 Rule (*if there are less than 30 seconds between thunder and lightning, go inside for at least 30 minutes after the last thunder).*

See Section 4.0 of the Health and Safety Guidance Manual (HSGM) for additional protective measures.

6.0 PROCEDURES

6.1 Introduction

Collecting a representative sample of surface water or sediment may be difficult because of water movement, stratification, or heterogeneous distribution of the targeted analytes. To collect representative samples, one must standardize sampling methods related to site selection, sampling frequency, sample collection, sampling devices, and sample handling, preservation, and identification. Regardless of quality control applied during laboratory analyses and subsequent scrutiny of analytical data packages, reported data are no better than the confidence that can be placed in the representativeness of the samples. Consult Appendix C for guidance on sampling that should be considered during project planning and that may be helpful to field personnel.

6.1.1 Surface Water Sampling Equipment

The selection of sampling equipment depends on the site conditions and sample type to be acquired. In general, the most representative samples are obtained from mid-channel at a stream depth of 0.5 foot in a well-mixed stream; however, project-specific planning documents will address site-specific sampling requirements including sample collection points and sampling equipment. The most frequently used samplers include the following:

- ~~Peristaltic pump~~
- Bailer
- Dip sampler
- Weighted bottle
- ~~Hand pump~~
- ~~Kemmerer~~
- Depth-integrating sampler

The dip sampler and weighted bottle sampler are used most often, and detailed discussions for these devices and the Kemmerer sampler are addressed subsequently in this section.

The criteria for selecting a sampler include:

1. Disposability and/or easy decontamination.
2. Inexpensive cost (if the item is to be disposed).
3. Ease of operation.

4. Non-reactive/non-contaminating properties - Teflon-coated, glass, stainless-steel or polyvinyl chloride (PVC) sample chambers are preferred (in that order).

Measurements collected for each sample (grab or each aliquot collected for compositing) shall include but not be limited to:

- Specific conductance
- Temperature
- pH
- Dissolved oxygen

Sample measurements shall be conducted as soon as the sample is acquired. Measurement techniques described in SOP SA-1.1 shall be followed. All pertinent data and results shall be recorded in a field notebook or on sample log sheets (see Attachment A) or an equivalent electronic form(s). These analyses may be selected to provide information on water mixing/stratification and potential contamination. Various types of water bodies have differing potentials for mixing and stratification.

In general, the following equipment if necessary for obtaining surface water samples:

- Required sampling equipment, which may include a remote sampling pole, weighted bottle sampler, Kemmerer sampler, or other device.

- Real-time air monitoring instrument (e.g., PID, FID) as directed in the project-specific planning document.

- Required PPE as directed in the project-specific planning document, which may include:

  - Nitrile surgeon's or latex gloves (layered as necessary).

  - Safety glasses.

  - Other items identified on the Safe Work Permit that may be required based on location-specific requirements (e.g., hearing protection, steel-toed work boots, hard hat). These provisions will be listed in the HASP or addressed by the FOL and/or SSO.

> ***Safety Reminder***
> The use of latex products may elicit an allergic reaction in some people. Should this occur, remove the latex gloves, treat for an allergic reaction, and seek medical attention as necessary.

  - Required paperwork (see SOP SA-6.3 and Attachments A and B to this SOP).

  - Required decontamination equipment.

  - Required sample containers.

  - Sealable polyethylene bags (e.g., Ziploc® baggies).

  - Heavy-duty cooler.

  - Ice.

- Paper towels and garbage bags.
- Chain-of-custody records and custody seals.

Dip Sampling

Specific procedures for collecting a dip or grab sample of surface water can vary based on site-specific conditions (e.g., conditions near the shore and how closely a sampler can safely get to the shore). The general procedure for collecting a sample using a pole or directly from the water body is as follows:

1. If using a remote sampling pole, securely attach the appropriate sample container to a pole of sufficient length to reach the water to be sampled. Samples for volatile analysis should be collected first. Use PPE as described in the HASP. When sample containers are provided pre-preserved or if the pole cannot accommodate a particular sample container, use a dedicated, clean, unpreserved bottle/container for sampling and transfer to an appropriately preserved container.
2. Remove the cap. Do not place the cap on the ground or elsewhere where it might become contaminated.
3. Carefully dip the container into the water just below the surface (or as directed by project-specific planning documents), and allow the bottle to fill. Sample bottles for volatile analysis must be filled with no headspace. Avoid contacting the bottom of the water body because this will disturb sediment that may interfere with the surface water sample.
4. Retrieve the container and carefully replace the cap securely. If using a container other than the sample bottle, pour the water from that container into the sample bottle and replace the cap securely.
5. Use a clean paper towel to clean and dry the outside of the container.
6. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
7. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

Constituents measured in grab samples collected near the water surface are only indicative of conditions near the surface of the water and may not be a true representation of the total concentration distributed throughout the water column and in the cross section. Therefore, as possible based on site conditions, the sampler may be required to augment dip samples with samples that represent both dissolved and suspended constituents and both vertical and horizontal distributions.

CAUTION

In areas prone to natural hazards such as alligators and snakes, etc., always use a buddy as a watch. Always have and use a lifeline or throwable device to extract persons who could potentially fall into the water. Be attentive to the signs, possible mounds indicating nests, and possible slides into the water. Remember that although snakes are typically encountered on the ground, it is not unheard of to see them on low-hanging branches. Be attentive to your surroundings because these may indicate that hazards are nearby.

Weighted Bottle Sampling

A grab sample can also be collected using a weighted holder that allows a bottle to be lowered to any desired depth, opened for filling, closed, and returned to the surface. This allows discrete sampling with depth. Several of these samples can be combined to provide a vertical composite. Alternatively, an open bottle can be lowered to the bottom and raised to the surface at a uniform rate so that the bottle collects sample throughout the total depth and is just filled on reaching the surface. The resulting sample using either method will roughly approach what is known as a depth-integrated sample.

A closed weighted bottle sampler consists of glass or plastic bottle with a stopper, a weight and/or holding device, and lines to open the stopper and lower or raise the bottle. The general procedure for sampling with this device is as follows:

1. Gently lower the sampler to the desired depth so as not to remove the stopper prematurely (watch for bubbles).
2. When the desired depth is reached, pull out the stopper with a sharp jerk of the stopper line.
3. Allow the bottle to fill completely, as evidenced by the absence of air bubbles.
4. Raise the sampler and cap the bottle.
5. Use a paper towel to clean and dry the outside of the container. This bottle can be used as the sample container as long as the bottle is an approved container type.
6. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
7. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

Kemmerer Sampler

If samples are desired at a specific depth, and the parameters to be measured do not require a Teflon-coated sampler, a standard Kemmerer sampler may be used. The Kemmerer sampler is a brass, stainless steel or acrylic cylinder with rubber stoppers that leave the ends open while it is lowered in a vertical position (thus allowing free passage of water through the cylinder). A "messenger" is sent down the line when the sampler is at the designated depth to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill sample bottles. The general procedure for sampling with this device is as follows:

1. Gently lower the sampler to the desired depth.
2. When the desired depth is reached, send down the messenger to close the cylinder and then raise the sampler.
3. Open the sampler valve to fill each sample bottle (filling bottles for volatile analysis first).
4. Use a paper towel to clean and dry the outside of the container.
5. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
6. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

6.1.2 Surface Water Sampling Techniques

Samples collected during site investigations may be grab samples or composite samples. The following general procedures apply to various types of surface water collection techniques:

- If a clean, pre-preserved sample container is not used, rinse the sample container least once with the water to be sampled before the sample is collected. This is not applicable when sample containers are provided pre-preserved because doing so will wash some or all of the preservative out of the bottle.
- For sampling moving water, collect the farthest downstream sample first, and continue sample collection in an upstream direction. In general, work from zones suspected of low contamination to zones of high contamination.
- Take care to avoid excessive agitation of the water because loss of volatile constituents could result.
- When obtaining samples in 40 mL vials with septum-lined lids for volatile organics analysis, fill the container completely (with a meniscus) to exclude any air space in the top of the bottle and to be sure that the Teflon liner of the septum faces in after the vial is filled and capped. Turn the vial upside down and tap gently on your wrist to check for air bubbles. If air bubbles rise in the bottle, add additional sample volume to the container.
- Do not sample at the surface, unless sampling specifically for a known constituent that is immiscible and on top of the water. Instead, invert the sample container, lower it to the approximate depth, and hold it at about a 45-degree angle with the mouth of the bottle facing upstream.

6.2 Onsite Water Quality Testing

Onsite water quality testing shall be conducted as described in SOP SA-1.1.

6.3 Sediment Sampling

6.3.1 General

If composite surface water samples are collected, sediment samples are usually collected at the same locations as the associated surface water samples. If only one sediment sample is to be collected, the sampling location shall be approximately at the center of the water body, in a depositional area if possible based on sample location restraints (see below), unless the SAP states otherwise.

Generally, coarser-grained sediments are deposited near the headwaters of reservoirs. Bed sediments near the center of a water body will be composed of fine-grained materials that may, because of their lower porosity and greater surface area available for adsorption, contain greater concentrations of contaminants. The shape, flow pattern, bathymetry (i.e., depth distribution), and water circulation patterns must all be considered when selecting sediment sampling sites. In streams, areas likely to have sediment accumulation (e.g., bends, behind islands or boulders, quiet shallow areas or very deep, low-velocity areas) shall be sampled, in general, and areas likely to show net erosion (i.e., high-velocity, turbulent areas) and suspension of fine solid materials shall be generally avoided. Follow instructions in the SAP, as applicable.

Chemical constituents associated with bottom material may reflect an integration of chemical and biological processes. Bottom samples reflect the historical input to streams, lakes, and estuaries with

respect to time, application of chemicals, and land use. Bottom sediments (especially fine-grained material) may act as a sink or reservoir for adsorbed heavy metals and organic contaminants (even if water column concentrations are less than detection limits). Therefore, it is important to minimize the loss of low-density "fines" during any sampling process.

Samples collected for volatile organic compound (VOC) analysis must be collected prior to any sample homogenization. Regardless of the method used for collection, the aliquot for VOC analysis must be collected directly from the sampling device (hand auger bucket, scoop, trowel), to the extent practical. If a device such as a dredge is used, the aliquot should be collected after the sample is placed in the mixing container prior to mixing.

In some cases, the sediment may be soft and not lend itself to collection by plunging Encore™ or syringe samplers into the sample matrix. In these cases, it is appropriate to open the sampling device, (Encore™ barrel or syringe) prior to sample collection, and carefully place the sediment in the device, filling it fully with the required volume of sample.

On active or former military sites, ordnance items may be encountered in some work areas. Care should be exercised when handling site media (such as if unloading a dredge as these materials may be scooped up). If suspected ordnance items are encountered, stop work immediately, move to shore and notify the Project Manager and Health and Safety Manager.

All relevant information pertaining to sediment sampling shall be documented as applicably described in SOP SA-6.3 and Attachment B or an equivalent electronic form.

6.3.2 Sampling Equipment and Techniques for Bottom Materials

A bottom-material sample may consist of a single scoop or core, or may be a composite of several individual samples in the cross section. Sediment samples may be obtained using onshore or offshore techniques.

SAFETY REMINDER

The following health and safety provisions apply when working on/over/near water:

- At least two people are required to be present at the sampling location in situations where the water depth and/or movement deem it necessary, each wearing a USCG-approved Personal Flotation Devices

- A minimum of three people are required if *any* of the following conditions are anticipated or observed:

  - Work in a waterway that is turbulent *or* swift that could sweep a sampler down stream should he or she fall in accidentally.

  - The underwater walking surface (e.g., stream/river bed) is suspected or observed to involve conditions that increase the potential for a worker to fall into the water. Examples include large/uneven rocks or boulders, dense mud or sediment that could entrap worker's feet, etc.

  - Waterway is tidal, and conditions such as those listed above could rapidly change.

The third person in the above condition must be equipped and prepared to render emergency support [e.g., lifeline, tethered Personal Flotation Device (Throwable Type IV, life saver), skiff, means to contact external emergency response support, etc.]

The following samplers may be used to collect sediment samples:

- Scoop sampler
- Dredge samplers
- Coring samplers

Each type of sampler is discussed below.

In general, the following equipment if necessary for obtaining sediment samples:

- Required sampling equipment, which may include a scoop sampler, dredge sampler, coring sampler, or stainless steel or pre-cleaned disposable trowel.
- Stainless bowl or pre-cleaned disposable bowl to homogenize sample.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in the project-specific planning document.
- Required PPE as directed in the project-specific planning document, which may include:
  - Nitrile surgeon's or latex gloves (layered as necessary).
  - Safety glasses.
  - Other items identified on the Safe Work Permit that may be required based on location-specific requirements (e.g., hearing protection, steel-toed work boots, hard hat). These provisions will be listed in the HASP or addressed by the FOL and/or SSO.
  - Required paperwork (see SOP SA-6.3 and Attachments A and B to this SOP).
  - Required decontamination equipment.
  - Required sample containers.
  - Sealable polyethylene bags (e.g., Ziploc® baggies).
  - Heavy-duty cooler.
  - Ice.
  - Paper towels and garbage bags.
  - Chain-of-custody records and custody seals.

Scoop Sampler

A scoop sampler consists of a pole to which a jar or scoop is attached. The pole may be made of bamboo, wood, PVC, or aluminum and be either telescoping or of fixed length. The scoop or jar at the end of the pole is usually attached using a clamp.

If the water body can be sampled from the shore or if the sampler can safely wade to the required location, the easiest and best way to collect a sediment sample is to use a scoop sampler. Scoop sampling also reduces the potential for cross-contamination. The general scoop sampling procedure is as follows:

1. Reach over or wade into the water body.

2. While facing upstream (into the current), scoop the sampler along the bottom in an upstream direction. Although it is very difficult not to disturb fine-grained materials at the sediment-water interface when using this method, try to keep disturbances to a minimum.

Dredge Samplers

Dredges are generally used to sample sediments that cannot easily be obtained using coring devices (e.g., coarse-grained or partially cemented materials) or when large quantities of sample are required. Dredges generally consist of a clam shell arrangement of two buckets. The buckets may either close upon impact or be activated by use of a "messenger." Some dredges are heavy and may require use of a winch and crane assembly for sample retrieval. The three major types of dredges are Peterson, Eckman and Ponar.

The Peterson dredge is used when the bottom is rocky, in very deep water, or when the flow velocity is high. The Peterson dredge shall be lowered very slowly as it approaches bottom, because it can force out and miss lighter materials if allowed to drop freely.

The Eckman dredge has only limited usefulness. It performs well where bottom material is unusually soft, as when covered with organic sludge or light mud. It is unsuitable, however, for sandy, rocky, and hard bottoms and is too light for use in streams with high flow velocities.

The Ponar dredge is a Peterson dredge modified by the addition of side plates and a screen on the top of the sample compartment. The screen over the sample compartment permits water to pass through the sampler as it descends, thus reducing the "shock wave." The Ponar dredge is easily operated by one person in the same fashion as the Peterson dredge. The Ponar dredge is one of the most effective samplers for general use on all types of substrates.

The general procedure for using dredge samplers is as follows:

1. Gently lower the dredge to the desired depth.

2. When the desired depth is reached, send the messenger down to cable to close the cylinder and then carefully raise the sampler.

3. Open the sampler to retrieve the sediment.

4. Transfer the sediment to the bowl in which it will be homogenized. Fill the sample bottle(s) for volatile analysis *prior to* homogenization. Homogenize the remainder of the sediment collected.

5. Fill the containers for all analyses other and VOCs.

6. Use a paper towel to clean and dry the outside of each container.

7. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

SAFETY REMINDER

Safety concerns using these dredges include lifting hazards, pinches, and compressions (several pinch points exist within the jaws and levers). In all cases, handle the dredge by the rope to avoid capturing fingers/hands.

Coring Samplers

Coring samplers are used to sample vertical columns of sediment. Many types of coring devices have been developed depending on the depth of water from which the sample is to be obtained, the nature of the bottom material, and the length of core to be collected. They vary from hand-push tubes to electronic vibrational core tube drivers.

Coring devices are particularly useful in pollutant monitoring because turbulence created by descent through the water is minimal, thus the fines at the sediment-water interface are only minimally disturbed. The sample is withdrawn intact, permitting the removal of only those layers of interest.

In shallow, wadeable waters, the use of a core liner or tube manufactured of Teflon or plastic is recommended for the collection of sediment samples. Caution should be exercised not to disturb the bottom sediments when the sample is obtained by wading in shallow water. The general procedure to collecting a sediment sample with a core tube is as follows:

1. Push the tube into the substrate until 4 inches or less of the tube is above the sediment-water interface. When sampling hard or coarse substrates, a gentle rotation of the tube while it is being pushed will facilitate greater penetration and decrease core compaction.

2. Cop the top of the tube to provide suction and reduce the chance of losing the sample.

3. Slowly extract the tube so as not to lose sediment from the bottom of the tube. Cap the bottom of the tube before removing it from the water. This will also help to minimize loss of sample.

4. Transfer the sediment to the bowl in which it will be homogenized. Fill the sample bottle(s) for volatile analysis prior to homogenization. Homogenize the remainder of the sediment collected.

5. Fill the containers for all analyses other and VOCs.

6. Use a paper towel to clean and dry the outside of each container.

7. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

In deeper, non-wadeable water bodies, sediment cores may be collected from a bridge or boat using different coring devices such as Ogeechee Sand Pounders, gravity cores, and vibrating coring devices.

All three devices utilize a core barrel with a core liner tube system. The core liners can be removed from the core barrel and replaced with a clean core liner after each sample. Before extracting the sediment from the coring tubes, the clear supernatant above the sediment-water interface in the core should be decanted from the tube. This is accomplished by turning the core tube to its side and gently pouring the liquid out until fine sediment particles appear in the waste liquid. Post-retrieval processing of samples is the same as above.

7.0 REFERENCES

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ATTACHMENT A
SURFACE WATER SAMPLE LOG SHEET

Tetra Tech NUS, Inc. **SURFACE WATER SAMPLE LOG SHEET**

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Stream
[] Spring
[] Pond
[] Lake
[] Other: _____
[] QA Sample Type: _____

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

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

SAMPLING DATA

	Color	pH	S.C.	Temp.	Turbidity	DO	Salinity	Other
Date:	Visual	Standard	mS/cm	Degrees C	NTU	mg/l	%	NA
Time:								
Depth:								
Method:								

SAMPLE COLLECTION INFORMATION

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS/NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

ATTACHMENT B
SOIL & SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

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

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

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

GRAB SAMPLE DATA:

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

COMPOSITE SAMPLE DATA:

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

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

APPENDIX C
GUIDANCE ON SAMPLING DESIGN AND SAMPLE COLLECTION

C.1 Defining the Sampling Program

Many factors are considered in developing a sampling program for surface water and/or sediment, including study objectives, accessibility, site topography, physical characteristics of the water body (e.g., flow and mixing), point and diffuse sources of contamination, and personnel and equipment available to conduct the study. For waterborne constituents, dispersion depends on vertical and lateral mixing within the body of water. For sediment, dispersion depends on bottom current or flow characteristics, sediment characteristics (e.g., density, size), and geochemical properties (that affect adsorption/desorption). The hydrogeologist developing the sampling plan must therefore know not only the mixing characteristics of streams and lakes but must also understand the role of fluvial-sediment transport, deposition, and chemical sorption.

C.1.1 Sampling Program Objectives

The scope of the sampling program must consider the sources and potential pathways for transport of contamination to or within a surface water body. Sources may include point sources (leaky tanks, outfalls, etc.) or nonpoint sources (e.g., contaminated runoff). The major pathways for surface water contamination (not including airborne deposition) are overland runoff, leachate influx to the water body, direct waste disposal (solid or liquid) into the water body, and groundwater flow influx from upgradient. The relative importance of these pathways, and therefore the design of the sampling program, is controlled by the physiographic and hydrologic features of the site, the drainage basin(s) that encompasses the site, and the history of site activities.

Physiographic and hydrologic features to be considered include slopes and runoff direction, areas of temporary flooding or pooling, tidal effects, artificial surface runoff controls such as berms or drainage ditches (and when they were constructed relative to site operation), and locations of springs, seeps, marshes, etc. In addition, the obvious considerations such as the locations of man-made discharge points to the nearest stream (intermittent or flowing), pond, lake, estuary, etc. shall be considered.

A more subtle consideration in designing the sampling program is the potential for dispersion of dissolved or sediment-associated contaminants away from the source. The dispersion could lead to a more homogeneous distribution of contamination at low or possibly non-detectable concentrations. Such dispersion does not, however, always readily occur. For example, obtaining a representative sample of contamination from a main stream immediately below an outfall or a tributary is difficult because the inflow frequently follows a stream bank with little lateral mixing for some distance. Sampling alternatives to overcome this situation include: (1) moving the sampling location far enough downstream to allow for adequate mixing, or (2) collecting integrated samples in a cross section. Also, non-homogeneous distribution is a particular problem with regard to sediment-associated contaminants, which may accumulate in low-energy environments (coves, river bends, deep spots, or even behind boulders) near or distant from the source while higher-energy areas (main stream channels) near the source may show no contaminant accumulation.

The distribution of particulates within a sample itself is an important consideration. Many organic compounds are only slightly water soluble and tend to adsorb onto particulate matter. Nitrogen, phosphorus, and heavy metals may also be transported by particulates. Samples must be collected with a representative amount of suspended material; transfer from the sampling device shall include transferring a proportionate amount of the suspended material.

C.1.2 Location of Sampling Stations

Accessibility is the primary factor affecting sampling costs. The desirability and utility of a sample for analysis and consideration of site conditions must be balanced against the costs of collection as controlled by accessibility. Bridges or piers are the first choice for locating a sampling station on a stream because bridges provide ready access and also permit the sampling technician to sample any point across the stream. A boat or pontoon (with an associated increase in cost) may be needed to sample locations on lakes, reservoirs, or larger rivers. Frequently, however, a boat will take longer to cross a water body and will hinder manipulation of the sampling equipment. Wading for samples is not recommended unless it is known that contaminant levels are low so that skin contact will not produce adverse health effects. This provides a built in margin of safety in the event that wading boots or other protective equipment should fail to function properly. If it is necessary to wade into the water body to obtain a sample, the sampler shall be careful to minimize disturbance of bottom sediments and must enter the water body downstream of the sampling location. If necessary, the sampling technician shall wait for the sediments to settle before taking a sample.

Under ideal and uniform contaminant dispersion conditions in a flowing stream, the same concentrations of each contaminant would occur at all points along the cross section. This situation is most likely downstream of areas of high turbulence. Careful site selection is needed to ensure, as nearly as possible, that samples are taken where uniform flow or deposition and good mixing conditions exist.

The availability of stream flow and sediment discharge records can be an important consideration in choosing sampling sites in streams. Stream flow data in association with contaminant concentration data are essential for estimating the total contaminant loads carried by the stream. If a gaging station is not conveniently located on a selected stream, the project hydrogeologist shall explore the possibility of obtaining stream flow data by direct or indirect methods. Remember these locations are also where you may encounter natural hazards as these are areas where they hunt. Always exercise extreme caution.

C.1.3 Frequency of Sampling

The sampling frequency and objectives of the sampling event will be defined by the project planning documents. For single-event site or area characterization sampling, both bottom material and overlying water samples shall be collected at the specified sampling stations. If valid data are available on the distribution of a contaminant between the solid and aqueous phases, it may be appropriate to sample only one phase, although this is not often recommended. If samples are collected primarily for monitoring purposes (i.e., consisting of repetitive, continuing measurements to define variations and trends at a given location), water samples should be collected at a pre-established and constant interval as specified in the project plans (often monthly or quarterly and during droughts and floods). Samples of bottom material should generally be collected from fresh deposits at least yearly, and preferably seasonally, during both spring and fall.

The variability in available water quality data shall be evaluated before determining the number and collection frequency of samples required to maintain an effective monitoring program.

C.2 Surface Water Sample Collection

C.2.1 Streams, Rivers, Outfalls and Drainage Features

Methods for sampling streams, rivers, outfalls, and drainage features (ditches, culverts) at a single point vary from the simplest of hand-sampling procedures to the more sophisticated multi-point sampling techniques known as the equal-width-increment (EWI) method or the equal-discharge-increment (EDI) methods (see below).

Samples from different depths or cross-sectional locations in the watercourse taken during the same sampling episode shall be composited. However, samples collected along the length of the watercourse or at different times may reflect differing inputs or dilutions and therefore shall not be composited. Generally, the number and type of samples to be taken depend on the river's width, depth, and discharge and on the suspended sediment the stream or river transports. The greater the number of individual points that are sampled, the more likely that the composite sample will truly represent the overall characteristics of the water.

In small streams less than about 20 feet wide, a sampling site can generally be found where the water is well mixed. In such cases, a single grab sample taken at mid-depth in the center of the channel is adequate to represent the entire cross section.

For larger streams, at least one vertical composite shall be taken with one sample each from just below the surface, at mid-depth, and just above the bottom. The measurement of dissolved oxygen (DO), pH, temperature, conductivity, etc., shall be made on each aliquot of the vertical composite and on the composite itself. For rivers, several vertical composites shall be collected, as directed in the project planning documents.

C.2.2 Lakes, Ponds and Reservoirs

Lakes, ponds, and reservoirs have a much greater tendency to stratify than rivers and streams. The relative lack of mixing requires that more samples be obtained. The number of water sampling sites on a lake, pond, or impoundment will vary with the size and shape of the basin. In ponds and small lakes, a single vertical composite at the deepest point may be sufficient. Similarly, measurement of DO, pH, temperature, etc. is to be conducted on each aliquot of the vertical composite and on the composite itself. In naturally formed ponds, the deepest point may have to be determined empirically; in impoundments, the deepest point is usually near the dam.

In lakes and larger reservoirs, several vertical composites shall be composited to form a single sample if a sample representative of the water column is required. These vertical composites are often collected along a transect or grid. In some cases, it may be of interest to form separate composites of epilimnetic and hypolimnetic zones. In a stratified lake, the epilimnion is the thermocline that is exposed to the atmosphere. The hypolimnion is the lower, "confined" layer that is only mixed with the epilimnion and vented to the atmosphere during seasonal "overturn" (when density stratification disappears). These two zones may thus have very different concentrations of contaminants if input is only to one zone, if the contaminants are volatile (and therefore vented from the epilimnion but not the hypolimnion), or if the epilimnion only is involved in short-term flushing (i.e., inflow from or outflow to shallow streams). Normally, however, a composite consists of several vertical composites with samples collected at various depths.

In lakes with irregular shape and with bays and coves that are protected from the wind, separate composite samples may be needed to adequately represent water quality because it is likely that only poor mixing will occur. Similarly, additional samples are recommended where discharges, tributaries, land use characteristics, and other such factors are suspected of influencing water quality.

Many lake measurements are now made in situ using sensors and automatic readout or recording devices. Single and multi-parameter instruments are available for measuring temperature, depth, pH, oxidation-reduction potential (ORP), specific conductance, DO, some cations and anions, and light penetration.

C.2.3 Estuaries

Estuarine areas are, by definition, zones where inland freshwaters (both surface and ground) mix with oceanic saline waters. Knowledge of the estuary type may be necessary to determine sampling locations. Estuaries are generally categorized into one of the following three types dependent on freshwater inflow and mixing properties:

- Mixed Estuary - characterized by the absence of a vertical halocline (gradual or no marked increase in salinity in the water column) and a gradual increase in salinity seaward. Typically, this type of estuary is shallow and is found in major freshwater sheet flow areas. Because this type of estuary is well mixed, sampling locations are not critical.

- Salt Wedge Estuary - characterized by a sharp vertical increase in salinity and stratified freshwater flow along the surface. In these estuaries, the vertical mixing forces cannot override the density differential between fresh and saline waters. In effect, a salt wedge tapering inland moves horizontally back and forth with the tidal phase. If contamination is being introduced into the estuary from upstream, water sampling from the salt wedge may miss it entirely.

- Oceanic Estuary - characterized by salinities approaching full-strength oceanic waters. Seasonally, freshwater inflow is small, with the preponderance of the fresh-saline water mixing occurring near or at the shore line.

Sampling in estuarine areas is normally based on the tidal phase, with samples collected on successive slack tides (i.e., when the tide turns). Estuarine sampling programs shall include vertical salinity measurements at 1- to 5-foot increments, coupled with vertical DO and temperature profiles.

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1.0 PURPOSE

This procedure provides general guidance and information pertaining to proper monitoring well design, installation, and development.

2.0 SCOPE

This procedure is applicable to the construction of monitoring wells. The methods described herein may be modified by project-specific requirements for monitoring well construction. In addition, many regulatory agencies have specific regulations pertaining to monitoring well construction and permitting. These requirements must be determined during the project planning phases of the investigation, and any required permits must be obtained before field work begins. Innovative monitoring well installation techniques, which typically are not used, will be discussed only generally in this procedure.

3.0 GLOSSARY

Monitoring Well - A well which is screened, cased, and sealed which is capable of providing a groundwater level and groundwater sample representative of the zone being monitored. Some monitoring wells may be constructed as open boreholes.

Piezometer - A pipe or tube inserted into the water bearing zone, typically open to water flow at the bottom and to the atmosphere at the top, and used to measure water level elevations. Piezometers may range in size from 1/2-inch-diameter plastic tubes to well points or monitoring wells.

Potentiometric Surface - The surface representative of the level to which water will rise in a well cased to the screened aquifer.

Well Point (Drive Point) - A screened or perforated tube (Typically 1-1/4 or 2 inches in diameter) with a solid, conical, hardened point at one end, which is attached to a riser pipe and driven into the ground with a sledge hammer, drop weight, or mechanical vibrator. Well points may be used for groundwater injection and recovery, as piezometers (i.e., to measure water levels) or to provide groundwater samples for water quality data.

4.0 RESPONSIBILITIES

Driller - The driller provides adequate and operable equipment, sufficient quantities of materials, and an experienced and efficient labor force capable of performing all phases of proper monitoring well installation and construction. The driller may also be responsible for obtaining, in advance, any required permits for monitoring well installation and construction.

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

5.0 PROCEDURES

5.1 Equipment/Items Needed

Below is a list of items that may be needed when installing a monitoring well or piezometer:

- Health and safety equipment (hard hats, safety glasses, etc.) as required by the Site Safety Officer.
- Well drilling and installation equipment with associated materials (typically supplied by the driller).
- Hydrogeologic equipment (weighted engineer's tape, water level indicator, retractable engineers rule, electronic calculator, clipboard, mirror and flashlight - for observing downhole activities, paint and ink marker for marking monitoring wells, sample jars, well installation forms, and a field notebook).
- Drive point installation tools (sledge hammer, drop hammer, or mechanical vibrator; tripod, pipe wrenches, drive points, riser pipe, and end caps).

5.2 Well Design

The objectives and intended use for each monitoring well must be clearly defined before the monitoring system is designed. Within the monitoring system, different monitoring wells may serve different purposes and, therefore, require different types of construction. During all phases of the well design, attention must be given to clearly documenting the basis for design decisions, the details of well construction, and the materials used. The objectives for installing the monitoring wells may include:

- Determining groundwater flow directions and velocities.
- Sampling or monitoring for trace contaminants.
- Determining aquifer characteristics (e.g., hydraulic conductivity).

Siting of monitoring wells shall be performed after a preliminary estimation of the groundwater flow direction. In most cases, groundwater flow directions and potential well locations can be determined by an experienced hydrogeologist through the review of geologic data and the site terrain. In addition, data from production wells or other monitoring wells in the area may be used to determine the groundwater flow direction. If these methods cannot be used, piezometers, which are relatively inexpensive to install, may have to be installed in a preliminary investigative phase to determine groundwater flow direction.

5.2.1 Well Depth, Diameter, and Monitored Interval

The well depth, diameter, and monitored interval must be tailored to the specific monitoring needs of each investigation. Specification of these items generally depends on the purpose of the monitoring system and the characteristics of the hydrogeologic system being monitored. Wells of different depth, diameter, and monitored interval can be employed in the same groundwater monitoring system. For instance, varying the monitored interval in several wells, at the same location (cluster wells) can help to determine the vertical gradient and the depths at which contaminants are present. Conversely, a fully penetrating well is usually not used to quantify or vertically locate a contaminant plume, since groundwater samples collected in wells that are screened over the full thickness of the water-bearing zone will be representative of average conditions across the entire monitored interval. However, fully penetrating wells can be used to establish the existence of contamination in the water-bearing zone. The well diameter desired depends upon the hydraulic characteristics of the water-bearing zone, sampling requirements, drilling method and cost.

The decision concerning the monitored interval and well depth is based on the following (and possibly other) information:

- The vertical location of the contaminant source in relation to the water-bearing zone.
- The depth, thickness and uniformity of the water-bearing zone.
- The anticipated depth, thickness, and characteristics (e.g., density relative to water) of the contaminant plume.
- Fluctuation in groundwater levels (due to pumping, tidal influences, or natural recharge/discharge events).
- The presence and location of contaminants encountered during drilling.
- Whether the purpose of the installation is for determining existence or non-existence of contamination or if a particular stratigraphic zone is being investigated.
- The analysis of borehole geophysical logs.

In most situations where groundwater flow lines are horizontal, depending on the purpose of the well and the site conditions, monitored intervals are 20 feet or less. Shorter screen lengths (5 feet or less) are usually required where flow lines are not horizontal, (i.e., if the wells are to be used for accurate measurement of the potentiometric head at a specific point).

Many factors influence the diameter of a monitoring well. The diameter of the monitoring well depends on the application. In determining well diameter, the following needs must be considered:

- Adequate water volume for sampling.
- Drilling methodology.
- Type of sampling device to be used.
- Costs.

Standard monitoring well diameters are 2, 4, 6, or 8 inches. Drive points are typically 1-1/4 or 2 inches in diameter. For monitoring programs which require screened monitoring wells, either a 2-inch or 4-inch-diameter well is preferred. Typically, well diameters greater than 4 inches are used in monitoring programs in which open-hole bedrock monitoring wells are used. With smaller diameter wells, the volume of stagnant water in the well is minimized, and well construction costs are reduced; however, the sampling devices that can be used are limited.

In specifying well diameter, sampling requirements must be considered (up to a total of 4 gallons of water may be required for a single sample to account for full organic and inorganic analyses, and split samples), particularly if the monitored formation is known to be a low-yielding formation. The unit volume of water contained within a monitoring well is dependent on the well diameter as follows:

Casing Inside Diameter (Inch)	Standing Water Length to Obtain 1 Gallon Water (Feet)
2	6.13
4	1.53
6	0.68

If a well recharges quickly after purging, then well diameter may not be an important factor regarding sample volume requirements.

Pumping tests for determining aquifer characteristics may require larger diameter wells (for installation of high capacity pumps); however, in small-diameter wells in-situ permeability tests can be performed during drilling or after well installation is completed.

5.2.2 Riser Pipe and Screen Materials

Well materials are specified by diameter, type of material, and thickness of pipe. Well screens require an additional specification of slot size. Thickness of pipe is referred to as "Schedule" for polyvinyl chloride (PVC) casing and is usually Schedule 40 (thinner wall) or 80 (thicker wall). Steel pipe thickness is often referred to as "Strength". Standard Strength is usually adequate for monitoring well purposes. With larger diameter pipe, the wall thickness must be greater to maintain adequate strength. The required thickness is also dependent on the method of installation; risers for drive points require greater strength than wells installed inside drilled borings.

The selection of well screen and riser materials depends on the method of drilling, the type of subsurface materials the well penetrates, the type of contamination expected, and natural water quality and depth. Cost and the level of accuracy required are also important. The materials generally available are Teflon, stainless steel, PVC galvanized steel, and carbon steel. Each has advantages and limitations (see Attachment A of this guideline for an extensive presentation on this topic). The two most commonly used materials are PVC and stainless steel. Properties of these two materials are compared in Attachment B. Stainless steel is a good choice where trace metals or organic sampling is required; however, costs are high. Teflon materials are extremely expensive, but are relatively inert and provide the least opportunity for water contamination due to well materials. PVC has many advantages, including low cost, excellent availability, light weight, ease of manipulation, and widespread acceptance. The crushing strength of PVC may limit the depth of installation, but the use of Schedule 80 materials may overcome some of the problems associated with depth. However, the smaller inside diameter of Schedule 80 pipe may be an important factor when considering the size of bailers or pumps required for sampling or testing. Due to this problem, the minimum well pipe size recommended for Schedule 80 wells is 4-inch I.D.

Screens and risers may have to be decontaminated before use because oil-based preservatives and oil used during thread cutting and screen manufacturing may contaminate samples. Metal pipe may corrode and release metal ions or chemically react with organic constituents, but this is considered a minor issue. Galvanized steel is not recommended where samples may be collected for metals analyses, as zinc and cadmium levels in groundwater samples may become elevated from leaching of the zinc coating.

Threaded, flush-joint casing is most often preferred for monitoring well applications. PVC, Teflon, and steel can all be obtained with threaded joints. Welded-joint steel casing is also acceptable. Glued PVC may release organic contaminants into the well, and therefore, should not be used if the well is to be sampled for organic constituents.

When the water-bearing zone is in consolidated bedrock, such as limestone or fractured granite, a well screen is often not necessary (the well is simply an open hole in bedrock). Unconsolidated materials, such as sands, clay, and silts require a screen. A screen slot size of 0.010 or 0.020 inch is generally used when a screen is necessary, and the annular borehole space around the screened interval is artificially packed with an appropriately sized sand, selected based on formation grain size. The slot size controls the quantity of water entering the well and prevents entry of natural materials or sand pack. The screen shall pass no more than 10 percent of the pack material, or in-situ aquifer material. The site geologist shall specify the combination of screen slot size and sand pack which will be compatible with the water-bearing zone, to maximize groundwater inflow and minimize head losses and movement of fines into the wells. For example, as a standard procedure, a Morie No. 1 or No. 10 to No. 20 U.S. Standard Sieve size filter pack is typically appropriate for a 0.020-inch slot screen; however, a No. 20 to No. 40 U.S. Standard Sieve size filter pack is typically appropriate for a 0.010-inch slot screen.

5.2.3 Annular Materials

Materials placed in the annular space between the borehole and riser pipe and screen include a sand pack when necessary, a bentonite seal, and cement-bentonite grout. The sand pack is usually a medium- to coarse-grained poorly graded, silica sand and should relate to the grain size of the aquifer sediments. The quantity of sand placed in the annular space is dependent upon the length of the screened interval, but should always extend at least 1 foot above the top of the screen. At least 1 to 3 feet of bentonite pellets or equivalent shall be placed above the sand pack. Cement-bentonite grout (or equivalent) is then placed to extent from the top of the bentonite pellets to the ground surface.

On occasion, and with the concurrence of the involved regulatory agencies, monitoring wells may be packed naturally (i.e., no artificial sand pack installed). In this case, the natural formation material is allowed to collapse around the well screen after the well is installed. This method has been used where the formation material itself is a relatively uniform grain size, or when artificial sand packing is not possible due to borehole collapse.

Bentonite expands by absorbing water and provides a seal between the screened interval and the overlying portion of the annular space and formation. Cement-bentonite grout is placed on top of the bentonite pellets, extending to the surface. The grout effectively seals the remaining borehole annulus and eliminates the possibility for surface infiltration reaching the screened interval. Grouting also replaces material removed during drilling and prevents hole collapse and subsidence around the well. A tremie pipe should be used to introduce grout from the bottom upward, to prevent bridging, and to provide a better seal. In shallow boreholes that don't collapse, it may be more practical to pour the grout from the surface without a tremie pipe.

Grout is a general term which has several different connotations. For all practical purposes within the monitoring well installation industry, grout refers to the solidified material which is installed and occupies the annular space above the bentonite pellet seal. Grout, most of the time, is made up of one or two assemblages of material, (e.g., cement and/or bentonite). A cement-bentonite grout, which is the most common type of grout used in monitoring well completions, normally is a mixture of cement, bentonite, and water at a ratio of one 90-pound bag of Portland Type I cement, plus 3 to 5 pounds of granular or flake-type bentonite, and 6-7 gallons of water. A neat cement consists of one ninety-pound bag of Portland Type I cement and 6-7 gallons of water. A bentonite slurry (bentonite and water mixed to a thick but pumpable mixture) is sometimes used instead of grout for deep well installations where placement of bentonite pellets is difficult. Bentonite chips are also occasionally used for annular backfill in place of grout.

In certain cases, the borehole may be drilled to a depth greater than the anticipated well installation depth. For these cases, the well shall be backfilled to the desired depth with bentonite pellets/chips or sand. A short (1- to 2-foot) section of capped riser pipe sump is sometimes installed immediately below the screen, as a silt reservoir, when significant post-development silting is anticipated. This will ensure that the entire screen surface remains unobstructed.

5.2.4 Protective Casing

When the well is completed and grouted to the surface, a protective steel casing is typically placed over the top of the well. This casing generally has a hinged cap and can be locked to prevent vandalism. The protective casing has a larger diameter than the well and is set into the wet cement grout over the well upon completion. In addition, one hole is drilled just above the cement collar through the protective casing which acts as a weep hole for the flow of water which may enter the annulus during well development, purging, or sampling.

A protective casing which is level with the ground surface (flush-mounted) is used in roadway or parking lot applications where the top of a monitoring well must be below the pavement. The top of the riser pipe is placed 4 to 5 inches below the pavement, and a locking protective casing is cemented in place to 3 inches below the pavement. A large diameter, manhole-type protective collar is set into the wet cement around the well with the top set level with or slightly above the pavement. An appropriately-sized id is placed over the protective sleeve. The cement should be slightly mounded to direct pooled water away from the well head.

5.3 Monitoring Well Installation

Pertinent data regarding monitoring well installation shall be recorded on log sheets as depicted and discussed in SOP SA-6.3. Attachments to this referenced SOP illustrate terms and physical construction of various types of monitoring wells.

5.3.1 Monitoring Wells in Unconsolidated Sediments

After the borehole is drilled to the desired depth, well installation can begin. The procedure for well installation will partially be dictated by the stability of the formation in which the well is being placed. If the borehole collapses immediately after the drilling tools are withdrawn, then a temporary casing must be installed and well installation will proceed through the center of the temporary casing, and continue as the temporary casing is withdrawn from the borehole. In the case of hollow-stem auger drilling, the augers will act to stabilize the borehole during well installation.

Before the screen and riser pipe are lowered into the borehole, all pipe and screen sections should be measured with an engineer's rule to ensure proper placement. When measuring sections, the threads on one end of the pipe or screen must be excluded while measuring, since the pipe and screen sections are screwed flush together.

After the screen and riser pipe are lowered through the temporary casing, the sand pack can be installed. A weighted tape measure must be used during the installation procedure to carefully monitor installation progress. The sand is slowly poured into the annulus between the riser pipe and temporary casing, as the casing is withdrawn. Sand should always be kept within the temporary casing during withdrawal in order to ensure an adequate sand pack. However, if too much sand is within the temporary casing (greater than 1 foot above the bottom of the casing) bridging between the temporary casing and riser pipe may occur. Centralizers may be used at the geologist's discretion, one above and one below the screen, to assure enough annular space for sand pack placement.

After the sand pack is installed to the desired depth (at least 1 foot above the top of the screen), then the bentonite pellet seal (or equivalent), can be installed in the same manner as the sand pack. At least 1 to 3 feet of bentonite pellets should be installed above the sand pack. Pellets should be added slowly and their fall monitored closely to ensure that bridging does not occur.

The cement-bentonite grout is then mixed and tremied into the annulus as the temporary casing or augers are withdrawn. Finally, the protective casing can be installed as detailed in Section 5.2.4.

5.3.2 Confining Layer Monitoring Wells

When drilling and installing a well in a confined aquifer, proper well installation techniques must be applied to avoid cross contamination between aquifers. Under most conditions, this can be accomplished by installing double-cased wells. This is accomplished by drilling a large-diameter boring through the upper aquifer, 1 to 5 feet into the underlying confining layer, and setting and pressure grouting or tremie grouting a large-diameter casing into the confining layer. The grout material must fill the space between the native material and the outer casing. A smaller diameter boring is then continued through the confining layer for

installation of the monitoring well as detailed for overburden monitoring wells. Sufficient time (determined by the field geologist), must be allowed for setting of the grout prior to drilling through the confined layer.

5.3.3 Bedrock Monitoring Wells

When installing bedrock monitoring wells, a large diameter boring is drilled through the overburden and approximately 5 –10 feet into bedrock. A casing (typically steel) is installed and either pressure grouted or tremie grouted in place. After the grout has cured, a smaller diameter boring is continued into bedrock to the desired depth. If the boring does not collapse, the well can be left open, and a screen is not necessary. If the boring collapses, then a screen is required and can be installed as detailed for overburden monitoring wells. If a screen is to be used, then the casing which is installed through the overburden and into the bedrock does not require grouting and can be removed when the final well installation is completed.

5.3.4 Drive Points

Drive points can be installed with either a sledge hammer, drop hammer, or a mechanical vibrator. The screen section is threaded and tightened onto the riser pipe with pipe wrenches. The drive point is simply pounded into the subsurface to the desired depth. If a heavy drop hammer is used, then a tripod and pulley setup is required to lift the hammer. Drive points typically cannot be manually driven to depths exceeding 10 feet.

Direct push sampling/monitoring point installation methods, using a direct push rig or drilling rig, are described in SOP SA-2.5.

5.3.5 Innovative Monitoring Well Installation Techniques

Certain innovative sampling devices have proven advantageous. These devices are essentially screened samplers installed in a borehole with only small-diameter tubes extending to the surface. This reduces drilling costs, decreases the volume of stagnant water, and provides a sampling system that minimizes cross-contamination from sampling equipment. Four manufacturers of these samplers include Timco Manufacturing Company, Inc., of Prairie du Sac, Wisconsin, BARCAD Systems, Inc., of Concord, Massachusetts, Westbay Instruments Ltd. of Vancouver, British Columbia, Canada and the University of Waterloo at Waterloo, Ontario, Canada.. Each manufacturer offers various construction materials.

5.4 Well Development Methods

The purpose of well development is to stabilize and increase the permeability of the gravel pack around the well screen, and to restore the permeability of the formation which may have been reduced by drilling operations. Wells are typically developed until all fine material and drilling water is removed from the well. Sequential measurements of pH, conductivity, turbidity, and temperature taken during development may yield information (stabilized values) regarding whether sufficient development has been performed. The selection of the well development method shall be made by the field geologist and is based on the drilling methods, well construction and installation details, and the characteristics of the formation that the well is screened in. The primary methods of well development are summarized below. A more detailed discussion may be found in Driscoll (1986).

5.4.1 Overpumping and Backwashing

Wells may be developed by alternatively drawing the water level down at a high rate (by pumping or bailing) and then reversing the flow direction (backwashing) so that water is passing from the well into the formation. This back and forth movement of water through the well screen and gravel pack serves to

~~remove fines from the formation immediately adjacent to the well, while preventing bridging (wedging) of sand grains. Backwashing can be accomplished by several methods, including pouring water into the well and then bailing, starting and stopping a pump intermittently to change water levels, or forcing water into the well under pressure through a water-tight fitting ("rawhiding"). Care should be taken when backwashing not to apply too much pressure, which could damage or destroy the well screen.~~

5.4.2 Surging with a Surge Plunger

A surge plunger (also called a surge block) is approximately the same diameter as the well casing and is aggressively moved up and down within the well to agitate the water, causing it to move in and out of the screens. This movement of water pulls fine materials into the well, where they may be removed by any of several methods, and prevents bridging of sand particles in the gravel pack. There are two basic types of surge plungers; solid and valved surge plungers. In formations with low yields, a valved surge plunger may be preferred, as solid plungers tend to force water out of the well at a greater rate than it will flow back in. Valved plungers are designed to produce a greater inflow than outflow of water during surging.

~~5.4.3~~ Compressed Air

~~Compressed air can be used to develop a well by either of two methods: backwashing or surging. Backwashing is done by forcing water out through the screens, using increasing air pressure inside a sealed well, then releasing the pressurized air to allow the water to flow back into the well. Care should be taken when using this method so that the water level does not drop below the top of the screen, thus introducing air into the formation and reducing well yield. Surging, or the "open well" method, consists of alternately releasing large volumes of air suddenly into an open well below the water level to produce a strong surge by virtue of the resistance of water head, friction, and inertia. Pumping of the well is subsequently done using the air lift method.~~

5.4.4 High Velocity Jetting

In the high velocity jetting method, water is forced at high velocities from a plunger-type device and through the well screen to loosen fine particles from the sand pack and surrounding formation. The jetting tool is slowly rotated and raised and lowered along the length of the well screen to develop the entire screened area. Jetting using a hose lowered into the well may also be effective. The fines washed into the screen during this process can then be bailed or pumped from the well.

6.0 RECORDS

A critical part of monitoring well installation is recording of all significant details and events in the site logbook or field notebook. The geologist must record the exact depths of significant hydrogeological features, screen placement, gravel pack placement, and bentonite placement.

A Monitoring Well Sheet (see Attachments to SOP SA-6.3) shall be completed, ensuring the uniform recording of data for each installation and rapid identification of missing information. Well depth, length, materials of construction, length and openings of screen, length and type of riser, and depth and type of all backfill materials shall be recorded. Additional information shall include location, installation date, problems encountered, water levels before and after well installation, cross-reference to the geologic boring log, and methods used during the installation and development process. Documentation is very important to prevent problems involving questionable sample validity. Somewhat different information will need to be recorded, depending on whether the well is completed in overburden (single- or double-cased), as a cased well in bedrock, or as an open hole in bedrock.

The quantities of sand, bentonite, and grout placed in the well are also important. The geologist shall calculate the annular space volume and have an idea of the quantity of material needed to fill the annular

space. Volumes of backfill significantly higher than the calculated volume may indicate a problem such as a large cavity, while a smaller backfill volume may indicate a cave-in or bridging of the backfill materials. Any problems with rig operation or down-time shall be recorded and may affect the driller's final fee.

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ATTACHMENT A

RELATIVE COMPATIBILITY OF RIGID WELL CASING MATERIAL (PERCENT)

Potentially-Deteriorating Substance	Type of Casing Material						
	PVC 1	Galvanized Steel	Carbon Steel	Lo-carbon Steel	Stainless Steel 304	Stainless Steel 316	Teflon*
Buffered Weak Acid	100	56	51	59	97	100	100
Weak Acid	98	59	43	47	96	100	100
Mineral Acid/ High Solids Content	100	48	57	60	80	82	100
Aqueous/Organic Mixtures	64	69	73	73	98	100	100
Percent Overall Rating	91	58	56	59	93	96	100

Preliminary Ranking of Rigid Materials:

1 Teflon*
2 Stainless Steel 316
3. Stainless Steel 304
4 PVC 1
5 Lo-Carbon Steel
6 Galvanized Steel
7 Carbon Steel

* Trademark of DuPont

RELATIVE COMPATIBILITY OF SEMI-RIGID OR ELASTOMERIC MATERIALS (PERCENT)

Potentially-Deteriorating Substance	Type of Casing Material								
	PVC Flexible	PP	PE Conv.	PE Linear	PMM	Viton**	Silicone	Neoprene	Teflon**
Buffered Weak Acid	97	97	100	97	90	92	87	85	100
Weak Acid	92	90	94	96	78	78	75	75	100
Mineral Acid/ High Solids Content	100	100	100	100	95	100	78	82	100
Aqueous/Organic Mixtures	62	71	40	60	49	78	49	44	100
Percent Overall Rating	88	90	84	88	78	87	72	72	100

Preliminary Ranking of Semi-Rigid or Elastomeric Materials:

1 Teflon*
2 Polypropylene (PP)
3. PVC Flexible/PE Linear
4 Viton*
5 PE Conventional
6 Plexiglas/Lucite (PMM)
7 Silicone/Neoprene

* Trademark of DuPont

Source: Barcelona et al., 1983

ATTACHMENT B

COMPARISON OF STAINLESS STEEL AND PVC FOR MONITORING WELL CONSTRUCTION

Characteristic	Stainless Steel	PVC
Strength	Use in deep wells to prevent compression and closing of screen/riser.	Use when shear and compressive strength are not critical.
Weight	Relatively heavier.	Light-weight; floats in water.
Cost	Relatively expensive.	Relatively inexpensive.
Corrosivity	Deteriorates more rapidly in corrosive water.	Non-corrosive -- may deteriorate in presence of ketones, aromatics, alkyl sulfides, or some chlorinated hydrocarbons.
Ease of Use	Difficult to adjust size or length in the field.	Easy to handle and work with in the field.
Preparation for Use	Should be steam cleaned if organics will be subsequently sampled.	Never use glue fittings -- pipes should be threaded or pressure fitted. Should be steam cleaned when used for monitoring wells.
Interaction with Contaminants*	May sorb organic or inorganic substances when oxidized.	May sorb or release organic substances.

* See also Attachment A.

S14 GH-1.3 SOIL AND ROCK DRILLING METHODS

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1.0 PURPOSE

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

2.0 SCOPE

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

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

3.0 GLOSSARY

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

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

4.0 RESPONSIBILITIES

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

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

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

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

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

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

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

5.0 PROCEDURES

5.1 General

The purpose of drilling boreholes is:

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

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

5.2 Drilling Methods

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

5.2.1 Continuous-Flight Hollow-Stem Auger Drilling

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

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

Disadvantages and limitations of this method of drilling include:

- Augering can only be done in unconsolidated materials.
- The inside diameter of hollow stem augers used for well installation should be at least 4 inches greater than the well casing. Use of such large-diameter hollow-stem augers is more expensive than the use of small-diameter augers in boreholes not used for well installation. Furthermore, the density of unconsolidated materials and depths become more of a limiting factor. More friction is produced with the larger diameter auger and subsequently greater torque is needed to advance the boring.
- The maximum effective depth for drilling is 150 feet or less, depending on site conditions and the size of augers used.
- In augering through clean sand formations below the water table, the sand will tend to flow into the hollow stem when the plug is removed for soil sampling or well installation. If the condition of "running" or "flowing" sands is persistent at a site, an alternative method of drilling is recommended, in particular for wells or boreholes deeper than 25 feet.

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

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

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

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

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

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

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

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

5.2.2 Continuous-Flight Solid-Stem Auger Drilling

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

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

5.2.3 Rotary Drilling

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

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

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

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

Disadvantages to using this method include:

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

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

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

Advantages to the fluid-rotary drilling method include:

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

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

Disadvantages to this method include:

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

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

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

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

5.2.4 Rotosonic Drilling

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

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

Advantages of this method include:

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

Disadvantages include:

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

5.2.5 Reverse Circulation Rotary Drilling

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

Advantages of the latter method include:

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

Disadvantages include:

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

5.2.6 Drill-through Casing Driver

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

The driven casing method is used in unconsolidated formations only. When the boring is to be used for later well installation, the driven casing used should be at least 4 inches larger in diameter than the well casing to be installed. Advantages to this method of drilling include:

- Split-barrel (split-spoon) sampling can be conducted while drilling.
- Well installation is easily accomplished.
- Drill rigs used are relatively small and mobile.
- The use of casing minimizes flow into the hole from upper water-bearing layers; therefore, multiple aquifers can be penetrated and sampled for rough field determinations of some water quality parameters.

Some of the disadvantages include:

- This method can only be used in unconsolidated formations.
- The method is slower than other methods (average drilling progress is 30 to 50 feet per day).
- Maximum depth of the borehole varies with the size of the drill rig and casing diameter used, and the nature of the formations drilled.
- The cost per hour or per foot of drilling may be substantially higher than other drilling methods.
- It is difficult and time consuming to pull back the casing if it has been driven very deep (deeper than 50 feet in many formations).

5.2.7 Cable Tool Drilling

A cable tool rig uses a heavy, solid-steel, chisel-type drill bit ("tool") suspended on a steel cable, which when raised and dropped, chisels or pounds a hole through the soils and rock. Drilling progress may be

expedited by the use of "slip-jars" which serve as a cable-activated down hole percussion device to hammer the bit ahead.

When drilling through the unsaturated zone, some water must be added to the hole. The cuttings are suspended in the water and then bailed out periodically. Below the water table, after sufficient ground water enters the borehole to replace the water removed by bailing, no further water needs to be added. When soft caving formations are encountered, it is usually necessary to drive casing as the hole is advanced to prevent collapse of the hole. Often the drilling can be only a few feet below the bottom of the casing. Because the drill bit is lowered through the casing, the hole created by the bit is smaller than the casing. Therefore, the casing (with a sharp, hardened casing shoe on the bottom) must be driven into the hole (see Section 5.2.5 of this guideline).

Advantages of the cable-tool method include the following:

- Information regarding water-bearing zones is readily available during the drilling. Even relative permeabilities and rough water quality data from different zones penetrated can be obtained by skilled operators.
- The cable-tool rig can operate satisfactorily in all formations, but is best suited for caving, boulder, cobble or coarse gravel type formations (e.g., glacial till) or formations with large cavities above the water table (such as limestones).
- When casing is used, the casing seals formation water out of the hole, preventing down hole contamination and allowing sampling of deeper aquifers for field-measurable water quality parameters.
- Split-barrel (split-spoon) or thin-wall (Shelby) tube samples can be collected through the casing.

Disadvantages include:

- Drilling is slow compared with rotary rigs.
- The necessity of driving the casing in unconsolidated formations requires that the casing be pulled back if exposure of selected water-bearing zones is desired. This process complicates the well completion process and often increases costs. There is also a chance that the casing may become stuck in the hole.
- The relatively large diameters required (minimum of 4-inch casing) plus the cost of steel casing result in higher costs compared to rotary drilling methods where casing is not required (e.g., such use of a hollow-stem auger).
- Cable-tool rigs have largely been replaced by rotary rigs. In some parts of the U.S., availability may be difficult.

5.2.8 Jet Drilling (Washing)

Jet drilling, which should be used only for piezometer or vadose zone sampler installation, consists of pumping water or drilling mud down through a small diameter (1/2- to 2-inch) standard pipe (steel or PVC). The pipe may be fitted with a chisel bit or a special jetting screen. Formation materials dislodged by the bit and jetting action of the water are brought to the surface through the annulus around the pipe. As the pipe is jetted deeper, additional lengths of pipe may be added at the surface.

Jet percussion is a variation of the jetting method, in which the casing is driven with a drive weight. Normally, this method is used to place 2-inch-diameter casing in shallow, unconsolidated sand formations, but this method has also been used to install 3- to 4-inch-diameter casings to a depth of 200 feet.

Jetting is acceptable in very soft formations, usually for shallow sampling, and when introduction of drilling water to the formation is acceptable. Such conditions would occur during rough stratigraphic investigation or installation of piezometers for water level measurement. Advantages of this method include:

- Jetting is fast and inexpensive.
- Because of the small amount of equipment required, jetting can be accomplished in locations where access by a normal drilling rig would be very difficult. For example, it would be possible to jet down a well point in the center of a lagoon at a fraction of the cost of using a drill rig.
- Jetting numerous well points just into a shallow water table is an inexpensive method for determining the water table contours, hence flow direction.

Disadvantages include the following:

- A large amount of foreign water or drilling mud is introduced above and into the formation to be sampled.
- Jetting is usually done in very soft formations which are subject to caving. Because of this caving, it is often not possible to place a grout seal above the screen to assure that water in the well is only from the screened interval.
- The diameter of the casing is usually limited to 2 inches.
- Jetting is only possible in very soft formations that do not contain boulders or coarse gravel, and the depth limitation is shallow (about 30 feet without jet percussion equipment).
- Large quantities of water are often needed.

5.2.9 Drilling with a Hand Auger

This method is applicable wherever the formation, total depth of sampling, and the site and groundwater conditions are such as to allow hand auger drilling. Hand augering can also be considered at locations where drill rig access is not possible. All hand auger borings will be performed according to ASTM D1452-80.

Samples should be taken continuously unless otherwise specified by the project plan documents. Any required sampling is performed by rotation, pressing, or driving in accordance with the standard or approved method governing use of the particular sampling tool. Typical equipment used for sampling and advancing shallow "hand auger" holes are Iwan samplers (which are rotated) or post hole diggers (which are operated like tongs). These techniques are slow but effective where larger pieces of equipment do not have access, and where very shallow holes are desired (less than 15 feet). Surficial soils must be composed of relatively soft and non-cemented formations to allow penetration by the auger.

5.2.10 Rock Drilling and Coring

When soil borings cannot be continued using augers or rotary methods due to the hardness of the soil or when rock or large boulders are encountered, drilling and sampling can be performed using a diamond bit corer in accordance with ASTM D2113.

Drilling is done by rotating and applying downward pressure to the drill rods and drill bit. The drill bit is a circular, hollow, diamond-studded bit attached to the outer core barrel in a double-tube core barrel. The use of single-tube core barrels is not recommended, as the rotation of the barrel erodes the sample and limits its use for detailed geological evaluation. Water or air is circulated down through the drill rods and annular space between the core barrel tubes to cool the bit and remove the cuttings. The bit cuts a core out of the rock which rises into an inner barrel mounted inside the outer barrel. The inner core barrel and rock core are removed by lowering a wire line with a coupling into the drill rods, latching onto the inner barrel and withdrawing the inner barrel. A less efficient variation of this method utilizes a core barrel that cannot be removed without pulling all of the drill rods. This variation is practical only if less than 50 feet of core is required.

Core borings are made through the casing used for the soil borings. The casing must be driven and sealed into the rock formation to prevent seepage from the overburden into the hole to be cored (see Section 5.3 of this guideline). A double-tube core barrel with a diamond bit and reaming shell or equivalent should be used to recover rock cores of a size specified in the project plans. The most common core barrel diameters are listed in Attachment A.

Soft or decomposed rock should be sampled with a driven split-barrel whenever possible or cored with a Denison or Pitcher sampler.

When coring rock, including shale and claystone, the speed of the drill and the drilling pressure, amount and pressure of water, and length of run can be varied to give the maximum recovery from the rock being drilled. Should any rock formation be so soft or broken that the pieces continually fall into the hole causing unsatisfactory coring, the hole should be reamed and a flush-joint casing installed to a point below the broken formation. The size of the flush-joint casing must permit securing the core size specified. When soft or broken rock is anticipated, the length of core runs should be reduced to less than 5 feet to avoid core loss and minimize core disturbance.

Advantages of core drilling include:

- Undisturbed rock cores can be recovered for examination and/or testing.
- In formations in which the cored hole will remain open without casing, water from the rock fractures may be recovered from the well without the installation of a well screen and gravel pack.
- Formation logging is extremely accurate.
- Drill rigs are relatively small and mobile.

Disadvantages include:

- Water or air is needed for drilling.
- Coring is slower than rotary drilling (and more expensive).
- Depth to water cannot accurately be determined if water is used for drilling.
- The size of the borehole is limited.

This drilling method is useful if accurate determinations of rock lithology are desired or if open wells are to be installed into bedrock. To install larger diameter wells in coreholes, the hole must be reamed out to the proper size after boring, using air or mud rotary drilling methods.

5.2.11 Drilling & Support Vehicles

In addition to the drilling method required to accomplish the objectives of the field program, the type of vehicle carrying the drill rig and/or support equipment and its suitability for the site terrain, will often be an additional deciding factor in planning the drilling program. The types of vehicles available are extensive, and depend upon the particular drilling subcontractor's fleet. Most large drilling subcontractors will have a wide variety of vehicle and drill types suited for most drilling assignments in their particular region, while smaller drilling subcontractors will usually have a fleet of much more limited diversity. The weight, size, and means of locomotion (tires, tracks, etc.) of the drill rig must be selected to be compatible with the site terrain to assure adequate mobility between borehole locations. Such considerations also apply to necessary support vehicles used to transport water and/or drilling materials to the drill rigs at the borehole locations. When the drill rigs or support vehicles do not have adequate mobility to easily traverse the site, provisions must be made for assisting equipment, such as bulldozers, winches, timber planking, etc., to maintain adequate progress during the drilling program.

Some of the typical vehicles which are usually available for drill rigs and support equipment are:

- Totally portable drilling/sampling equipment, where all necessary components (tripods, samplers, hammers, catheads, etc.) may be hand carried to the borehole site. Drilling/sampling methods used with such equipment include:

  - Hand augers and lightweight motorized augers.
  - Retractable plug samplers--driven by hand (hammer).
  - Motorized cathead - a lightweight aluminum tripod with a small gas-engine cathead mounted on one leg, used to install small-diameter cased borings. This rig is sometimes called a "monkey on a stick."

- Skid-mounted drilling equipment containing a rotary drill or engine-driven cathead (to lift hammers and drill string), a pump, and a dismounted tripod. The skid is pushed, dragged, or winched (using the cathead drum) between boring locations.

- Small truck-mounted drilling equipment using a Jeep, stake body or other light truck (4 to 6 wheels), upon which are mounted the drill and/or a cathead, a pump, and a tripod or small drilling derrick. On some rigs, the drill and/or a cathead are driven by a power take-off from the truck, instead of by a separate engine.

- Track-mounted drilling equipment is similar to truck-mounted rigs, except that the vehicle used has wide bulldozer tracks for traversing soft ground. Sometimes a continuous-track "all terrain vehicle" is also modified for this purpose. Some types of tracked drill rigs are called "bombardier" or "weasel" rigs.

- Heavy truck-mounted drilling equipment is mounted on tandem or dual tandem trucks to transport the drill, derrick, winches, and pumps or compressors. The drill may be provided with a separate engine or may use a power take-off from the truck engine. Large augers, hydraulic rotary and reverse circulation rotary drilling equipment are usually mounted on such heavy duty trucks. For soft-ground sites, the drilling equipment is sometimes mounted on vehicles having low pressure, very wide diameter tires and capable of floating; these vehicles are called "swamp buggy" rigs.

- Marine drilling equipment is mounted on various floating equipment for drilling borings in lakes, estuaries and other bodies of water. The floating equipment varies, and is often manufactured or customized by the drilling subcontractor to suit specific drilling requirements. Typically, the range of flotation vehicles include:

  - Barrel-float rigs - a drill rig mounted on a timber platform buoyed by empty 55-gallon drums or similar flotation units.

  - Barge-mounted drill rigs.

  - Jack-up platforms - drilling equipment mounted on a floating platform having retractable legs to support the unit on the sea or lake bed when the platform is jacked up out of the water.

  - Drill ships - for deep ocean drilling.

In addition to the mobility for the drilling equipment, similar consideration must be given for equipment to support the drilling operations. Such vehicles or floating equipment are needed to transport drill water, drilling supplies and equipment, samples, drilling personnel, etc. to and/or from various boring locations.

5.2.12 Equipment Sizes

In planning subsurface exploration programs, care must be taken in specifying the various drilling components, so that they will fit properly in the boring or well.

For drilling open boreholes using rotary drilling equipment, tri-cone drill bits are employed with air, water or drilling mud to remove cuttings and cool the bit. Tri-cone bits are slightly smaller than the holes they drill (i.e., 5-7/8-inch or 7-7/8-inch bits will nominally drill 6-inch and 8-inch holes, respectively).

For obtaining split-barrel samples of a formation, samplers are commonly manufactured in sizes ranging from 2 inches to 3-1/2 inches in outside diameter. However, the most commonly used size is the 2-inch O.D., 1-3/8-inch I.D. split-barrel sampler. When this sampler is used and driven by a 140-pound (± 2-pound) hammer dropping 30 inches (± 1 inch), the procedure is called a Standard Penetration Test, and the blows per foot required to advance the sampler into the formation can be correlated to the formation's density or strength.

In planning the drilling of boreholes using hollow-stem augers or casing, in which thin-wall tube samples or diamond core drilling will be performed, refer to the various sizes and clearances provided in Attachment A of this guideline. Sizes selected must be stated in the project plan documents.

5.2.13 Estimated Drilling Progress

To estimate the anticipated rates of drilling progress for a site, the following must be considered:

- The speed of the drilling method employed.

- Applicable site conditions (e.g., terrain, mobility between borings, difficult drilling conditions in bouldery soils, rubble fill or broken rock, etc.).

- Project-imposed restrictions (e.g., drilling while wearing personal protective equipment, decontamination of drilling equipment, etc.).

Based on recent experience in drilling average soil conditions (no boulders) and taking samples at 5-foot intervals, for moderate depth (30 feet to 50 feet) boreholes (not including installation or development of wells), the following daily rates of total drilling progress may be anticipated for the following drilling methods:

Drilling Method	Average Daily Progress (linear feet)
Hollow-stem augers	75'
Solid-stem augers	50'
Mud-Rotary Drilling	100' (cuttings samples)
Rotosonic Drilling	100'-160' (continuous core)
Reverse-Circulation Rotary	100' (cuttings samples)
Skid-Rig with driven casing	30'
Rotary with driven casing	50'
Cable Tool	30'
Hand Auger	Varies
Continuous Rock Coring	50'

5.3 Prevention of Cross-Contamination

A telescoping or multiple casing technique minimizes the potential for the migration of contaminated groundwater to lower strata below a confining layer. The telescoping technique consists of drilling to a confining layer utilizing a spun casing method with a diamond cutting or augering shoe (a method similar to the rock coring method described in Section 5.2.10, except that larger casing is used) or by using a driven-casing method (see Section 5.2.6 of this guideline) and installing a specified diameter steel well casing. The operation consists of three separate steps. Initially, a drilling casing (usually of 8-inch diameter) is installed followed by installation of the well casing (6-inch-diameter is common for 2-inch wells). This well casing is driven into the confining layer to ensure a tight seal at the bottom of the hole. The well casing is sealed at the bottom with a bentonite-cement slurry. The remaining depth of the boring is drilled utilizing a narrower diameter spun or driven casing technique within the outer well casing. A smaller diameter well casing with an appropriate length of slotted screen on the lower end, is installed to the surface.

Clean sand is placed in the annulus around and to a point of about 2 feet above the screen prior to withdrawal of the drilling casing. The annular space above the screen and to a point 2 feet above the bottom of the outer well casing is sealed with a tremied cement-bentonite slurry which is pressure-grouted or displacement-grouted into the hole. The remaining casing annulus is backfilled with clean material and grouted at the surface, or it is grouted all the way to the surface.

5.4 Cleanout of Casing Prior to Sampling

The boring hole must be completely cleaned of disturbed soil, segregated coarse material and clay adhering to the inside walls of the casing. The cleaning must extend to the bottom edge of the casing and, if possible, a short distance further (1 or 2 inches) to bypass disturbed soil resulting from the advancement of the casing. Loss of wash water during cleaning should be recorded.

For disturbed samples both above and below the water table and where introduction of relatively large volumes of wash water is permissible, the cleaning operation is usually performed by washing the material out of the casing with water; however, the cleaning should never be accomplished with a strong, downward-directed jet which will disturb the underlying soil. When clean out has reached the bottom of the casing or slightly below (as specified above), the string of tools should be lifted one foot off the bottom with the water still flowing, until the wash water coming out of the casing is clear of granular soil particles. In formations where the cuttings contain gravel and other larger particles, it is often useful to repeatedly raise and lower the drill rods and wash bit while washing out the hole, to surge these large particles upward out of the hole. As a time saver, the drilling contractor may be permitted to use a split-barrel (split-spoon) sampler with the ball check valve removed as the clean-out tool, provided the material below the spoon is not disturbed and the shoe of the spoon is not damaged. However, because the ball check valve has been removed, in some formations it may be necessary to install a flap valve or spring sample retainer in the split-spoon bit, to prevent the sample from falling out as the sampler is withdrawn from the hole. The use of jet-type chopping bits is discouraged except where large boulders and cobbles or hard-cemented soils are encountered. If water markedly softens the soils above the water table, clean out should be performed dry with an auger.

For undisturbed samples below the water table, or where wash water must be minimized, clean out is usually accomplished with an appropriate diameter clean out auger. This auger has cutting blades at the bottom to carry loose material up into the auger, and up-turned water jets just above the cutting blades to carry the removed soil to the surface. In this manner, there is a minimum of disturbance at the top of the material to be sampled. If any gravel material washes down into the casing and cannot be removed by the clean out auger, a split-barrel sample can be taken to remove it; bailers and sandpumps should not be used. For undisturbed samples above the groundwater table, all operations must be performed in a dry manner.

If all of the cuttings created by drilling through the overlying formations are not cleaned from the borehole prior to sampling, some of the problems which may be encountered during sampling include:

- When sampling is attempted through the cuttings remaining in the borehole, all or part of the sampler may become filled with the cuttings. This limits the amount of sample from the underlying formation which can enter and be retained in the sampler, and also raises questions as to the validity of the sample.

- If the cuttings remaining in the borehole contain coarse gravel and/or other large particles, these may block the bit of the sampler and prevent any materials from the underlying formation from entering the sampler when the sampler is advanced.

- In cased borings, should sampling be attempted through cuttings which remain in the lower portion of the casing, these cuttings could cause the sampler to become bound into the casing, such that it becomes very difficult to either advance or retract the sampler.

- When sampler blow counts are used to estimate the density or strength of the formation being sampled, the presence of cuttings in the borehole will usually give erroneously high sample blow counts.

To confirm that all cuttings have been removed from the borehole prior to attempting sampling, it is important that the site geologist measure the "stickup" of the drill string. This is accomplished by measuring the assembled length of all drill rods and bits or samplers (the drill string) as they are lowered to the bottom of the hole, below some convenient reference point of the drill string, then measuring the height of this reference point above the ground surface. The difference of these measurements is the

depth of the drill string (lower end of the bit or sampler) below the ground surface, which must then be compared with the depth of sampling required (installed depth of casing or depth of borehole drilled). If the length of drill string below grade is more than the drilled or casing depth, the borehole has been cleaned too deeply, and this deeper depth of sampling must be recorded on the log. If the length of drill string below grade is less than the drilled or casing depth, the difference represents the thickness of cuttings which remain in the borehole. In most cases, an inch or two of cuttings may be left in the borehole with little or no problem. However, if more than a few inches of cuttings are encountered, the borehole must be recleaned prior to attempting sampling.

5.5 Materials of Construction

The effects of monitoring well construction materials on specific chemical analytical parameters are described and/or referenced in SOP GH-2.8. However, there are several materials used during drilling, particularly drilling fluids and lubricants, which must be used with care to avoid compromising the representativeness of soil and ground water samples.

The use of synthetic or organic polymer slurries is not permitted at any location where soil samples for chemical analysis are to be collected. These slurry materials could be used for installation of long-term monitoring wells, but the early time data in time series collection of ground water data may then be suspect. If synthetic or organic polymer muds are proposed for use at a given site, a complete written justification including methods and procedures for their use must be provided by the site geologist and approved by the Project Manager. The specific slurry composition and the concentration of suspected contaminants for each site must be known.

For many drilling operations, potable water is an adequate lubricant for drill stem and drilling tool connections. However, there are instances, such as drilling in tight clayey formations or in loose gravels, when threaded couplings must be lubricated to avoid binding. In these instances, to be determined in the field by the judgment of the site geologist and noted in the site logbook, and only after approval by the Project Manager, a vegetable oil or silicone-based lubricant should be used. Petroleum based greases, etc. will not be permitted. Samples of lubricants used must be provided and analyzed for chemical parameters appropriate to the given site.

5.6 Subsurface Soil Samples

Subsurface soil samples are used to characterize subsurface stratigraphy. This characterization can indicate the potential for migration of chemical contaminants in the subsurface. In addition, definition of the actual migration of contaminants can be obtained through chemical analysis of the soil samples. Where the remedial activities may include in-situ treatment or excavation and removal of the contaminated soil, the depth and areal extent of contamination must be known as accurately as possible.

Engineering and physical properties of soil may also be of interest should site construction activities be planned. Soil types, grain size distribution, shear strength, compressibility, permeability, plasticity, unit weight, and moisture content are some of the physical characteristics that may be determined for soil samples.

Penetration tests are also described in this procedure. The tests can be used to estimate various physical and engineering parameters such as relative density, unconfined compressive strength, and consolidation characteristics of soils.

Surface protocols for various soil sampling techniques are discussed in SOP SA-1.3. Continuous-core soil sampling and rock coring are discussed below. The procedures described here are representative of

a larger number of possible drilling and sampling techniques. The choice of techniques is based on a large number of variables such as cost, local geology, etc. The final choice of methods must be made with the assistance of drilling subcontractors familiar with the local geologic conditions. Alternative techniques must be based upon the underlying principles of quality assurance implicit in the following procedures.

The CME continuous sample tube system provides a method of sampling soil continuously during hollow-stem augering. The 5-foot sample barrel fits within the lead auger of a hollow-auger column. The sampling system can be used with a wide range of I.D. hollow-stem augers (from 3-1/4-inch to 8-1/4-inch I.D.). This method has been used to sample many different materials such as glacial drift, hard clays and shales, mine tailings, etc. This method is particularly used when SPT samples are not required and a large volume of material is needed. Also, this method is useful when a visual description of the subsurface lithology is required. Rotosonic drilling methods also provide a continuous soil sample.

5.7 Rock Sampling (Coring) (ASTM D2113-83)

Rock coring enables a detailed assessment of borehole conditions to be made, showing precisely all lithologic changes and characteristics. Because coring is an expensive drilling method, it is commonly used for shallow studies of 500 feet or less, or for specific intervals in the drill hole that require detailed logging and/or analyzing. Rock coring can, however, proceed for thousands of feet continuously, depending on the size of the drill rig, and yields better quality data than air-rotary drilling, although at a substantially reduced drilling rate. Rate of drilling varies widely, depending on the characteristics of lithologies encountered, drilling methods, depth of drilling, and condition of drilling equipment. Average output in a 10-hour day ranges from 40 to over 200 feet. Down hole geophysical logging or television camera monitoring is sometimes used to complement the data generated by coring.

Borehole diameter can be drilled to various sizes, depending on the information needed. Standard sizes of core barrels (showing core diameter) and casing are shown in Figure 1.

Core drilling is used when formations are too hard to be sampled by soil sampling methods and a continuous solid sample is desired. Usually, soil samples are used for overburden, and coring begins in sound bedrock. Casing is set into bedrock before coring begins to prevent loose material from entering the borehole, to prevent loss of drilling fluid, and to prevent cross-contamination of aquifers.

Drilling through bedrock is initiated by using a diamond-tipped core bit threaded to a drill rod (outer core barrel) with a rate of drilling determined by the downward pressure, rotation speed of drill rods, drilling fluid pressure in the borehole, and the characteristics of the rock (mineralogy, cementation, weathering).

FIGURE 1

STANDARD SIZES OF CORE BARRELS AND CASING

Coring Bit Size	Nominal*		Set Size*	
	O.D.	I.D.	O.D.	I.D.
RWT	1 5/32	3/4	1.160	0.735
EWT	1 1/2	29/32	1.470	0.905
EX, EXL, EWG, EWM	1 1/2	13/16	1.470	0.845
AWT	1 7/8	1 9/32	1.875	1.281
AX, AXL, AWG, AWM	1 7/8	1 3/16	1.875	1.185
BWT	2 3/8	1 3/4	2.345	1.750
BX, BXL, BWG, BWM	2 3/8	1 5/8	2.345	1.655
NWT	3	2 5/16	2.965	2.313
NX, NXL, NWG, NWM	3	2 1/8	2.965	2.155
HWT	3 29/32	3 3/16	3.889	3.187
HWG	3 29/32	3	3.889	3.000
2 3/4 x 3 7/8	3 7/8	2 3/4	3.840	2.690
4 x 5 1/2	5 1/2	4	5.435	3.970
6 x 7 3/4	7 3/4	6	7.655	5.970
AX Wire line ___|___/	1 7/8	1	1.875	1.000
BX Wire line ___|___/	2 3/8	1 7/16	2.345	1.437
NX Wire line ___|___/	3	1 15/16	2.965	1.937

* All dimensions are in inches; to convert to millimeters, multiply by 25.4.

___|___/ Wire line dimensions and designations may vary according to manufacturer.

FIGURE 1
STANDARD SIZES OF CORE BARRELS AND CASING
PAGE TWO

Size Designations			Casing Coupling					Approximate Core Diameter	
Casing; Casing coupling; Casing bits; Core barrel bits	Rod; rod couplings	Casing O.D., Inches	O.D., Inches	I.D., Inches	Casing bit O.D., Inches	Core barrel bit O.D., Inches*	Drill rod O.D., Inches	Normal, Inches	Thinwall, Inches
RX	RW	1.437	1.437	1.188	1.485	1.160	1.094	—	0.735
EX	E	1.812	1.812	1.500	1.875	1.470	1.313	0.845	0.905
AX	A	2.250	2.250	1.906	2.345	1.875	1.625	1.185	1.281
BX	B	2.875	2.875	2.375	2.965	2.345	1.906	1.655	1.750
NX	N	3.500	3.500	3.000	3.615	2.965	2.375	2.155	2.313
HX	HW	4.500	4.500	3.938	4.625	3.890	3.500	3.000	3.187
RW	RW	1.437	Flush Joint	No Coupling	1.485	1.160	1.094	—	0.735
EW	EW	1.812			1.875	1.470	1.375	0.845	0.905
AW	AW	2.250			2.345	1.875	1.750	1.185	1.281
BW	BW	2.875			2.965	2.345	2.125	1.655	1.750
NW	NW	3.500			3.615	2.965	2.625	2.155	2.313
HW	HW	4.500			4.625	3.890	3.500	3.000	3.187
PW	—	5.500			5.650	—	—	—	—
SW	—	6.625			6.790	—	—	—	—
UW	—	7.625			7.800	—	—	—	—
ZW	—	8.625			8.810	—	—	—	—
—	AX __|__\	—	—	—	—	1.875	1.750	1.000	—
—	BX __|__\	—	—	—	—	2.345	2.250	1.437	—
—	NX __|__\	—	—	—	—	2.965	2.813	1.937	—

* All dimensions are in inches; to convert to millimeters, multiply by 25.4.

__\|__/ Wire line dimensions and designations may vary according to manufacturer.

NOMINAL DIMENSIONS FOR DRILL CASINGS AND ACCESSORIES. (DIAMOND CORE DRILL MANUFACTURERS ASSOCIATION). 288-D-2889

5.7.1 Diamond Core Drilling

A penetration of typically less than 6 inches per 50 blows using a 140-lb. hammer dropping 30 inches with a 2-inch split-barrel sampler shall be considered an indication that soil sampling methods may not be applicable and that coring may be necessary to obtain samples.

When formations are encountered that are too hard to be sampled by soil sampling methods, the following diamond core drilling procedure may be used:

- Firmly seat a casing into the bedrock or the hard material to prevent loose materials from entering the hole and to prevent the loss of drilling fluid return. Level the surface of the rock or hard material when necessary by the use of a fishtail or other bits. If the drill hole can be retained open without the casing and if cross-contamination of aquifers in the unconsolidated materials is unlikely, leveling may be omitted.

- Begin the core drilling using a double-tube swivel-core barrel of the desired size. After drilling no more than 10 feet (3 m), remove the core barrel from the hole and take out the core. If the core blocks the flow of the drilling fluid during drilling, remove the core barrel immediately. In soft materials, a large starting size may be specified for the coring tools; where local experience indicates satisfactory core recovery or where hard, sound materials are anticipated, a smaller size or the single-tube type may be specified and longer runs may be drilled. NX/NW size coring equipment is the most commonly used size.

- When soft materials are encountered that produce less than 50 percent recovery, stop the core drilling. If soil samples are desired, secure such samples in accordance with the procedures described in ASTM Method D 1586 (Split-barrel Sampling) or in Method D 1587 (Thin-Walled Tube Sampling); sample soils per SOP SA-1.3. Resume diamond core drilling when refusal materials are again encountered.

- Since rock structures and the occurrence of seams, fissures, cavities, and broken areas are among the most important items to be detected and described, take special care to obtain and record these features. If such broken zones or cavities prevent further advance of the boring, one of the following three steps shall be taken: (1) cement the hole; (2) ream and case; or (3) case and advance with the next smaller size core barrel, as conditions warrant.

- In soft, seamy, or otherwise unsound rock, where core recovery may be difficult, M-design core barrels may be used. In hard, sound rock where a high percentage of core recovery is anticipated, the single-tube core barrel may be employed.

5.7.2 Rock Sample Preparation and Documentation

Once the rock coring has been completed and the core recovered, the rock core shall be carefully removed from the barrel, placed in a core tray (previously labeled "top" and "bottom" to avoid confusion), classified, and measured for percentage of recovery as well as the rock quality designation (RQD). Each core shall be described, classified, and logged using a uniform system as presented in SOP GH-1.5. If moisture content will be determined or if it is desirable to prevent drying (e.g., to prevent shrinkage of clay formations) or oxidation of the core, the core shall be wrapped in plastic sleeves immediately after logging. Each plastic sleeve shall be labeled with indelible ink. The boring number, run number, and the footage represented in each sleeve shall be included, as well as designating the top and bottom of the core run.

After sampling, rock cores shall be placed in the sequence of recovery in well-constructed wooden boxes provided by the drilling contractor. Rock cores from two different borings shall not be placed in the same core box unless accepted by the Project Geologist. The core boxes shall be constructed to accommodate at least 20 linear feet of core in rows of approximately 5 feet each and shall be constructed with hinged tops secured with screws, and a latch (usually a hook and eye) to keep the top securely fastened down. Wood partitions shall be placed at the end of each core run and between rows.

The depth from the surface of the boring to the top and bottom of the drill run and run number shall be marked on the wooden partitions with indelible ink. A wooden partition (wooden block) shall be placed at the end of each run with the depth of the bottom of the run written on the block. These blocks will serve to separate successive core runs and indicate depth intervals for each run. The order of placing cores shall be the same in all core boxes. Rock core shall be placed in the box so that, when the box is open, with the inside of the lid facing the observer, the top of the cored interval contained within the box is in the upper left corner of the box, and the bottom of the cored interval is in the lower right corner of the box. The top and bottom of each core obtained and its true depth shall be clearly and permanently marked on each box. The width of each row must be compatible with the core diameter to prevent lateral movement of the core in the box. Similarly, an empty space in a row shall be filled with an appropriate filler material or spacers to prevent longitudinal movement of the core in the box.

The inside and outside of the core box lid shall be marked by indelible ink to show all pertinent data on the box's contents. At a minimum, the following information shall be included:

- Project name.
- Project number.
- Boring number.
- Run numbers.
- Footage (depths).
- Recovery.
- RQD (%).
- Box number and total number of boxes for that boring (Example: Box 5 of 7).

For easy retrieval when core boxes are stacked, the sides and ends of the box shall also be labeled and include project number, boring number, top and bottom depths of core and box number.

Prior to final closing of the core box, a photograph of the recovered core and the labeling on the inside cover shall be taken. If moisture content is not critical, the core shall be wetted and wiped clean for the photograph. (This will help to show true colors and bedding features in the cores).

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ATTACHMENT A

DRILLING EQUIPMENT SIZES

Drilling Component	Designation or Hole Size (Inches)	O.D. (Inches)	I.D. (Inches)	Coupling I.D. (Inches)
Hollow-stem augers (Ref. 7)	6 1/4	5	2 1/4	
	6 3/4	5 3/4	2 3/4	—
	7 1/4	6 1/4	3 1/4	—
	13 1/4	12	6	—
Thin Wall Tube Samplers (Ref. 7)	—	2	1 7/8	—
	—	2 1/2	2 3/8	—
	—	3	2 7/8	—
	—	3 1/2	3 3/8	—
	—	4 1/2	4 3/8	—
	—	5	4 3/4	—
Drill Rods (Ref. 7)	RW	1 3/32	23/32	13/32
	EW	1 3/8	15/16	7/16
	AW	1 3/4	1 1/4	5/8
	BW	2 1/8	1 3/4	3/4
	NW	2 5/8	2 1/4	1 3/8
	HW	3 1/2	3 1/16	2 3/8
	E	1 5/16	7/8	7/16
	A	1 5/8	1 1/8	9/16
	B	1 7/8	1 1/4	5/8
	N	2 3/8	2	1
				Wall Thickness (Inches)
Driven External Coupled Extra Strong Steel* Casing (Ref. 8)	2 1/2	2.875	2.323	0.276
	3	3.5	2.9	0.300
	3 1/2	4.0	3.364	0.318
	4	4.5	3.826	0.337
	5	5.63	4.813	0.375
	6	6.625	5.761	0.432
	8	8.625	7.625	0.500
	10	10.750	9.750	0.500
	12	12.750	11.750	0.500

* Add twice the casing wall thickness to casing O.D. to obtain the approximate O.D. of the external pipe couplings.

ATTACHMENT A
DRILLING EQUIPMENT SIZES
PAGE TWO

Drilling Component	Designation or Hole Size (Inches)	O.D. (Inches)	I.D. (Inches)	Coupling I.D. (Inches)
Flush Coupled Casing (Ref. 7)	RX	1 7/16	1 3/16	1 3/16
	EX	1 13/16	1 5/8	1 1/2
	AX	2 1/4	2	1 29/32
	BX	2 7/8	2 9/16	2 3/8
	NX	3 1/2	3 3/16	3
	HX	4 1/2	4 1/8	3 15/16
Flush Joint Casing (Ref. 7)	RW	1 7/16	1 3/16	
	EW	1 13/16	1 1/2	
	AW	2 1/4	1 29/32	
	BW	2 7/8	2 3/8	
	NW	3 1/2	3	
	HW	4 1/2	4	
	PW	5 1/2	5	
	SW	6 5/8	6	
	UW	7 5/8	7	
	ZW	8 5/8	8	
Diamond Core Barrels (Ref. 7)	EWM	1 1/2	7/8**	
	AWM	1 7/8	1 1/8**	
	BWM	2 3/8	1 5/8**	
	NWM	3	2 1/8	
	HWG	3 7/8	3	
	2 3/4 x 3 7/8	3 7/8	2 11/16	
	4 x 5 1/2	5 1/2	3 15/16	
	6 x 7 3/4	7 3/4	5 15/16	
	AQ (wireline)	1 57/64	1 1/16**	
	BQ (wireline)	2 23/64	1 7/16**	
	NQ (wireline)	2 63/64	1 7/8	
	HQ (wireline)	3 25/32	2 1/2	

** Because of the fragile nature of the core and the difficulty to identify rock details, use of small-diameter core (1 3/8") is not recommended.

S15 XRF SOP STANDARD OPERATING PROCEDURE FOR FIELD PORTABLE X-RAY FLUORESCENCE

Standard Operating Procedures for Field Portable X-Ray Fluorescence

1.0 PURPOSE

This procedure is for the semiquantitative analysis of metallic lead particles and chemical compounds of lead in soil using a field portable x-ray fluorescence (FPXRF) spectrometer. This method is a screening method to be used with confirmatory lead analysis using United States Environmental Protection Agency (USEPA)-approved methods.

2.0 SCOPE, APPLICATION, AND LIMITATIONS

2.1 Scope of Procedure

The FPXRF spectrometer can simultaneously analyze several chemical elements in a matter of minutes. The "light" elements that cannot be detected are: lithium, beryllium, sodium, magnesium, aluminum, silicon, and phosphorus. Generally, elements of atomic number 16 or greater can be detected and quantified by FPXRF. The focus of this SOP, however, is lead and its chemical compounds.

Although it is possible to used FPXRF to measure analytes in situ, this SOP requires removal of a soil or sediment sample from its native environment prior to analysis. By removing and homogenizing the sample prior to analysis, more precise and accurate results are obtained.

2.2 Limitations

When present in a sample, x-ray spectral lines from different elements can be very close in energy. If the energy difference between the two lines is less than the resolution of the detector, then the detector will not be able to fully resolve them. No instrument can fully compensate for this interference. It is important for an operator to understand this limitation of FPXRF instruments and to consult with the manufacturer of the FPXRF instrument to evaluate options to minimize this limitation. Therefore, the instrument manufacturer should be consulted prior to selecting a FPXRF instrument for lead analysis.

Typical lead detection limits range from about 10 to 50 mg/kg of lead in soil or sediment. Detection limits depend on several factors: the analyte of interest, the type of detector used, the type of excitation source, the strength of the excitation source, data acquisition times, physical matrix effects, chemical matrix effects, and interelement spectral interferences. These factors vary from sample to sample and instrument to instrument.

The results generated by this method are not directly comparable to results generated in a laboratory using acid digestion or other forms of chemical digestion followed by various analytical methods. The difference arises largely because the field samples and laboratory samples are treated differently prior to analysis and the measurement techniques do not measure exactly the same quantities. However, a correlation between laboratory and FPXRF data is often possible which will allow one to translate field results into equivalent laboratory results.

Grinding of a sample with a mortar and pestle prior to analysis, which may be conducted for quantitative analysis, is not required in this SOP.

2.3 Analyst Training

Use of this method is restricted to personnel both trained and knowledgeable in the operation of an XRF instrument or under the supervision of a trained and knowledgeable individual. Proper training for the safe operation of the instrument should be completed by the analyst prior to analysis.

3.0 ACRONYMS AND ABBREVIATIONS

DQO: Data quality objectives.
eV: Electron Volt. A unit of energy equivalent to the amount of energy gained by an electron passing through a potential difference of one volt.
FP: Fundamental parameter.
FPXRF: Field portable x-ray fluorescence.
MCA: Multichannel analyzer for measuring pulse amplitude.
Mg/kg: milligrams per kilogram.
MDL: Method detection limit.
NIST: National Institute of Standards and Technology.
PC: Personal computer.
PQL: Practical quantitation limit.
QC: Quality control.
REM: Roentgen Equivalent Man. The dosage of ionizing radiation that will cause the same amount of injury to human tissue as 1 roentgen of X-rays.
ROI: Region of interest.
RPD: Relative percent difference.
SRM: Standard reference material. A standard containing certified amounts of metals in soil or sediment.
SSCS: Site specific calibration standard.
TLD Thermal luminescent detector.
USGS: United States Geological Survey.
XRF: X-ray fluorescence.

4.0 RESPONSIBILITIES

Analyst/Chemist - Responsible for all aspects of sample preparation and analysis including equipment maintenance. Also responsible for maintaining chain-of-custody of samples after receipt from sampling personnel.

5.0 PROCEDURES

5.1 Summary of Method

Radiation from an electrically excited x-ray tube is used to generate characteristic x-ray emissions from elements in a sample. For measurement, the sample is positioned in front of the probe window. This is done by collecting a soil or sediment sample, preparing it as described in Section 5.9, and placing it in a sample cup or thin-walled plastic bag. The sample is then placed on top of the window inside a protective cover for analysis. Sample analysis is then initiated by exposing the sample to primary radiation from the source. Fluorescent and backscattered x-rays from the sample enter through the detector window and are converted into electric pulses in the detector. The detector in FPXRF instruments is usually either a solid-state detector or a gas-filled proportional counter. Within the detector, energies of the characteristic x-rays are converted into a train of electric pulses, the amplitudes of which are linearly proportional to the energy of the x-rays. An electronic multichannel analyzer (MCA) measures the pulse amplitudes, which is the basis of qualitative x-ray analysis. The length of time the sample actually is exposed to each source is referred to as the count time or data acquisition time. The number of counts at a given energy per unit of time is representative of the element concentration in a sample and is the basis for quantitative analysis. Most FPXRF instruments are menu-driven from software built into the units or from personal computers (PCs).

5.2 Interferences

5.2.1 Physical Matrix Effects

Physical matrix effects result from variations in the physical character of the sample. These variations may include such parameters as particle size, uniformity, homogeneity, and surface condition. For example, if any analyte exists in the form of very fine particles in a coarser-grained matrix, the analyte's concentration measured by the FPXRF will vary depending on how fine particles are distributed within the coarser-grained matrix. If the fine particles "settle" to the bottom of the sample cup, the analyte concentration measurement will be higher than if the fine particles are not mixed in well and stay on top of the coarser-grained particles in the sample cup.

Homogeneity is always a concern when dealing with soil and sediment samples. Every effort should be made to thoroughly mix and homogenize soil samples before analysis. Field studies have shown heterogeneity of the sample generally has the largest impact on comparability with confirmatory samples. Some of the effects of sample heterogeneity can be offset by making multiple measurements on a given sample.

5.2.2 Moisture Effects

When sample moisture content is between 5 and 20 percent, the overall error from moisture may be minimal. However, moisture content may be a major source of error when analyzing samples of soil that are saturated with water. This error is minimized by drying the samples in a convection or toaster oven. Microwave drying is not recommended because field studies have shown that microwave drying can increase variability between FPXRF data and confirmatory analysis and because metal fragments in the sample can cause arcing to occur in a microwave.

5.2.3 Chemical Matrix Effects and Spectral Overlap

Chemical matrix effects result from differences in the concentrations of interfering elements. These effects occur as either spectral interferences (peak overlaps) or as x-ray absorption and enhancement phenomena. Both effects are common in soils contaminated with heavy metals. Arsenic may interfere with lead analyses, but only when arsenic is present at relatively high concentrations. In addition, lead may interfere with arsenic analyses, particularly when lead is at concentrations greater than several 100 mg/kg (as observed at Site 10 at Portsmouth Naval Shipyard). The effects can be corrected mathematically through the use of fundamental parameter (FP) coefficients. The effects also can be compensated for using site specific calibration standards (SSCS), which contain all the elements present on site that can interfere with one another.

Elements that may cause chemical matrix effects are:
1) within 5 atomic numbers of lead in the period table of the elements,
2) excited by the source,
3) at a concentration greater than 10 percent of the target element's concentration, and
4) present in varying concentrations in the area under investigation.

Because the possible chemical interferences for lead are not likely to be present at significant concentrations in the soil samples, chemical interferences for lead are not expected.

The instrument manual should be consulted for further details.

5.2.4 Other Interferences

Ambient temperature changes can affect the electronic gain of the amplifiers thus producing instrument drift. Most FPXRF instruments have a built-in automatic gain control. If the automatic gain control is allowed to make periodic adjustments, the instrument will compensate for the influence of temperature changes on its energy scale. If the FPXRF instrument has an automatic gain control function, the

operator will not have to adjust the instrument's gain unless an error message appears. If an error message appears, the operator should follow the manufacturer's procedures for troubleshooting the problem. Often, this involves performing a new energy calibration.

5.3 Safety

5.3.1 Radiation Safety

Radiation safety for each specific instrument can be found in the operator's manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer. The analyst should be aware of the local state and national regulations that pertain to the use of radiation-producing equipment and radioactive materials with which compliance is required. Licenses for radioactive materials are of two types; (1) general license which is usually provided by the manufacturer for receiving, acquiring, owning, possessing, using, and transferring radioactive material incorporated in a device or equipment, and (2) specific license which is issued to named persons for the operation of radioactive instruments as required by local state agencies. There should be a person appointed within the organization that is solely responsible for properly instructing all personnel, maintaining inspection records, and monitoring x-ray equipment at regular intervals.

A copy of the radioactive material licenses and leak tests should be present with the instrument at all times and available to local and national authorities upon request. X-ray tubes do not require radioactive material licenses or leak tests, but do require approvals and licenses, which vary from state to state. In addition, fail-safe x-ray warning lights should be illuminated whenever an x-ray tube is energized. Provisions listed above concerning radiation safety regulations, shielding, training, and responsible personnel apply to x-ray tubes just as to radioactive sources. In addition, a log of the times and operating conditions should be kept whenever an x-ray tube is energized. Finally, an additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply. The danger of electric shock is as substantial as the danger from radiation but is often overlooked because of its familiarity.

Radiation monitoring equipment may be used with the handling of the instrument. Thermal luminescent detectors (TLD) in the form of badges and rings are used to monitor operator radiation exposure. If used, the TLDs should be worn in the area of most frequent exposure. The maximum permissible whole-body dose from occupational exposure is 5 Roentgen Equivalent Man (REM) per year. Possible exposure pathways for radiation to enter the body are ingestion, inhaling, and absorption. The best precaution to prevent radiation exposure is distance and shielding. The health and safety plan will specify the need for monitoring.

5.3.2 Protective Equipment

Analysts must wear disposable plastic gloves whenever sample aliquots are being transferred from one vessel to another. Consult the health and safety plan for other protection requirements.

5.4 Apparatus and Materials

Apparatus and materials may consist of the following as specified by the methods outline in this SOP and field specific requirements as provided in the QAPP.

<u>FPXRF spectrometer with data processing unit</u>: May be any instrument meeting the following specifications:

- Detection limit for lead in soil equal to 50 mg/kg or less.
- Can accept plastic sample cups or plastic bags as sample containers during measurement.
- Has either an x-ray tube or radioactive source as the excitation source.
- Has an output device that is legible under the anticipated field conditions
- Resolution of 0.17 keV or better.
- MDL no greater than 50 mg/kg of lead in soil.

Aluminum drying pans or aluminum foil: Sized suitably to hold as much as 50 grams of sample and fit into the drying oven.

Calibration verification check sample: A National Institute of Standards and Technology (NIST) or other Standard reference materials (SRMs) that contain lead in a concentrations range that is compatible with the project objectives to verify the accuracy of the instrument. SRMs can be obtained from the NIST, the U.S. Geological Survey (USGS), the Canadian National Research Council, and the national bureau of standards in foreign nations. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories. Acceptable limits for SRM percent recoveries are usually provided with the SRM. In their absence, a limit of ± 30 percent will be used as a guideline.

Instrument Blank: May be silicon dioxide, a Teflon block, a quartz block, "clean" sand, or lithium carbonate.

Lead calibration check standard: Supplied by the FPXRF manufacturer.

Method blank material for performing method blank checks: May be lead-free silica sand or lithium carbonate that undergoes the same preparation procedure as the samples.

Spare battery chargers.

Polyethylene sample cups: 31 millimeters (mm) to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

X-ray window film: Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 micrometers (µm) thick.

Sample containers: glass or plastic to store samples.

Sieves: Stainless steel, Nylon, or equivalent for preparing soil and sediment samples if necessary.

Trowels: for collecting soil samples.

Plastic bags: used for collection and homogenization of soil samples. May also be used as sample presentation device.

Drying oven: standard convection or toaster oven, for soil samples that require drying.

5.5 Sample Collection, Preservation, and Handling

Samples shall be provided to the FPXRF analyst in plastic bags or other lead-free containers. The analyst is responsible for maintaining chain-of-custody of all samples until all analyses have been successfully completed. No sample preservation is necessary. All samples shall be handled in accordance with sample handling SOPs in effect for the field event.

5.6 Preventive Maintenance

Refer to the instrument manual for specific manufacturer's recommendations.

5.7 Standardization or Calibration

Instrument standardization or calibration procedures vary among FPXRF instruments. Users of this SOP should follow the standardization/calibration procedures outlined in the operator's manual for each specific FPXRF instrument. Generally, three types of calibration procedures exist:

- fundamental parameters (FP) based on proprietary calibration algorithms,
- Empirical, based on site-specific calibration standards (SSCS),
- Compton scattering based on Compton peak ratios.

Some FPXRF instruments can be calibrated using multiple methods. SSCS calibrations will not be used for this project.

A set of pure element standards for commonly sought analytes is supplied by the instrument manufacturer, if required for the instrument; not all instruments require the pure element standards. The standards are used to set the region of interest (ROI) for each element. They also can be used as energy calibration and resolution check samples.

5.8 Quality Control

The quality control (QC) program required for this method includes analysis of QC check standards, blank analyses, duplicate analyses and field duplicate samples. For all the above areas, any identified problems and corrective action must be documented in the instrument run log, analysis narrative report, and instrument maintenance log or standards log (as applicable). Count times shall be at least 60 seconds but typically will be less than 120 seconds. The goal will be to obtain the same level of relative error for each sample analysis. Identical operating conditions will be used for each sample.

5.8.1 Energy Calibration Checks

5.8.1.1 Prior to conducting sample analyses, run an energy calibration check to determine whether an FPXRF instrument is operating within resolution and stability tolerances. The energy calibration check determines whether the characteristic x-ray lines are shifting, which would indicate drift within the instrument. This check also serves as a gain check in the event that ambient temperatures are fluctuating greatly (> 10 to 20°F).

5.8.1.2 Repeat the energy calibration check at the instrument manufacturer-recommended frequency. Generally, this will be at the beginning of each working day, after the batteries are changed or the instrument is shut off, at the end of each working day, and at any other time when the instrument operator believes that drift is occurring during analysis. A pure element such as iron, manganese, copper, or lead may be used for the energy calibration check. A manufacturer recommended count time per source should be used for the check.

5.8.2 Laboratory Blanks

Two types of blank samples shall be analyzed for FPXRF analysis: instrument blanks and method blanks.

5.8.2.1 At the beginning of each day, at the end of each day, and after every 20th sample or when potential contamination of the instrument is suspected an instrument blank is used to verify that no contamination exists in the spectrometer or on the probe window.

If the lead concentration in the blank exceeds the method detection limit (MDL), (see Section 5.10.3) check the probe window and other potentially contaminated instrument components for contamination. If contamination is not causing the elevated blank readings problem, "zero" the instrument according to manufacturer's instructions.

5.8.2.2 After every 20th sample analyze a method blank. If the method blank lead concentration exceeds the practical quantitation limit (PQL, see Section 5.10.4), identify the cause of the elevated lead concentration and reanalyze all samples since the last acceptable method blank.

5.8.3 Calibration Verification Checks

5.8.3.1 After performing each blank check (Section 5.8.3), analyze a calibration verification check sample to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest.

5.8.3.2 If the measured lead percent recovery (See Section 5.10.1) is less than 70 percent or greater than 130 percent, reanalyze the check sample. If the value continues to fall outside this acceptance range, the instrument should be recalibrated, or restandardized according to the manufacture instructions and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

5.8.4 Laboratory Duplicate Samples

Laboratory duplicate samples are two portions of the same sample that have been prepared and homogenized together, and then split and analyzed in the same manner by the XRF analyst.

5.8.4.1 Analyze a laboratory duplicate at a frequency of 1 per 20 or once per day, whichever is more frequent.

5.8.4.2 If the computed RPD (See Section 5.10.2) exceeds 50 percent reanalyze both samples. If the RPD again exceeds 50 percent RPD consider whether the high degree of imprecision is caused by sample heterogeneity or other causes. If it is attributed to sample heterogeneity, increase the number of readings made per sample to try to limit the imprecision and repeat the analyses. If this does not correct the problem notify the FOL.

5.9 Sample Analysis

Note:

Operation of the FPXRF instruments will vary according to the manufacturers' protocols. Before operating any FPXRF instrument, consult the manufacturer's manual. Most manufacturers recommend that their instruments be allowed to warm up for 15 to 30 minutes before analysis of samples. This will help prevent or minimize drift or energy calibration problems. Each FPXRF instrument should be operated according to the manufacturer's recommendations. Count times from 30 seconds to as long as 300 seconds per source can be employed, depending on the data quality needs of the project. As count times increase, the detector collects a larger number of x-rays from the sample, including more x-rays from elements that are present at comparatively lower concentrations. For that reason, the longer the count time, the lower the detection limits and the less uncertainty there is with a recorded result. Count time is user-selectable through the instrument's software.

Note:

Section 5.8 identifies the appropriate frequencies for conducting various QC sample analyses and the associated acceptance limits and corrective actions for potentially unsuitable conditions. The specified QC analysis frequencies are minimum frequencies. More frequent QC sample analyses are permitted, especially when diagnosing quality problems.

5.9.1 Ensure that calibration checks and blanks have been analyzed according to Sections 5.8.1 through 5.8.3. Count times shall be at least 60 seconds.

5.9.2 Acquire enough soil sample to fill an 8-ounce jar and separate from it all particles greater than the size of a pea.

5.9.3 Homogenize the remaining finer grained portion of the sample by simple mixing until it appears as uniform in texture and composition as practicable. Mixing may be done in a beaker or other suitable lead-free container. If the sample is moist and has high clay content, it may be kneaded in a plastic bag. Mixing shall continue for at least two minutes to ensure that the sample is well mixed.

5.9.4 Place approximately 20 to 50 grams (one U.S. nickel weighs about 5 grams) in a suitable container *e.g., aluminum drying pan) for drying.

5.9.5 Dry the homogenized sample from Step 5.9.4 for approximately 1 to 2 hours (or until sample is dry) in the toaster oven at a temperature not greater than 150°C.

5.9.6 Re-homogenize the dried sample aliquot in a beaker or other suitable lead-free container to obtain a well mixed soil sample. Mixing shall continue for at least one minute.

5.9.7 Place a portion (approximately 1.5 cubic inches) of the dried, homogenized sample aliquot into the instrument manufacturer's recommended sample cup (e.g., a 31.0-mm polyethylene sample cup (or equivalent) or a plastic bag.

5.9.8 If using a disposable plastic sample cup, ensure the cup is at least three-quarters full and cover with mylar (or other) film per the manufacturer's recommendations.

5.9.9 In accordance with the instrument operating instructions, present the sample to the instrument and acquire a measurement, then record the measurement. The count time shall be the same as was used for the calibrations, calibration checks, and blank analyses.

CAUTION

Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases by the square of the distance from the radioactive source. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample surface should be flat and smooth to provide a good contact surface.

5.9.10 Remix the sample in the plastic bag (or rotate the sample cup approximately one-third of a turn) then acquire another measurement.

5.9.11 Repeat Step 5.9.10.

5.9.12 Based on the degree of precision demonstrated by the three individual measurements, determine whether additional readings should be acquired on the sample. This determination shall be based on professional judgment of the FPXRF analyst and shall consider the degree of precision observed during calibration checks and previous sample analyses. The objective will be to ensure that the average reading reported for each sample is representative of the true sample concentration. If the analyst feels that non-representative readings are being obtained the analyst shall correct the analytical system or notify the FOL prior to continuing with analyses.

5.9.13 Ensure that measured results are reported to the following standards

- Results < 1000 mg/kg (or parts per million) are reported to two significant figures and results > 1000 mg/Kg are reported to three significant figures.
- All values < MDL shall be reported as the MDL and flagged with the letter "U".
- All values > MDL and < PQL shall be reported as is and flagged with the letter "B".

5.10 Calculations

5.10.1 Percent Recovery: The equation for determining percent recovery of calibration verification check standards and standard reference materials is:

$$\%R = \frac{\text{Experimental Concentration}}{\text{Certified or Known Concentration}} \times 100\%$$

5.10.2 Relative Percent Difference: The equation for determining relative percent difference for laboratory and field duplicate samples is:

$$RPD = \frac{|\text{Amount in Sample 1} - \text{Amount in Sample 2}|}{0.5\,(\text{Amount in Sample 1} + \text{Amount in Sample 2})} \times 100\%$$

5.10.3 Method Detection Limit (MDL):

Because the analyses governed by this SOP are semi-quantitative, the manufacturer-specified detection limit will be reported as the MDL unless the specified detection limit is less than 10 mg/kg. Care will taken to ensure that the appropriate count time is consistent with the reported detection limit. However, no value less than 10 mg/kg will be reported as an MDL.

5.10.4 Practical Quantitation Limit (MDL):

Multiply the MDL by 3 to obtain the PQL: PQL = MDL*3

6.0 REFERENCES

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USEPA, Region I, Northeast Waste Management Officials' Association (NEWMOA) Technology Review Committee Advisory Opinion. Innovative Technology: X-Ray Fluorescence Field Analysis. September 21, 1999.

ATTACHMENT 1-C

FIELD FORMS

Tetra Tech NUS, Inc.

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

DAILY ACTIVITIES CHECKLIST

Startup Checklist

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

Exit Checklist

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

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

Tetra Tech NUS, Inc.

DAILY ACTIVITIES RECORD

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

CLIENT: _____ **LOCATION:** _____

DATE: _____ **ARRIVAL TIME:** _____

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

CONTRACTOR: _____ **DRILLER:** _____

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

COMMENTS: _____

_____

_____

_____

APPROVED BY:

_____ _____

Tt NUS REPRESENTATIVE DRILLER

DATE: _____

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : _____

INSTRUMENT NAME/MODEL: _____

SITE NAME: _____

MANUFACTURER: _____

PROJECT No.: _____

SERIAL NUMBER: _____

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

Tetra Tech NUS, Inc.

BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING No.: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	MATERIAL DESCRIPTION: Color	MATERIAL DESCRIPTION: Material Classification	USCS *	Remarks	PID/FID Reading (ppm): Sample	PID/FID Reading (ppm): Sampler BZ	PID/FID Reading (ppm): Borehole**	PID/FID Reading (ppm): Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated reponse read.

Drilling Area Background (ppm): ____

Remarks: _____

Converted to Well: Yes _____ No _____ Well I.D. #: _____

MONITORING WELL MATERIALS
CERTIFICATE OF CONFORMANCE

Well Designation:_____ Site Geologist: _____

Site Name: _____ Drilling Company: _____

Date Installed: _____ Driller:_____

Project Name: _____ Project Number: _____

Material	Brand/Description	Source/Supplier	Sample Collected ?
Well Casing			
Well Screen			
End Cap			
Drilling Fluid			
Drilling Fluid Additives			
Backfill Material			
Annular Filter Pack			
Bentonite Seal			
Annular Grout			
Surface Cement			
Protective Casing			
Paint			
Rod Lubricant			
Compressor Oil			

To the best of my knowledge, I certify that the above described materials were used during installation of this monitoring well.

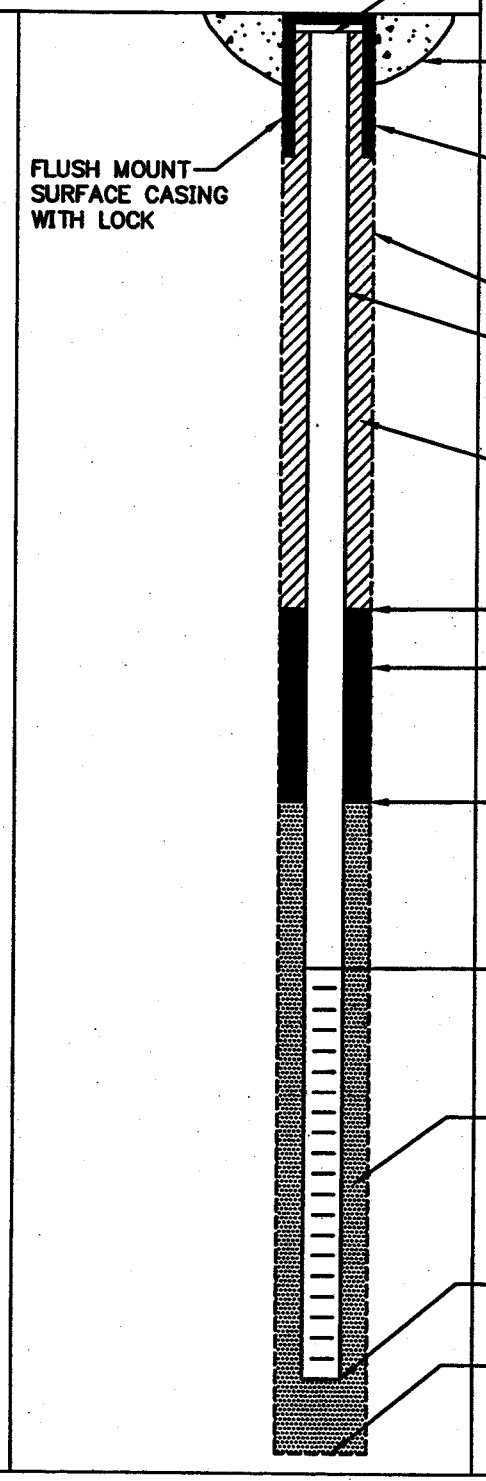
Signature of Site Geologist:_____

WELL NO.: _____

Tetra Tech NUS, Inc.

OVERBURDEN
MONITORING WELL SHEET
FLUSH - MOUNT

PROJECT _____	LOCATION _____	DRILLER _____
PROJECT NO. _____	BORING _____	DRILLING METHOD _____
DATE BEGUN _____	DATE COMPLETED _____	
FIELD GEOLOGIST _____		DEVELOPMENT METHOD _____
GROUND ELEVATION _____	DATUM _____	



ELEVATION TOP OF RISER: _____

TYPE OF SURFACE SEAL: _____

TYPE OF PROTECTIVE CASING: _____

I.D. OF PROTECTIVE CASING: _____

DIAMETER OF HOLE: _____

TYPE OF RISER PIPE: _____

RISER PIPE I.D.: _____

TYPE OF BACKFILL/SEAL: _____

ELEVATION/DEPTH TOP OF SEAL: ____/____

TYPE OF SEAL: _____

ELEVATION/DEPTH TOP OF SAND: ____/____

ELEVATION/DEPTH TOP OF SCREEN: ____/____

TYPE OF SCREEN: _____

SLOT SIZE x LENGTH: _____

TYPE OF SAND PACK: _____

DIAMETER OF HOLE IN BEDROCK: _____

ELEVATION / DEPTH BOTTOM OF SCREEN: ____/____

ELEVATION / DEPTH BOTTOM OF SAND: ____/____

ELEVATION/DEPTH BOTTOM OF HOLE: ____/____

BACKFILL MATERIAL BELOW SAND: _____

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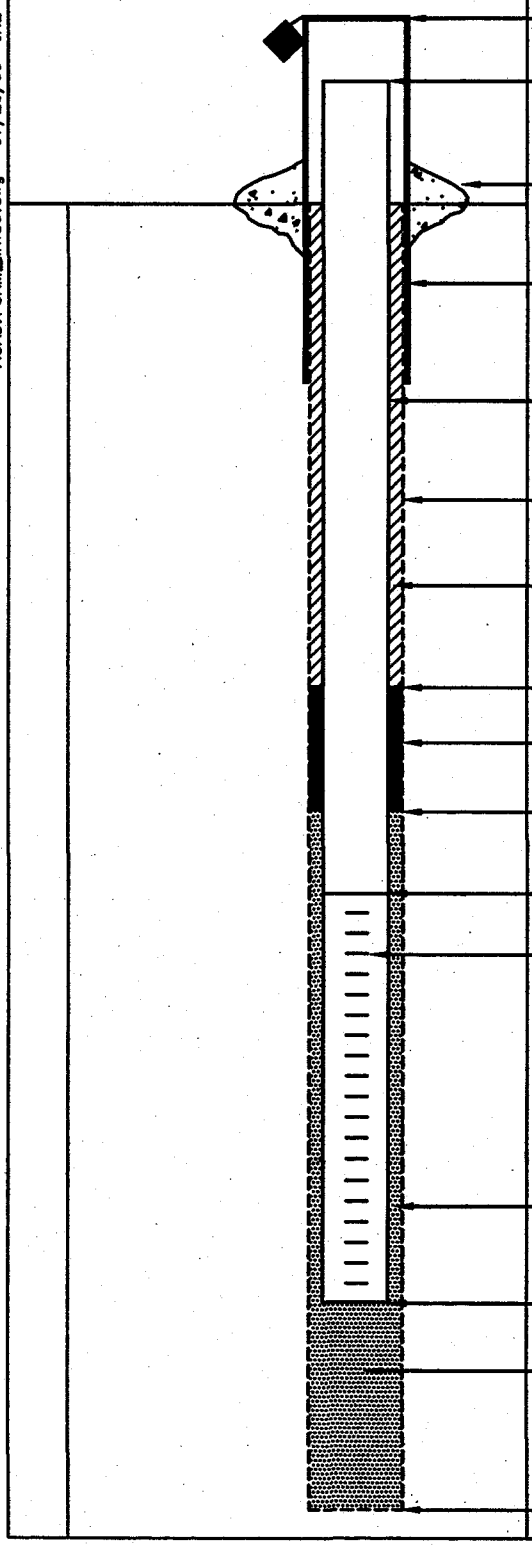
WELL NO.: _____

Tetra Tech NUS, Inc.

OVERBURDEN MONITORING WELL SHEET STICK-UP

PROJECT _____	LOCATION _____	DRILLER _____
PROJECT NO. _____	BORING _____	DRILLING METHOD _____
DATE BEGUN _____	DATE COMPLETED _____	
FIELD GEOLOGIST _____		DEVELOPMENT METHOD _____
GROUND ELEVATION _____	DATUM _____	



ELEVATION/HEIGHT OF TOP OF SURFACE CASING: ____/____

ELEVATION/HEIGHT OF TOP OF RISER PIPE: ____/____

TYPE OF SURFACE SEAL: _____

I.D. OF SURFACE CASING: _____
TYPE OF SURFACE CASING: _____

RISER PIPE I.D.: _____
TYPE OF RISER PIPE: _____

BOREHOLE DIAMETER: _____

TYPE OF BACKFILL: _____

ELEVATION/DEPTH TOP OF SEAL: ____/____

TYPE OF SEAL: _____

DEPTH TOP OF SAND PACK: _____

ELEVATION/DEPTH TOP OF SCREEN: ____/____

TYPE OF SCREEN: _____

SLOT SIZE x LENGTH: _____

I.D. OF SCREEN: _____

TYPE OF SAND PACK: _____

ELEVATION/DEPTH BOTTOM OF SCREEN: ____/____

ELEVATION/DEPTH BOTTOM OF SAND PACK: ____/____
BACKFILL MATERIAL BELOW SAND: _____

ELEVATION/DEPTH OF HOLE: ____/____

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Tetra Tech NUS, Inc.

GROUNDWATER LEVEL MEASUREMENT SHEET

Project Name: _____ **Project No.:** _____

Location: _____ **Personnel:** _____

Weather Conditions: _____ **Measuring Device:** _____

Tidally Influenced: Yes ___ No ___ **Remarks:** _____

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

* All measurements to the nearest 0.01 foot

LOW FLOW PURGE DATA SHEET

PROJECT SITE NAME: _____ WELL ID.: _____

PROJECT NUMBER: _____ DATE: _____

Time (Hrs.)	Water Level (Ft. below TOC)	Flow (mL/Min.)	pH (S.U.)	S. Cond. (mS/cm)	Turb. (NTU)	DO (mg/L)	Temp. (Celcius)	ORP mV	Salinity % or ppt	Comments

SIGNATURE(S): _____

PAGE___OF___

Tetra Tech NUS, Inc.

MONITORING WELL DEVELOPMENT RECORD

Page ___ of _____

Well: _____ Depth to Bottom (ft.): _____ Responsible Personnel: _____
Site: _____ Static Water Level Before (ft.): _____ Drilling Co.: _____
Date Installed: _____ Static Water Level After (ft.): _____ Project Name: _____
Date Developed: _____ Screen Length (ft.): _____ Project Number: _____
Dev. Method: _____ Specific Capacity: _____
Pump Type: _____ Casing ID (in.): _____

Time	Estimated Sediment Thickness (Ft.)	Cumulative Water Volume (Gal.)	Water Level Readings (Ft. below TOC)	Temperature (Degrees C)	pH	Specific Conductance (Units _____)	Turbidity (NTU)	Remarks (odor, color, etc.)

Tetra Tech NUS, Inc.

GROUNDWATER SAMPLE LOG SHEET

Project Site Name: _____
Project No.: _____

[] Domestic Well Data
[] Monitoring Well Data
[] Other Well Type: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____
Type of Sample:
[] Low Concentration
[] High Concentration

SAMPLING DATA:

Date: / Time:	Color (Visual)	pH (S.U.)	S.C. (mS/cm)	Temp. (^{0}C)	Turbidity (NTU)	DO (mg/l)	Salinity (%)	Other
Method:								

PURGE DATA:

Date:	Volume	pH	S.C.	Temp.	Turbidity	DO	Salinity	Other
Method:								
Monitor Reading (ppm):								
Well Casing Diameter & Material								
Type:								
Total Well Depth (TD):								
Static Water Level (WL):								
One Casing Volume(gal/L):								
Start Purge (hrs):								
End Purge (hrs):								
Total Purge Time (min):								
Total Vol. Purged (gal/L):								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

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

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

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

GRAB SAMPLE DATA:

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

COMPOSITE SAMPLE DATA:

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

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

Tetra Tech NUS, Inc.

SURFACE WATER SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Stream
[] Spring
[] Pond
[] Lake
[] Other: _____
[] QA Sample Type: _____

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

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

SAMPLING DATA:

	Color (Visual)	pH (S.U.)	S.C. (mS/cm)	Temp. (^{0}C)	Turbidity (NTU)	DO (mg/l)	Salinity (%)	Other
Date:								
Time:								
Depth:								
Method:								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD

Duplicate ID No.:

Signature(s):

Tetra Tech NUS, Inc.

QA SAMPLE LOG SHEET

Page___ of ___

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

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

SAMPLING DATA:

Date: _____
Time: _____
Method: _____

WATER SOURCE:

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

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

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

RINSATE INFORMATION (If Applicable):

Media Type: _____
Equipment Used: _____
Equipment Type:

[] Dedicated
[] Reusable

SAMPLE COLLECTION INFORMATION:

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

OBSERVATIONS / NOTES:

Signature(s):

Tetra Tech NUS, Inc.

FIELD PORTABLE XRF SAMPLE ANALYSIS LOG SHEET

Page___ of ___

Project Site Name: ______________________ Run Length/Duration: ______________________
Project No.: ______________________ Measurement Units: ______________________
Analyzed By: ______________________ Reference/Calibration: ______________________

SAMPLING AND FIELD PORTABLE XRF RESULTS DATA

SAMPLE ID	ANALYSIS DATE	ANALYSIS TIME	ELEMENT ANALYSIS	RESULT RUN 1	RESULT RUN 2	RESULT RUN 3	RESULTS AVERAGE	ERROR RUN 1	ERROR RUN 2	ERROR RUN 3	ERROR AVERAGE

Tetra Tech NUS, Inc.

CONTAINER SAMPLE & INSPECTION SHEET

Page_____ of _____

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

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

CONTAINER SOURCE

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

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

OTHER: _____

CONTAINER DESCRIPTION

COLOR: _____

CONDITION: _____

MARKINGS: _____

VOL. OF CONTENTS: _____

OTHER: _____

CONTAINER DISPOSITION

SAMPLED: _____

OPENED BUT NOT SAMPLED:
Reason _____
_____

NOT OPENED:
Reason _____
_____

CONTENTS DESCRIPTION

SINGLE PHASED: _____
_____

MULTIPHASE :

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

MONITOR READING

SAMPLE and/or INSPECTION DATE & TIME

_____HRS.

METHOD:_____

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

ANALYSIS

TETRA TECH NUS
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: _____

ATTACHMENT 1-D

ANALYTICAL LABORATORY SOPS
(provided on compact disc)

ATTACHMENT 1-D
LABORATORY STANDARD OPERATING PROCEDURES

- Empirical SOP-100 Metals Digestion/Preparation Methods 3005A, 3010A, 3020A, 3030, 3040A, 3050B, USEPA CLP ILM04.1 Aqueous and Soil/Sediment. USEPA Method 200.7, Standard Methods 3030C. Rev. 19
- Empirical SOP-103 Mercury Analysis in Water by Cold Vapor Techniques Methods USEPA SW-846 7470A, 245.1, CLP ILM04.1, Rev. 16
- Empirical SOP-104 Mercury Analysis in Soil/Sediment by Manual Cold Vapor Techniques Methods USEPA SW-846 7471A, 245.5, CLP ILM04.1, Rev. 17
- Empirical SOP-105 Metals Analysis by ICP Technique Methods USEPA 200.7, SW-846 6010B, 245.5, Standard Methods 19th edition 2340B, CLP ILM04.1, Rev. 14
- Empirical SOP-145 Determination of Inorganic Anions in Water by Ion chromatography Using the Dionex DX-500 Ion Chromatograph with Hydroxide Eluent and Dionex Column AS18 by Method 300.0/SW846 9056 Guidance, Rev. 6
- Empirical SOP 187 Electrometric Determination of pH Methods (USEPA) 150.1, Standard Methods 4500-H+B and 9040B For Waters, Liquids and Liquid Wastes, 9045C for Soils and Soil Wastes, Rev. 6
- Empirical SOP-198 Toxicity Characteristics Leaching Procedure (SW-846 Method 1311), Rev. 6
- Empirical SOP-201 GC/MS Semivolatiles by Method 625 and SW-846 8270C, 07/05/06, Rev. 18
- Empirical SOP-202 GC/MS Volatiles by Method 624 and SW-846 8260B, 09/29/06, Rev. 21
- Empirical SOP-208 GC/ECD Chlorinated Acid Herbicides by SW-846, Method 8151A, 10/04/06, Rev. 13
- Empirical SOP-211 GC/ECD Organochlorine Pesticides/PCBs by EPA Method 608 and SW-846 8081A/8082, 10/04/06, Rev. 9
- Empirical SOP-221 Total Organic Carbon (TOC) by Standard Method 5310C, USEPA 415.1, SW-846 9060, and Lloyd Kahn Method, Rev. 7
- Empirical SOP-231 GC/MS Low Level PAHs by Method 625 and SW-846 8270C, 07/05/06, Rev. 3
- Empirical SOP-300 GC/MS Semivolatiles BNA-Aqueous Matrix Extraction Using SW-846 Method 3510C for 8270C/625 Analysis, Rev. 17
- Empirical SOP-302 Pesticide/PCB Aqueous Matrix Extraction for EPA 608 and SW-846 8081A/8082 Using Method 3510C , Rev. 16
- Empirical SOP-304 Herbicides Aqueous Matrix by EPA SW-846 8151A , Rev. 11
- Empirical SOP-327 Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC) SW-846 Method 8330A, Rev. 14
- Empirical SOP-329 Soxhlet Extraction BNA and Pesticide/PCB Using SW-846 Method 3541, Rev. 16
- Empirical SOP-404 Laboratory Sample Receiving Log-in and Storage Standard Operating Procedures, Rev. 12
- Empirical SOP-405 Analytical Laboratory Waste Disposal, Rev. 4
- Empirical SOP-410 Standard Operating Procedures for Laboratory Sample Storage, Secure Areas, and Sample Custody, Rev. 6
- Columbia Analytical Services HPLC-Perchlorate, Perchlorate in Water, Soil, and Solid Wastes Using HPLC/ESI/MS.

STANDARD OPERATING PROCEDURE

for

PERCHLORATE IN WATER, SOILS AND SOLID WASTES USING HIGH PERFORMANCE LIQUID CHROMATOGRAPHY/ELECTROSPRAY IONIZATION/MASS SPECTROMETRY (HPLC/ESI/MS)

SOP No.: HPLC-6850
Revision: 3

June 12, 2007

Approved by: _____ _____
Supervisor Date

_____ _____
QA Coordinator Date

_____ _____
Laboratory Manager Date

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1 Mustard St. Suite 250
Rochester, NY 14609

Annual review of this SOP has been performed
and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date:_____

1. SCOPE AND APPLICATION

1.1. This SOP uses the DoD Perchlorate Handbook (May 2007) and EPA SW-846 Method 6850 for the Analysis of Perchlorate in Water, Soils, and Solid Wastes using High Performance Liquid Chromatography/Electrospray Ionization/Mass Spectrometry (HPLC/ESI/MS). Although this method is entirely capable of analyzing drinking water, most regulatory agencies require the use of a drinking water method in the 300 series. This method is not equivalent to any current drinking water method.

1.2. This SOP is written to specifically address the additional requirements of the DoD, however it is also compliant with SW-846 6850. Samples which are not from the DoD may be analyzed with or without the additional DoD requirements and be compliant with 6850. If the samples are not from the DoD, samples may be reported if they meet the less stringent 6850 requirements listed in Table 2.

1.3. This method is for the determination of perchlorate in groundwater, surface water, wastewater, soil, sediment and sludge using reverse phase high performance liquid chromatography (HPLC) coupled with electrospray ionization (ESI) mass spectrometry (MS). This SOP also details the procedures for tissue samples using an FDA procedure for the extraction. Other matrices (example – biota) may be applicable to this method if the lab demonstrates the acceptability of the matrix.

1.4. The reporting limits listed below are for guidance only. Reporting Limits are highly matrix-dependent and not always achievable.

Matrix	Reporting Limit
Water	0.20 ug/L
Soil	2.0 ug/kg
Tissue	0.60 ug/kg
Biota	To be determined
Milk	To be determined

2. METHOD SUMMARY

Solids are first extracted prior to analysis with reagent water using an ultrasonic bath. Tissue samples are extracted with acetonitrile and acetic acid and passed through an SPE cartridge. Water samples are not extracted. Extracts and water samples are filtered, if needed. An appropriate volume of the sample or sample extract is introduced into a HPLC/MS instrument. Perchlorate is separated by HPLC from the sample matrix, partially fragmented via negative electrospray ionization and detected by MS using mass-to-charge (m/z) ratios 83, 85 and 89. Quantitation is performed using m/z 83 and internal standard calibration. Isotopically-labeled perchlorate ($Cl^{18}O_4^-$), m/z 89, serves as the internal standard. The 83/85 isotopic ratio reflects the isotopic ratio of naturally occurring $^{35}Cl/^{37}Cl$ and is used for additional confirmation of perchlorate identification.

3. DEFINITIONS

3.1. **Preparation Batch**: a group of 20 or fewer samples of the same matrix prepared together on the same day. See ADM-BATCH for more information.

3.2. **Analytical Batch**: a group of 20 or fewer samples analyzed together. See ADM-BATCH for more information.

3.3. **Internal Standards –** Internal standards are analytes which are similar to the analytes of interest but which are not found in the samples. The chosen internal standards are used to calibrate the instrument's response.

3.4. **Continuing Calibration Verification (CCV):** standard analyzed at a routine frequency throughout the sequence to establish the instrument is meeting calibration criteria.

3.5. **Independent Calibration Verification (ICV):** standard analyzed from a source that is different than that used to calibrate the instrument. The ICV is a check on the purity and accuracy of the calibration standards and can be used in preparing matrix spike solutions for spiking of QA/QC samples.

3.6. **Relative Percent Difference (RPD):** measure of the relative difference between two values. See QAM.

3.7. **Method Detection Limit (MDL) -** also called LoD (Limit of Detection): a statistically derived value representing the lowest level of target analyte that may be measured by the instrument with 99% confidence that the value is greater than zero.

3.8. **Method Reporting Limit (MRL):** The minimum amount of a target analyte that can be measured and reported quantitatively. The MRL is equivalent to Practical Quantitation Level (PQL), reporting limit, and Limit of Quantitation (LoQ).

3.9. **QA/QC Samples**: Samples added to a sample preparation batch, or an analytical batch to provide quality assurance checks on the analysis.

3.9.1. **Duplicate Sample (DUP):** a laboratory duplicate. The duplicate sample is a separate sample aliquot that is processed in an identical manner as the sample proper.

3.9.2. **Laboratory Control Sample (LCS):** a method blank that has been fortified with the analyte of interest. Used to verify laboratory performance.

3.9.3. **Method Blank (MB):** a laboratory blank that is used to check that reagents and equipment are free of target analytes. The Method Blank follows the same preparative techniques as samples. Unless stated in project specific data quality objectives, reagents are considered free of target analyte contamination if the level detected in the Method Blank is less than the ½ MRL.

3.9.4. **Matrix Spike Sample (MS):** a duplicate sample aliquot that has been fortified with the analyte of interest. Used to observe the effects of the sample matrix on target analyte recovery.

3.9.5. **Matrix Spike Sample/ Matrix Spike Duplicate Sample (MS/MSD):** matrix spike samples prepared in duplicate. It is used to evaluate method precision and matrix effects.

4. INTERFERENCES

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

4.2. Sample extracts with any visible particulates should be filtered through a minimum 0.45-µm nominal pore size membrane or frit to remove particulates and prevent damage to the instrument, columns and flow systems. Filters specifically designed for IC or HPLC applications should be used.

4.3. Hydrogen sulfate ion $H^{34}SO_4^-$, formed from a minor sulfur isotope, is commonly present in samples. $H^{34}SO_4^-$ elutes before perchlorate but at high concentrations can tail into the retention time of the perchlorate peak and elevate its baseline at m/z 99. Quantitation of perchlorate based on m/z 83 and 89 avoids this potential interference from $H^{34}SO_4^-$.

4.4. Glassware must be scrupulously cleaned. Glassware should be rinsed as soon as possible after use with the last solvent used. This should then be followed by detergent washing with hot water and rinses with tap water and organic-free reagent water. After washing, the glassware should then be drained, dried, and heated in a laboratory oven at 130°C for two to three hours before reuse. Solvent rinses with acetonitrile may be substituted for the oven heating. After drying and cooling, glassware should then be stored in a clean environment to prevent accumulation of dust or other contaminants

5. SAFETY

5.1. The toxicity or carcinogenicity of each reagent used in this procedure may not be precisely defined. Material safety data sheets (MSDS) are available on each solvent and standard and should be reviewed as part of employee training.

5.2. Personal protective clothing (safety glasses, gloves and lab coat) are recommended when prepping samples, handling standards, or performing maintenance on pressurized systems.

5.3. The use of pressurized gases is required for this procedure. Care should be taken when moving cylinders. All gas cylinders must be secured to a wall or an immovable counter with a chain or a cylinder clamp at all times. Sources of flammable gases (e.g., pressurized hydrogen) should be clearly labeled.

5.4. The proper use of syringes and glass pipettes should be part of employee training. Care should be taken to avoid personal injury as a result of improper handling techniques.

5.5. Refer to the Safety Manual for further discussion of general safety procedures and information.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Water samples should be collected in purchased, pre-cleaned 125-mL polyethylene bottles.

6.2. For solids, collect samples in purchased clean, 4-oz amber glass jars.

6.3. Tissue samples should be frozen upon collection and kept frozen until just before analysis.

6.4. Prepare and analyze samples and extracts within 28 days of collection.

6.5. Care should be taken to avoid temperature extremes during shipment and storage

6.6. Samples will be refrigerated to 0-6°C upon sample login

6.7. For sample handling see SMO-GEN SOP.

7. APPARATUS AND EQUIPMENT

7.1. Volumetric flasks, 100-mL and other sizes as needed.

7.2. Automatic precision pipetters and disposable tips - 10-10,000 µL capacity or gas tight syringes.

7.3. Disposable autosampler vials, 2mL capacity.

7.4. Disposable plastic centrifuge tubes - 50 mL and 20 mL scintillation vials.

7.5. Disposable plastic micro-beakers.

7.6. Sample bottles - polyethylene or glass of sufficient volume to allow replicate analyses.

7.7. Disposable PTFE 0.45-µm, surfactant-free, membrane syringe filters.

7.8. Glass 5-mL luer-lock syringes or equivalent.

7.9. SPE Cartridges –

7.9.1. C_{18} (2000 mg) chromatography columns or equivalent for extract solution cleanup.

7.9.2. For tissue samples – Supelclean™, Envi™-carb SPE cartridges, 500 mg bed x 6 mL; P/N 57094; Supelco

7.10. High Performance Liquid Chromatograph/Mass Spectrometer - An analytical system combining a high performance liquid chromatograph for separation of the sample components, electrospray ionization interface and mass spectrometer for fragmentation and detection of sample components. Agilent LC/MS. See Appendix A of the Quality Assurance Manual for specific parts and serial numbers.

7.10.1. High Performance Liquid Chromatograph – The instrument contains a programmable solvent delivery system and all required accessories including injection loop, analytical columns, purging gases, etc. The chromatographic system must be capable of interfacing with a mass spectrometer.

7.10.2. Electrospray ionization (ESI) source – The ESI source generates gas phase ions of perchlorate from the liquid phase.

7.10.3. Mass Spectrometer – A quadrupole mass analyzer, Agilent 1100 LC/MS quadrupole mass spectrometer. Instrument tuning and instrument conditions are based on the manufacturer's instructions. Tuning is not required prior to daily analyses. Tuning parameters are available from the instrument manufacturer, along with mass tuning solutions and instructions on how to optimize the mass spectrometer.

7.10.4. Analytical column – 4 mm x 250 mm, peptides-impregnated, reverse-phase HPLC column. K' (Prime) Technologies, Inc. KP-RPPX250 column or equivalent.

7.10.5. Data system capable of performing analyte signal acquisition, peak integration, instrument calibration and analyte quantification

7.11. Analytical balance, capable of ±0.0001 g accuracy.

7.12. Centrifuge - capable of 5000 rpm and/or generating g-force of such strength as needed for providing adequate separation of a solid suspension and thus yielding a clear supernatant for sample injection and analysis.

8. PREVENTIVE MAINTENANCE

8.1. A maintenance log will be kept documenting maintenance performed on the analytical system. Log entries will include the date maintenance was performed, symptoms of the problem, serial numbers of major equipment upgrades or replacements (defined as any non-consumable part essential to operation), phone logs from technical support contacts, a description of the maintenance performed, a description of the check performed (attach standard data to logbook) to assure the system has returned to acceptable levels of operation, and the analyst's initials.

8.2. Any major maintenance performed on the instrument must be recorded in a maintenance log and must include the date, the person performing , the type and the outcome of the maintenance. Also include a data file number of a chromatogram exhibiting acceptable chromatography.

8.3. Routine maintenance such as changing columns, fresh mobile phase, changing guard columns or inlet filters is noted in the run log.

9. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

9.1. Reagents – follow guidelines for storage and expiration in ADM-DATANTRY. Follow appropriate reagent check procedures in ADM-CTMN.

9.1.1. Acetonitrile, CH_3CN – HPLC grade or equivalent

9.1.2. Acetic acid (glacial), CH_3CO_2H

9.1.2.1. 1% Acetic Acid – dilute 10 mL of glacial acetic acid to 900 mL with UPDI water.

9.1.3. UPDI water-HPLC grade: prepared by taking Ultra-Pure-De-Ionized water from the VOA lab Millipore polishing system and filtering through a 0.45u nylon filter using a sidearm flask and vacuum. Used for preparing all reagents, mobile phases, standards, and blanks. May also use purchased HPLC grade water – Fisher W5-4.

9.2. **Standards Preparation General Information and Disclaimers**

9.2.1. All Standards for this analysis are prepared in UPDI-HPLC grade water.

9.2.2. All of the preparation instructions are general guidelines. Other technical recipes may be used to achieve the same results. Example – a 20 mg/L standard may be made by adding 1 mL of 200 mg/L to 10 mLs or may be made by adding 4 mL of 50 mg/L to 10 mLs. The preparation depends upon the final volume needed and the initial concentration of the stock. Reasonable dilution technique is used.

9.2.3. The initial calibration curve given is typical, but also subject to variation due to detection levels needed. A minimum of five calibration standards is recommended as well as a blank standard. The lowest concentration level shall be at the reporting level and at least three times the MDL. The remaining levels should define the working linear range of the analytical system.

9.2.4. Vendors and vendors' products are sometimes listed for the ease of the analyst using this SOP, but products and purchased concentrations are examples only and subject to change at any time. All purchased standards are certified by the vendor. Certificates of Analysis are kept in the department until the standards are no longer being used – at which time they are filed with QA. Certificates of Analysis are available upon request. Purchased standards are routinely checked against an independent source for both analyte identification and analyte concentration.

9.2.5. All Standards must be traceable using the CAS lot system (ADM-DATANTRY).

9.3. Stock standard solution (1000 mg/L ClO_4^-) - This solution is purchased commercially as a certified standard. Stock standards may be stored at room temperature for a period of up to 12 months. Expiration dates should be clearly specified on the label.

9.4. Intermediate Standard (1.0 mg/L ClO_4^-) – Dilute 100 uL of 1000 mg/L Intermediate standard solution to 100 mL with UPDI-HPLC water. Working standard may be stored at room temperature for up to 1 month. Expiration dates should be clearly specified on the label.

9.5. Calibration standards – Prepare by adding various amounts of the 1.0 mg/L intermediate standard solution to UPDI-HPLC water directly in scintillation vials. Spike each calibration standard with 10uL of internal standard spiking solution. The final internal standard concentration is exactly the same (i.e. 1.0 µg/L $Cl^{18}O_4^-$) for each calibration standard. Although the 0.10 ug/L standard is included in the curve, the lab uses 0.20 ug/L as the reporting limit (LoQ).

Calibration Concentration (ug/L)	Volume (uL) of 1.0 mg/L ClO_4^- working std	Final Volume (mL)
0.10	1.0	10
0.20	2.0	10
0.50	5.0	10
1.00	10.0	10
2.00	20.0	10
5.00	50.0	10

9.6. Internal standard spiking solution (approximately 1000 µg/L $Cl^{18}O_4^-$) - Purchased commercially. Store at room temperature. Expires per manufacturer's indications or 6 months if no other indication is given.

9.6.1. Each standard and sample is spiked with 10 µL of internal standard spiking solution per 10 mL of sample or standard to obtain a final internal standard concentration of approximately 1 µg/L $Cl^{18}O_4^-$. An alternate internal standard concentration may be used provided that it falls within the same concentration range as the external calibration curve.

9.7. HPLC Mobile Phase – Prepare by combining 500 mL acetonitrile, 500 mL reagent water and 5 mL acetic acid.

9.8. Second Source Standard – The second source standard is purchased at 1000 mg/L from a different manufacturer than the Stock standard solution. Expires per manufacturer's indications or 6 months if no other indication is given.

9.9. Independent Calibration Verification Standard (ICV): The ICV is prepared from the second source standard at a concentration level within the calibration range of the method. Prepare fresh each use.

9.10. Continuing Calibration Verification Standard (CCV): The CCV is prepared from the intermediate standard solution at a concentration level within the calibration range of the method. Prepare fresh each use.

9.11. LODV – A standard prepared at a concentration approximately 2 times the MDL.

9.12. LCS/MS Spiking Solution (1 mg/L) – Dilute 100 uL of the 1000 mg/L Second Source Perchlorate Standard to 100 mL with UPDI-HPLC. Use this to spike the MS and the LCS. Store at room temperature. Expires in 6 months.

9.13. Laboratory Control Sample (LCS) (0.2 ug/L) – Dilute 2.0 uL of 1 mg/L LCS/MS solution to 10 mL UPDI-HPLC water. Process as a sample.

9.14. Interference Check Sample (ICS) (0.2 ug/L) –

9.14.1. ICS Matrix preparation - 5000 mg/L each of chloride, sulfate, carbonate by dissolving 8.240g NaCl +7.395g Na_2SO_4 +5.294g Na_2CO_3 in DI and diluting to 1000ml.

9.14.2. ICS – prepared upon use – add 2.0 mL of ICS matrix to 8.0 mL HPLC water. Add 2.0 uL of 1 mg/L perchlorate standard (second source = LCS/MS Spiking Solution) and 10 uL internal standard. Prepare and process as a sample.

9.15. MS Water (0.2 ug/L) – Add 2.0 uL of 1.0 mg/L spiking solution to 10 mL of water sample or UPDI-HPLC. Process as a sample.

9.16. The LCS/MS for Soil (+2.0 ug/kg) - Add 2.0 uL of 1.0 mg/L spiking solution to 1 g of Ottawa sand or soil sample. Process as a sample

9.17. LCS/MS for tissue (+2.5 ug/kg) – add 25uL of 1.0 mg/L spiking solution to 10 g of Gerber Turkey &Gravy baby food or sample. Process as a sample.

9.18. The LCS/MS for biota, and milk is under development.

9.19. MB – Process an analyte-free matrix as a sample. Use DI for water samples, Ottawa sand for soil samples, and Gerber Turkey&Gravy baby food for tissue. The MB for biota and milk is under development.

9.20. Mass Calibration Standard – a proprietary mix from Agilent. Use, store, and discard as per manufacturer's instructions.

9.21. Tune Verification Standard – a standard containing 100 ug/L of target perchlorate and internal standard. Data is acquired in scan mode.

10. RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. Final review and sign-off of the data is performed by the department supervisor or designee.

11. PROCEDURE

11.1. Be sure the analysts performing the test have a current IDC or CDC (see section 19). Be sure the instrument has a current MDL study (see section 14 and ADM-MDL).

11.2. Sample Preparation

11.2.1. Water Sample Preparation

11.2.1.1. Dispense 10.0 mL of sample or standard into a 20 mL disposable scintillation vial.

11.2.1.2. Add 10 µL of internal standard spiking solution to the sample tube, cap and shake until mixed well. The final concentration of internal standard in the sample must be exactly the same as that in the calibration standards

11.2.1.3. If needed, filter the sample solution using a plastic syringe fitted with a 0.45-µm PTFE membrane filter. Dispense the sample into an autosampler vial for analysis. If any of the samples in the prep batch are filtered, filter the entire prep batch, including QC.

11.2.2. Solid Sample Preparation

11.2.2.1. Weigh 1 g of solid sample, recording the weight to 0.01 g into a 20 mL disposable scintillation vial.

11.2.2.2. Add 10 mL of UPDI-HPLC water, 10 µL of internal standard spiking solution and 50 µL of acetic acid to the sample tube. Vortex the mixture, followed by sonication (ultrasonic bath) for a minimum of 10 minutes, followed by additional vortexing.

11.2.2.3. Centrifuge the sample for 5 minutes, if necessary, to separate the solids from the extract solution.

11.2.2.4. If needed, filter the supernatant extract solution using a plastic syringe fitted with a 0.45-µm PTFE membrane filter. Dispense the extract sample into an autosampler vial for analysis. If any of the samples in the prep batch are filtered, filter the entire prep batch, including QC. NOTE: The final concentration of internal standard in the sample extract must be exactly the same as that in the calibration standards.

11.2.2.5. If large quantities of organic contaminants are not believed to be present in the solid sample extract (i.e. supernatant extract is relatively clear and not highly colored), proceed to instrument analysis. If necessary, however, a cleanup step using a C_{18} column may be performed to remove organic contaminants from the supernatant extract solution.

11.2.2.5.1. Activate the C_{18} cartridge column by pushing approximately 5 mL of methanol through the column, followed by 5 mL of UPDI-HPLC water. A flow rate of approximately 0.5 mL/min is recommended. Care should be taken not to let the column become dry.

11.2.2.5.2. Pull approximately 6 mL of the supernatant extract solution through the activated column. Use vacuum, if necessary.

11.2.2.5.3. Discard the first 2 mL of eluted sample extract. The initially eluted sample extract is discarded because it is diluted by the solution remaining in the column following the activation process.

11.2.2.5.4. Collect the remaining eluted sample extract (approximately 4 mL) in a clean container. Filter the sample extract using a plastic syringe fitted with a 0.45-µm membrane filter. Dispense the extract sample into an autosampler vial for analysis. Quantitative recovery of the eluted extract sample is not necessary because there is no dilution or concentration of the sample.

11.2.3. Tissue Sample Preparation

11.2.3.1. Weigh a 10 g test portion of homogenized tissue sample into a 50 mL polypropylene conical centrifuge tube.

11.2.3.2. Using a 100 uL syringe, add 30 uL of 1000 ug/L labeled internal standard to the tube (Assume the total volume of the sample extract to be 30 mL. The final concentration will be 1 ug/L in the extract.)

11.2.3.3. Spike QC samples with target per section 9.

11.2.3.4. Add 15 mL of HPLC grade acetonitrile to each tube with tissue sample. Cap and shake by hand vigorously by hand for 1 minute.

11.2.3.5. Add 5 mL of 1% acetic acid and shake vigorously by hand for 2 minutes.

11.2.3.6. Centrifuge the tube for 15 minutes at setting 4.

11.2.3.7. Condition each Supelclean™, Envi™-carb SPE cartridge with 6mL of acetonitrile, allowing acetonitrile level to drop to frit, but not go dry. Add 6 mL of 1% acetic acid to cartridge and allow to elute. Turn up the vacuum and dry the cartridges for 30 seconds. Discard the aqueous portion used to prepare the cartridges.

11.2.3.8. Load conditioned Supelclean™, Envi™-carb SPE cartridges with approximately 5 mL of supernatant from the centrifuged samples. Allow to pass through the column quickly under vacuum while collecting eluent.

11.2.3.9. Passing the first 1 mL to waste, filter sample extract with 0.45 µm PTFE syringe filter directly into an autosampler vial.

11.3. Instrument Set-up

11.3.1. Instrument conditions for analysis are set to optimize response and separation of the analytes. Analysts are advised to adjust the HPLC systems to particular analytical needs. Once optimized, the same conditions are to be used for analysis of all standards, client samples, and QC samples. The current system uses the conditions in Table 1. Actual instrument conditions are optimized and recorded in the instrument daily run log.

11.3.2. Mass Calibration –

- Frequency – Upon instrument setup and as needed (QC failures, large ion mass deviations, major instrument maintenance, tune won't meet criteria)
- Acceptance Criteria – the calibration range must bracket the ion masses of interest without greatly exceeding the range. The most recent mass calibration must be used for an analytical run and the same mass calibration must be used for all data files in an analytical run. Mass calibration must be verified by acquiring a full scan continuum mass spectrum of a perchlorate stock standard. Perchlorate ions should be within ±0.3 *m/z* of mass 83, 85 and 89.
- Corrective action – Correct the problem and recalibrate if the mass calibration fails.

11.3.3. Retention Time

11.3.3.1. Due to the use of the internal standard, formal retention time studies are not needed. The retention time varies due to sample matrix conductivity, however the internal standard retention time varies at the same rate. It is the relative retention time between the sample and the internal standard which is most critical and must be monitored (see section 12). The retention times of the sample and internal standard are summarized and included in the report.

11.3.4. Interference Threshold study –

- Frequency – at initial setup and when major changes occur in the method's operating procedures (addition of cleanup procedure, column changes, mobile phase changes).

- Acceptance Criteria – Measure the threshold of common suppressors (chloride, sulfate, carbonate, bicarbonate) that can be present in the system without affecting the quantitation of perchlorate. The threshold is the concentration of the common suppressors where perchlorate recovery falls outside an 85-115% window.

11.4. Initial Calibration

11.4.1. Start up entire HPLC system and allow baseline response to settle.

11.4.2. All injections of standards and samples and associated QC are to be the same. 30 uL injections are used.

11.4.3. The first sample through the system should be an instrument blank. This blank is simply an injection of UPDI-HPLC and internal standard. As the first injection of the day, this helps to further settle the baseline and equilibrate the system.

11.4.4. A pre-curve blank is run to check for system and reagent cleanliness. Any potentially interfering peaks should be noted. This blank is prepared alongside the calibration standards.

11.4.5. Follow ADM-ICAL – if there are discrepancies between ADM-ICAL and this SOP, follow this SOP.

11.4.6. Process a minimum of 5 calibration standards and a blank (as described in Section 9). The calibration standards are pH adjusted, but not filtered. The lowest calibration standard must be at (or below) the reporting limit and the standard at the reporting limit must be at least three times the MDL.

11.4.7. Analyte responses are tabulated and recorded in the integration software.

11.4.8. Calibration Curve –

11.4.8.1. Response Factor - To obtain the average response factor, calculate the RF (as below) for each calibration point. Calculate the mean of the RFs to use for sample calculation. Calculate the Standard Deviation of the RFs and the Percent Relative Standard Deviation of the RFs to evaluate the linearity. The limit for the calibration is 20% RSD.

$$RF_s = \frac{(A_s)(C_{IS})}{(A_{IS})(C_s)}$$

Where:

A_x	=	Area of the characteristic quantitation ion for compound x.
A_{ISTD}	=	Area of the characteristic quantitation ion for the specified internal standard.
C_x	=	The concentration of the compound added (ppb).
C_{ISTD}	=	The concentration of the specified internal standard (ppb).

Calculate standard deviation (SD) and the percent relative standard deviations (%RSD) for each analyte from the mean with:

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(\overline{RF_i} - RF)^2}{N - 1}}$$

where:

$\underline{RF_i}$	=	RF for each of the calibration levels
RF	=	mean of initial RFs
N	=	Number of RF values (i.e., 5)

$$\%RSD = \frac{(SD)}{(\overline{RF_x})} 100.$$

where:

RSD	=	relative standard deviation.
RF	=	mean of 5 initial RFs for a compound.
SD	=	standard deviation of average RFs for a compound

11.4.9. The concentration corresponding to the absolute value of the calibration curve's Y-intercept must be ≤ LOD.

11.4.10. All of the ICAL criteria must be acceptable before samples may be analyzed.

11.4.11. Routinely, after the high point of the curve, a blank is run to prove that there is no carry-over from the autosampler. It should be noted if there are any target hits above ½ of the MRL.

11.4.12. Verify the calibration by analyzing an independent calibration verification standard. Acceptance criteria for the ICV is 85-115% recovery.

11.4.13. Samples immediately following an initial calibration and ICV may skip the system blank and the opening CCV. Set up the rest of the sequence to comply with the frequency requirements of the QC listed in section 12.

11.5. Daily Sample Analysis

11.5.1. When an ICAL is not run on the day of sample analysis, run blanks initially to warm up the system. Do not begin the sequence until the blanks are less than ½ the MRL. Analyze the tune verification and begin with a CCV. Set up the rest of the sequence to comply with the frequency requirements of the QC listed in section 12.

11.6. Evaluation of Sample Analysis

11.6.1. Examine solution concentrations of target analytes in the samples. If the concentration is greater than the high calibration standard, reanalyze the sample at a dilution. As a general rule, dilutions should be performed to keep target analytes in the upper half of the calibration range. See ADM-DIL.

11.6.2. Check for possible carryover. Pay particular attention to situations where samples containing low levels of target analyte were analyzed one or two injections after samples containing levels of target analyte near or over the high end of the calibration range. Reanalyze as needed to confirm.

11.6.3. Check peak integrations. Where possible, all integrations should be performed consistent with integration of the corresponding calibration standards and with ADM-INT.

11.6.4. Monitor the retention times of the ions in samples and QC. The retention times of the ClO_3^- m/z 83 peak and the IS $Cl^{18}O_3^-$ m/z 89 peak should not vary by more than 0.2 min from each other.

11.6.5. Monitor the mass ratio between ions 83/85. The ion ratio should fall within the established window of 2.3-3.8. If criteria not met, the sample(s) must be repeated. Reextract using cleanup procedures or alternative techniques to confirm the presence of perchlorate such as post spikes or dilutions to reduce the interference. Qualify the data and discuss in the case narrative as necessary.

12. QUALITY ASSURANCE/QUALITY CONTROL REQUIREMENTS

12.1. Daily tune verification – the daily tune verification is performed before the opening CCV. The quantitation ions must be within ±0.3 *m/z* of actual.

12.2. Initial Calibration Criteria: Calibration criteria is given in section 11.

12.3. Method Blank:

- Frequency - Process one MB with each preparation batch of 20 or fewer samples. The MB should be carried through all stages of sample preparation and analysis.
- Acceptance Criteria - The result of the MB must be less than ½ the reporting limit.
- Corrective Action - If it is not, determine the source of the contamination and reprep and reanalyze the associated samples.

12.4. Reagent blanks –

- Frequency – prior to calibration and after each batch is analyzed. When perchlorate concentrations in the samples are over the linear range, any samples which follow the overrange sample will be repeated if the sample concentrations were greater than ½ the RL.
- Acceptance Criteria – must be less than ½ RL.
- Corrective Action – reanalyze reagent blanks until no carryover is observed (<1/2 MRL). Reanalyze all samples processed since the contaminated blank. If the closing reagent blank fails, reanalyze all samples since the last clean reagent blank.

12.5. MS –

- Frequency - Prepare one MS (or MS/MSD pair) with each batch of 20 or fewer samples of the same matrix.
- Acceptance Criteria - The limit for the water and soil is 80-120% of the true value.
- Corrective Action - If recovery of MS fails on a sample batch the samples are acceptable only if the LCS meets acceptance criteria. If the LCS passes, but the MS fails, report the MS recoveries and note outliers in the case narrative.

12.6. LCS –

- Frequency - Prepare one LCS with each batch of 20 or fewer samples of the same matrix.
- Acceptance Criteria (recovery) - The limit for the water and soil LCS is 80-120%. The limits for biota and milk are under development.
- Corrective Action - If the LCS fails on a sample batch, reprep and reanalyze the sample batch. If a duplicate LCS was run with the batch and one LCS was slightly out, but the other LCS was in, the data is accepted.

12.7. DUP/MSD Precision –

- Frequency - Precision for each prep batch of 20 or fewer samples of the same matrix must be established by either analyzing a sample in duplicate (DUP) or analyzing a MS in duplicate (MSD).
- Acceptance Criteria – (for all samples above the reporting limit), the RPD value between samples or MS/MSD results must not exceed 15%.
- Corrective Action - examine the chromatograms and benchsheets for potential matrix interferences. Reanalyze the pair if deemed appropriate. The outlying RPD should be mentioned in the Case Narrative so that data may be flagged appropriately.

12.8. The ICV is discussed in Section 11.

12.9. CCV –

- Frequency – bracketing every 10 or fewer samples
- Acceptance Criteria - All samples must be bracketed by an acceptable CCV. The acceptance limit is 15% difference relative to the true value.
- Corrective Action - If the CCV does not meet acceptance criteria, correct the problem and achieve an acceptable CCV. Recalibration may be necessary. All samples bound by the unacceptable CCV must be reanalyzed with acceptable QC.

12.10. Internal standard area response verification:

- Frequency – The IS is added to every sample, spiked sample, standard, instrument blank, and method blank
- Acceptance Criteria - The internal standard area counts for each sample and QC standard must be monitored throughout the analysis and the IS area must be within ±50% of the average of the internal standard area counts of the ICAL standards.
- Corrective Action - If the internal standard area counts exceed ± 50% of the average of the internal standard area counts of the ICAL standards, rerun the sample at increasing dilutions until the acceptance criteria are met. If the criteria cannot be met with dilution, the interference are suspected and the sample must be reprepared using additional pretreatment steps. Thoroughly document corrective action in the case narrative.

12.11. Internal standard and Target retention times

- Frequency – IS is added to every sample, spiked sample, standard, instrument blank, and method blank.
- Acceptance Criteria – The RRT (relative retention time) must be 0.98-1.02. Calculate the RRT in a sample by dividing the retention time of the perchlorate ion by the retention time of the internal standard.
- Corrective Action – Thoroughly document in the case narrative.

12.12. MDL – see section 14 and ADM-MDL.

12.13. LODV

- Frequency – before and directly after every analytical batch of samples. It may be run more frequently to reduce reanalysis rate.
- Acceptance criteria – the recovery must be within 30% of the true value. Exception – any samples with concentrations above the MRL may be reported.
- Corrective Action – if there are samples below the MRL, correct the problem, obtain a compliant LODV and reanalyze affected samples.

12.14. ICS

- Frequency – one ICS is extracted with every batch of 20 or fewer samples. At least one ICS must be analyzed daily.
- Acceptance Criteria – the recovery must be within 30% of the true value.
- Corrective action – Correct the problem and reanalyze all samples associated with a failed ICS.

13. DATA REDUCTION AND REPORTING

13.1. Identify perchlorate in the sample chromatograms:

13.1.1. Compare the retention times of the ClO_4^-, m/z 83, peak and the internal standard $Cl^{18}O_4^-$, m/z 89, peak. The retention times should not vary by more than 0.2 minutes from each other. The relative retention time (Retention time of perchlorate ion divided by the retention time of the internal standard) must be 0.98 – 1.02.

13.1.2. Evaluate the relative abundances of the m/z 83 and 85 ions in the chromatogram. To confirm the presence of perchlorate, the 83/85 peak area counts ratio should be 2.3-3.8. Repeat the samples if ratio criteria not met or flag the data as estimated and discuss in the case narrative.

13.2. Calculate the perchlorate concentration for each sample using the internal standard calibration. Use m/z 83 and 89 from the internal standard for quantitation. Calculate the concentration of each of the sample matrices as follows:

13.2.1. Water Samples –

13.2.1.1. From Response Factor Calibration:

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})(D)}{(A_{is})(\overline{RF})}$$

Where:

A_s = Area (or height) of the peak for the analyte in the sample.

A_{is} = Area (or height) of the peak for the internal standard.

C_{is} = Concentration of the internal standard in the concentrated sample extract in µg/L.

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D=1. The dilution factor is always dimensionless.

$\overline{RF}$ = Mean response factor from the initial calibration. Unlike calibration factors for external standard calibration, the response factor is dimensionless.

13.2.2. Solid Samples

$$\text{Final Result } (\mu g/kg\ ClO_4^-) = \frac{(C)(V)(D)}{M}$$

Where:
C = Concentration in extract ($\mu g/L\ ClO_4^-$)
V = Volume of sample extract used for analysis (mL)
D = Dilution factor (if needed)
M = Mass of initial sample extracted (g)

13.3 LIMS adjusts the result on soil samples based on Dry Weight.

13.4 Adjustments to the Method Reporting Limit - MRLs are elevated for insufficient sample volume, matrix interference, and dilutions.

13.5 Data Review: Data is reviewed according to ADM-DREV.

14. METHOD PERFORMANCE

14.1. MDL study – refer to ADM-MDL for more information. If there are conflicts between ADM-MDL and this SOP, this SOP is to be followed.

14.1.1. Design - The MDL study must be performed in the matrix of interest using a standard at a concentration that is 1 to 10 times the estimated MDL value. The standard is taken through the entire preparation and analysis procedure.

14.1.2. Frequency - A full MDL study must be conducted at initial setup and subsequently once per 12 month period and when major changes occur in the method's operating procedures (addition of cleanup procedures, column changes, mobile phase changes). If no changes have been made to the method, quarterly MDL verification checks (see below) may be performed in lieu of the yearly MDL study.

14.1.3. Acceptance criteria – The study must produce an MDL value which is no more than 10 times lower than the spike level (10x rule). The calculated MDL must be 3 times less than the reporting limit. The MDL must be verified by an acceptable MDL verification check (see below) prior to the analysis of samples.

14.1.4. Corrective Action –

14.1.4.1. If the MDL study does not meet the 10x rule, adjust the spike concentration and repeat the study.

14.1.4.2. If the MDL is not 3 times less than the reporting limit, correct the problem and repeat the MDL study or raise the reporting limit accordingly.

14.1.4.3. If the MDL verification check fails, see below.

14.2. MDL verifications checks –

14.2.1. Concentration – the MDL verification check is a low-level spike at approximately 2 times the MDL taken through the entire preparation process.

14.2.2. Frequency – with each new MDL study prior to the analysis of samples and quarterly if an annual study will not be performed.

14.2.3. Acceptance criteria – must produce a signal at least 3 times the instrument's noise level and meet the isotope ratio criteria of 2.3 to 3.8. Recovery should be within 30% of its true value.

14.2.4. Corrective action – re-perform the MDL study. Perform maintenance, if needed. The MDL verification check must pass before samples are analyzed.

14.3. File the MDL study and the MDL check with the QA office.

14.4. Demonstration of Capability is performed upon instrument set-up , whenever a new analyst begins independent analysis, and annually thereafter according to ADM-TRANDOC and section 19 below. The documentation of this method performance is retained by the Quality Assurance office.

15. WASTE MANAGEMENT AND POLLUTION PREVENTION

Standards are prepared on an as-needed basis in small quantities and minimum sample volumes are used during analysis. All expired standards are disposed of as hazardous and hazardous samples are disposed of either as acidic aqueous waste or as hazardous aqueous waste depending on their analytes' concentrations. These tasks are turned over to staff qualified and equipped for disposal. See SMO-SPLDIS for more discussion.

16. CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

If data is produced that is out of control, the samples are to be re-analyzed with in-control QA whenever possible. See corrective actions in Section 12 of this SOP and in the applicable Figures in Section 12 of the Quality Assurance Manual.

17. CONTINGENCIES FOR HANDLING OUT OF CONTROL OR UNACCEPTABLE DATA

If data is produced that is out of control and is not to be re-analyzed due to sample volume restrictions, holding times, or QC controls can not be met, follow the procedures in Section 15 of the Quality Assurance Manual.

18. REFERENCES

EPA SW-846 *Perchlorate in Water, Soils and Solid Wastes Using High Performance Liquid Chromatography/Electrospray Ionization/ Mass Spectrometry*. Method 6850. January 2007.

DoD Perchlorate Handbook, Revision 1, Change 1, August 2007.

Rapid Determination of Perchlorate Anion in Foods by Ion Chromatography – Tandem Mass Spectrometry. U.S. Food and Drug Administration. Rev.2. April 12, 2005. (for tissue preparation)

19. TRAINING OUTLINE

19.1. Read the most current SOP. Demonstrate a general understanding of the methodology and chemistry. Follow policies in ADM-TRANDOC.

19.2. Observe Sample Preparation and Analysis. Follow HPLC Training Plan Form.

19.3. Participate in the methodology, documentation, and data reduction with guidance.

19.4. Demonstrate Competency by performing the analysis independently. Analyze four replicates of the LCS. If recovery is within acceptable limits, complete Training Plan Form, IDC certificate and summary spreadsheet and file with QA. An IDC study must be successfully performed before the new analyst may analyze samples independently. Continued demonstration of Capability is performed annually by analyzing a PE, blind, or new 4 replicate study.

20. METHOD MODIFICATIONS

- This lab uses dilute (1%) acetic acid in the extraction of soil samples and QC. This was part of the original draft methods for 6850. The lab has continued to use the acetic acid for the soils because it causes the particulates to settle more completely in the extract and produces better baselines in the chromatography.

21. INSTRUMENT- SPECIFIC ADDENDUM

Instrument manuals are located near the instrument.

22. ATTACHMENTS

- Table 1: Recommended Instrument Settings.
- Table 2: Method 6850 Requirements

23. CHANGES FROM PREVIOUS REVISION

- Changed SOP title and SOP code to reflect that this SOP can be used for any DoD and any 6850 work. Provided explanation in Section 1 about the use of this SOP for DoD and for 6850 without DoD. Added Table 2 for further clarification.
- Made changes below mostly due to 2007 revision to DoD Perchlorate Handbook
- 1- Noted that this SOP is not to be used for drinking water.
- 2-Added that waters are not extracted and that solids are extracted in an ultrasonic bath
- 3-Eliminated SSCV and that DoD names the first CCV of day = ICV
- 2, 4, 11 – Eliminated filtering of all samples. Samples are filtered when needed
- 9 and 11 – Eliminated 0.10 standard from ICAL. Noted that lowest ICAL standard is to be at (not below) the RL and that it must be more than 3 times the MDL. Eliminated 10 standard from ICAL.
- 9 – Eliminated purchased 8 mg/L IS stock – now purchasing 1000 ug/L.
- 9 and Table 1 - Modified mobile phase from 52.7% CHCN3/ 46.8%H20 / 0.5%acetic to 50/50/0.1
- 9- Changed the concentration of the IS in the injections from 2 ug/L to 1 ug/L – changed quantity of IS additions throughout.
- 9- Eliminated the addition of acetic acid to the ICAL standards, ICV, and CCV
- 9-Changed "Tuning Standard" to "Mass Calibration Standard" and added "Tune Verification Standard" as a separate item. Tune verification standard changed from 1000ug/L to 100 ug/L and added that data is aquired in scan mode
- 9-Increased ICS Matrix from 1000 mg/L each to 5000 mg/L each. Modified prep of ICS.
- 11 and Table 1-Changed volume of injections from 25 uL to 30 uL
- 11 – Changed from mandatory filtering of samples to optional with need to filter all of prep batch if any samples in batch are filtered.
- 11-Eliminated the addition of acetic acid in the water preparation
- 11- Changed from weighing solid samples in a centrifuge tube to a scintillation vial.
- 11- Changed the mass calibration from 99, 101, 107 ions to 83, 85, and 89 ions
- 11-Eliminated Retention Time studies – added explanation for use of RRTs instead.
- 11-Incorporated 4/27/07 changes regarding the use of the Response Factor
- 11-Deleted confusing information regarding Analytical Sequence – referenced section 12 for QC frequency - Added "Daily Sample Analysis" which notes use of warm up blanks, tune verification and opening CCV.
- 11- Added to the Evaluation of Sample Analysis the monitoring of the retention time difference between the sample (83) and internal standard (89).
- 12- Added daily tune verification
- 12 – Changed the MS limit from 75-125 to 80-120%
- 12 – Changed the LCS limit from 85-115 to 80-120%
- 12 – Changed the RPD limit from 20 to 15
- 12- Added "average of the internal standard area counts" for the comparison of the IS area response
- 13 – Changed the limit for the RT difference of from 0.1 to 0.2. Added the RRT limit here.
- 13 - Changed from reporting "as received" to corrected for dry weight

- 14 – Added that the MDL verification check must meet the isotope ratio limit. Changed the corrective action. Eliminated the option to adjust the reported MDL based on the MDL checks.
- 18-Added 6850 to References, Updated Perchlorate Handbook reference, Eliminated DoD SOP reference
- 20 – Added explanation of method modification of using acetic acid in soil extraction
- 22 – Added Table 2
- Table 1 – changed the Fragmentor from 200 V to 210 V

TABLE 1

EXAMPLE HPLC/MS INSTRUMENT SETTINGS

Autosampler

Injection Volume: 30 µL

Analytical Column

Column Temperature: 30 ºC

HPLC Pump

Flow Rate: 0.5 mL/min

Run Time: 13.0 min

Mobile Phase:

50% CH_3CN/ 50% H_2O / 0.1% CH_3CO_2H

Mass Spectrometer

Ionization Mode: Electrospray

Polarity: Negative

SIM Parameters:

SIM Ion	Fragmentor	Gain (EMV)	Actual Dwell
83	210 V	3.0	392 msec
85			392 msec
89			392 msec

Spray Chamber:

Gas Temperature	Drying Gas (N_2)	Nebulizer Pressure
320 ºC	12.0 L/min	50 psig

Capillary voltage:

Negative
1450 V

TABLE 2
Method 6850 Requirements

Samples which are NOT for DoD may be reported if the following are met instead of the requirements in the text of this SOP.

Blanks (Method, Reagent, any others)	**Result less than MRL**
LODV – may be run at MRL and called Low CCV or LLOQ – may satisfy CCV frequency requirement	**±50%**
Ion Ratio for identification confirmation	**Ratio within 30% of average peak area ratio of mid-range CCV**
Matrix Spike Soil Recovery	**70-130%**
RPD limit for MSD in samples in low range of curve	**50%**
Internal Standard Area Counts	**Compare to CCV in daily run instead of ICAL**
RRT of 0.98-1.02 not required	

METALS DIGESTION/PREPARATION

METHODS

USEPA SW846

3005A, 3010A, 3030C, 3031, 3050B

USEPA CLPILM 04.1 Aqueous & Soil/Sediment (NJDEP does not accept CLPILM 04.1 after June, 2003)

Addendum for USEPA CLPILM 05.2 Aqueous & Soil/Sediment

USEPA Methods for Chemical Analysis of Water and Wastes

200.7, Standard Methods 3030C

SOP NUMBER: **SOP-100**

REVISION NUMBER: **19**

APPROVED BY:

SECTION MANAGER

QUALITY ASSURANCE OFFICER

07/25/06

EFFECTIVE DATE

04/10/08

DATE OF LAST REVIEW

METALS DIGESTION/PREPARATION

References:
Methods 3005A/USEPA CLPILM0 4.1 Aqueous, 3010A, 3030C, 3031, 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.

C. OILS

1. Method 3031, "Digestion Procedure for Oils".

   a. This method is used to prepare samples containing oils, greases or waxes for analysis by inductively coupled argon plasma emission spectroscopy (ICP).

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, soil or oil 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 then is customary, a four times concentration 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 HNO_3 to a pH <2 immediately once sampled. If dissolved metals are to be analyzed the sample should be filtered before the HNO_3 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.

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.

C. OILS

1. Samples are taken in high density polyethylene 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. Method 3005A and USEPA CLPILM0 4.1, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP Spectroscopy", SW846, July, 1992.

   a. This digestion procedure may not be sufficiently vigorous to destroy some metal complexes.

2. Method 200.7

3. Method 3010A

   a. See method 6010B.

B. SOLIDS

1. Method 3050B

   a. 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.

2. USEPA CLP ILM0 4.1

   a. 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.

C. OILS

1. Method 3031

   a. These digestates can have very high dissolved solids, which may necessitate the use of internal standards, dilutions, or the method of standard addition.

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 (HNO_3). 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 (H_2SO_4). 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.

9. Base oil, analyte-free. Oil 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 bound logbook 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 logbook 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 logbook where information is recorded. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in a bound logbook. 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 a logbook. Each analyst must be sure to "Z" out the unused area of each logbook page.

2. PREPARATION

A. Laboratory control sample

1. Aqueous

a. This solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO_3, 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 digestion logbook.

b. For four times concentrated samples: The solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO_3, 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% HNO_3 and 5% HCl. Use 0.125 mLs HNO_3 and 0.3125 mLs HCl to each 50 mL vessel.

2. Solids

a. A 1.0 ±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. LCSS(date)A and then B 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 recorded in the sample digestion logbook.

3. Oils

a. **An analyte free oil MUST be used or explosive reactions can occur.** An analyte free oil (wesson oil which has been analyzed previously to prove that it is < MDL.) is spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier. i.e. LCSO(date)A and then B etc.

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 then 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. 85° for oil samples. Record in digestion logbook.

IX. PROCEDURE

A. Glassware preparation for oil 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% HNO_3 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 HNO_3 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 HNO_3 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 HNO_3 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 HNO_3. **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 HNO_3 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 HNO_3 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 digestion log book. 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 digestion log book.

h. The sample is now ready for analysis.

i. The digestion log 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 HNO_3 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 digestion log 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 of the well-mixed sample into the digestion vessel.

b. Add 1.5 mL concentrated HNO_3 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 log book. 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 HNO_3 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 USACE 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 digestion log 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. Adjust filtrate volume to 50 mL with DI water.

f. 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 homogenity. 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 equivelent 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 digestion log 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 digestion log.

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 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 digestion log book

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 <u>no</u> 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°C $\pm$ 5°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°C ± 5°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 USACE 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 digestion log 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.

E. Oils

1. Method 3031, "**Digestion Procedure for Oils**"

NOTE: THIS METHOD IS VERY TIME CONSUMING--DISCUSS SUB-CONTRACTING SAMPLES WITH YOUR SUPERVISOR AS SOON AS THEY COME IN THE DOOR.

a. Homogenize sample and Weigh approximately (to the nearest 0.01 g) a 0.5 g representative portion of the sample into a 250 mL beaker. Separate and weigh proportional aliquots of the phases if more than one phase is present. Record the exact mass in the digestion log. Larger or smaller sample sizes can be used if needed.

g. Add 0.5 g of potassium permanganate powder. If larger sample sizes are used, increase the amount of potassium permanganate so that the ratio of oil to postassium permanganate is still 1:1. Mix the oil and permanganate thoroughly until homogenous. Thick oils and tars that cannot be mixed should be heated to achieve mixing (the oil may react mildly). It is important to record the amount of potassium permanganate used for each sample if analysis is by ICP-AES and correction is to be made for the amount of manganese. If more than 10% of the sample is aromatic material, such as xylene, then the reaction will be incomplete. If this is the case, increase the amount of potassium permanganate. If the sample is a mixture of oil and other non-organic materials, reduce the amount of potassium permanganate.

NOTE: All steps requiring the use of acids should be conducted under a fume hood by properly trained personnel using appropriate laboratory safety equipment. This should include face shields and latex gloves.

h. Cautiously add 1.0 mL concentrated H_2SO_4, and stir with an appropriate stirring device. If larger sample sizes are used, increase the volume of the sulfuric acid so that the ratio of oil to sulfuric acid is 1 g to 2 mL. The H_2SO_4 can be added dropwise or all at once, depending on analytical needs. (Generally, dropwise is preferred when low reporting limits are needed.)

NOTE: To prevent a strong exothermic reaction, H_2SO_4 should be added dropwise to all samples unfamiliar to the analyst and to all samples that are known to be highly reactive.

The reaction can take several seconds to begin, but when it occurs it will be very quick, vigorous, and exothermic. Generally larger sample sizes will react faster than smaller. Likewise, lower average molecular weight materials will react faster than heavier. Do not be mislead by an initial lack of reactivity. A grey-white vapor will be ejected from the beaker (SO_3) and splattering and bubbling can occur. The beaker will become very hot. This step is complete when no more gases are given off and the sample would be a thick black lumpy paste. Allow the beaker to cool as needed.

NOTE: Care must be taken when working with very light organic materials, such as diesel fuels, as they may flash. Generally, the lower the average molecular weight of the meterial correlates to a greater danger of flashing. The danger of flashing is reduced by adding the sulfuric acid dropwise.

NOTE: If more than 10% of the sample is aromatic material, such as xylene, only a little grey-white vapor will form. This will reduce accuracy and complicate nebulization. If there is a signigicant amount of non-hydrocarbon material, a sputtering recation will occur and black MnO_2 particulates will be given off. See section (b.) above under procedure.

i. Add 2 mL of concentrated HNO_3 and stir. This reaction will be slightly exothermic. If larger sample sizes are used, it is not always necessary to increase the volume of HNO_3 proportionately, depending on analytical needs. Some reddish-brown vapor (NO_2) may be given off. Allow the reaction to continue until complete, that is when the digestate no longer gives off fumes. Allow the beaker to cool as needed.

j. Add 10 mL of concentrated HCl and stir. If larger sample sizes are used, it is not always necessary to increase the volume of HCl proportionately, depending on analytical needs. This reaction will be slightly exothermic and gas formation and foaming will occur. Lighter oils will foam more than will heavier oils. If excess foaming occurs, add water to prevent sample loss. Allow the beaker to cool as needed.

k. Heat the beaker until there is no further gas evolution. (temperature should not exceed 150 °C to prevent volatilization). There may be additional foaming or other milder reactions which may result in overflow from the beaker. If excess foaming occurs, either remove the beaker from the heating source until foaming subsides or add sufficient

water to prevent overflow. The final digestate should be a clear yellow liquid with black or dark reddish-brown particulates.

l. Filter the digestate through Whatman 41 filter paper and collect filtrate in a volumetric flask or beaker.

m. Wash the digestion beaker and filter paper, while still in the funnel, with no more than 5 mL of hot HCl.

   NOTE: The purpose of this next step is to recover antimony, barium, and silver that may not have been completely solubilized. If the sample is not being prepared for these analytes, the next step may be skipped.

n. (Optional) After having washed the filter paper, remove the filter and residue from the funnel and place it back in the beaker. Add 5 mL of conc. HCl and place the beaker back on the heating source until the filter paper dissolves (temperature should not exceed 150 °C ±5 °C to prevent volatilization). Remove the beaker from the heating source and wash the cover and sides with reagent grade water and then filter the residue and collect the filtrate in the same flask or beaker as in sections f. and g. above. Allow the filtrate to cool and quantitatively transfer to a volumetric flask. Bring to volume.

o. (Optional) If the filtrate is collected in a beaker, the filtrate can be heated again to drive off excess HC1. This can reduce matrix effects in sample introduction (temperature should not exceed 150 °C ± 5 °C to prevent volatilization). When sufficient HCl has been removed, remove the beaker from the heating source, allow to cool, and then transfer the contents to a volumetric flask and bring to volume. However, if too much HCl is removed, barium, silver and antimony can be lost.

p. Analyze the flitrate by ICP-AES. Depending on the final volume selected, the total solids in the digestate may be high enough to cause nebulization problems. Problems due to high dissolved solids may be corrected by 1) following optional Section i., 2) using internal standards, 3) using flow injection analysis, or 4) using other matrix correction procedures.

   Manganese Removal Steps

   NOTE: The purpose of these next steps is to remove the manganese in the digest by precipitating it as manganese ammonium phosphate

under alkaline conditions. Elements that do not form insoluble phosphates, such as arsenic, are filtered out and can be analyzed at lower concentrations.

q. Take the digestate, or portion of digestate and reduce the volume to remove as much HCl as possible without going below 10 mL. Then add conc. NH_4OH until pH is 7 or greater. For most matrices, the digestate will change colors (often from yellow to brown) at pH 7. A mild exothermic reaction will occur immediately.

r. Add at least 2 g ammonium phosphate for each 1 g of potassium permanganate used in the digestion and stir. An excess of phosphate is needed for good analyte recovery. Then add enough water and mix to ensure maxium precipitation. A pink or yellow silky amorphous precipitate, manganese ammounium phosphate, will form. If too much NH_4OH is used some of the manganese ammonium phosphate can be solubilized. Stir until precipitation is complete. Some ammonium phosphate may remain unreacted at the bottom of the beaker.

s. Filter the digestate through Whatman 41 filter paper (or equivalent) and collect filtrate in a volumetric flask or beaker.

t. Heat the filtrate to volatilize the ammonia (temperature should not exceed 150 °C ± 5 °C to prevent volatilization). The volume of filtrate can be reduced by heating to no less thatn 10 mL. If too much water is removed ay ammonium chloride formed will solidify. If this occurs, either add enough water to dissolve the solids or filter out the solids and wash the residue with deionized water. The filtrate can be analyzed by ICP-AES.

u. The digestion log 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. **NOTE: The blank for OILs MUST include an analyte-free oil or explosive reactions can occur.**

   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.

   e. Recoveries of standard reference materials or laboratory control samples spiked with organo-metallic standards recoveries should be **±25% of their true values for OILS.**

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

   v. For TCLP samples, a spike 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 aliquot must be added to the extract after filtration but before preservation.)

   d. The CLH project requires that a high and a low spike be prepared and analyzed. Spikes shoud 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-405, 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-431 for common environmental laboratory definitions.

MERCURY ANALYSIS IN WATER

BY MANUAL COLD VAPOR TECHNIQUE

METHODS USEPA SW846 7470A and 245.1 CLP-M 4.1 (NJDEP does not accept CLPILM 04.1 after June, 2003), Addendum for USEPA CLP ILM 05.2

SOP NUMBER: **SOP-103**

REVISION NUMBER: **16**

APPROVED BY:

SECTION MANAGER

TECHNICAL DIRECTOR

EFFECTIVE DATE: **01/28/09**

DATE OF LAST REVIEW **01/28/09**

MERCURY ANALYSIS IN WATER BY MANUAL COLD VAPOR

References:
SW846 Method 7470A
USEPA Method 245.1
USEPA SOW ILM04.1
See Addendum for SOW ILM05.2

I. SCOPE AND APPLICATION

A. This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. This method can also be used for sludge-type wastes. All samples must be subjected to an appropriate dissolution procedure prior to analysis.

B. In addition to inorganic forms of mercury, organic materials may also be present. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. Potassium permanganate oxidizes many of these compounds, but recent studies have shown that a number of organic mercurials, including phenol mercuric acetate and methyl mercuric chloride, are only partially oxidized by this reagent. Potassium persulfate has been found to give approximately 100% recovery when used as the oxidant step following the addition of the permanganate has been included to insure that organo-mercury compounds, if present, will be oxidized to the mercuric ion before measurement. A heat step is required for methyl mercuric chloride when present in or spiked to a natural system. For distilled water the heat step is not necessary.

C. The range of the method may be varied through instrument and/or recorder expansion. Using a 30 mL sample, a detection limit of 0.2 µg Hg/L can be achieved.

II. SUMMARY OF METHOD

A. The flameless AA procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. Organic mercury compounds are oxidized and the mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of a flow injection Mercury system. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner.

III. SAMPLE HANDLING AND PRESERVATION

A. Samples are preserved by acidification with nitric acid to a pH of 2 or lower immediately at the time of collection, and refrigeration to 4°C.

B. The holding time for the mercury digestion is 28 days from time of sampling.

IV. INTERFERENCES

A. Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.

B. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

C. Sea waters, brines and industrial effluents high in chlorides require additional permanganate (as much as 6.25 mL in 30 mL of sample). During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation at 253 nm. Care must be taken to assure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by using an excess of hydroxylamine sulfate reagent (6.25 mL to 30 mL of sample).

D. Samples containing high concentrations of oxidizable organic materials, as evidenced by high chemical oxygen demand values, may not be completely oxidized of organic mercury will be low. The problem can be eliminated by reducing the sample volume or by increasing the amount of potassium persulfate (and consequently stannous chloride) used in the digestion.

V. Safety

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

B. The toxicity and carcinogenicity of each reagent in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices. Normal accepted laboratory practices should be followed during reagent preparation and instrument operation. Always wear safety glasses or full-face shield for eye protection when working with these reagents. Each laboratory is responsible for maintaining a current safety plan, a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

C. Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. The analyst should use chemical resistant gloves when handling concentrated mercury standards.

D. The analyst should make sure that the system is vented to fresh permanganate in a bottle located at the back. Otherwise Hg vapors could be vented to the room.

VI. EQUIPMENT/APPARATUS

A. Perken Elmer Flow injection Mercury system

B. Mod Block Digester set to maintain 95±2°C for 2 hours.

C. Polypropylene sample digestion vessels with snap or screw caps or equivalent. **Five vessels of each lot of digestion vessels must be taken through analysis to check for mercury.**

VII. REAGENTS AND STANDARD PREPARATION

A. REAGENTS

1. Concentrated sulfuric acid suitable for Hg determination.

2. Concentrated nitric acid suitable for Hg determination.

3. Stannous chloride: in a 1000 mL volumetric flask add approximately 500 mL D.I. water, 30 mL concentrated HCl, add 11 grams stannous chloride crystals swirl to mix and dilute to 1000 mLs. Prepare fresh daily.

4. 3% HCl Carrier Solution: Dilute 30 mL of concentrated metals grade HCl to one liter. Prepare fresh daily.

5. Sodium chloride-hydroxylamine chloride solution: dissolve 120 grams of sodium chloride and 120 grams of hydroxylamine hydrochloride (very high grade --Do not get from Tennessee Reagents) in D.I. water and dilute to 1 liter. Note: this is normally made up 2 Liters at a time.

5. Potassium permanganate: 5% solution, w/v: dissolve 200 grams of potassium permanganate in 4000 mL of D.I. water. Should have "suitable for mercury determination" written on the side of the potassium permanganate bottle. This reagent takes overnight stirring (minimum of 3 hours if absolutely necessary). Use stirring bar already in the reagent bottle for this purpose. It is very easy to contaminate with mercury.

6. Potassium persulfate: 5% solution, w/v: dissolve 100 grams of potassium persulfate in 2000 mL D.I. water. Slight heating with stirring may be necessary to completely dissolve. The formation of crystals in this solution is not a problem.

B. STANDARDS

1. Traceability

a. A bound logbook 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 logbook 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, and expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in a bound logbook. 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 a logbook. Each analyst must be sure to "Z" out the unused area of each logbook page.

NOTE: All standard solutions should be prepared using class A volumetric flasks, class A volumetric pipettes (or calibrated Eppendorfs). All standards, blanks, and samples are taken through the digestion process.

a. Stock mercury solution: (100 µg/mL). Order from manufacturer already prepared. This solution is given a unique identifier.

b. Primary source and secondary source mercury standard solutions at 200 ug/L: dilute 2 mL of stock solution to 1000 mL in a 1000 mL volumetric flask, with 1.5 mL concentrated HNO_3.

c. Calibration standards

i. Prepared from the primary source working standard. The preparation of the calibration standards, etc. is described below.

a. Dilute the volumes below to 30 mLs in a 70 mL polypropylene vessel. (Note: The standards are diluted to 10 mLs for the initial step of the digestion. From that point when 25 mLs of DI water are added to samples, 15 mLs of DI water is added to the standards.

ug/L Hg	mLs of 200 ug/L standard in 30 mLs
0.20	0.03
0.50	0.075
1.0	0.15
2.0	0.30
4.0	0.60
6.0	0.90
10.0	1.5

iii. Appropriate reagents are added as below in the sample preparation section.

iv. Prepare one vessel for each.

v. It is necessary to digest the calibration standards.

e. Calibration verification standards

i. Initial calibration verification (ICV) solution – 4.0 ug/L

a. Prepared by diluting 0.6 mL of the second source standard to 30 mL with reagent water in a 70 mL polypropylene vessel. (TV = 4.0 ug/L)

b. Appropriate reagents are added as below in the sample preparation section.

c. It is necessary to digest the ICV standards for Method 7470A, Method 245.1 does not require digestion of standards.

ii. Continuing calibration verification (CCV) solution

a. Prepared from the primary source standard.

b. Prepared by diluting 0.3 mL of the primary standard at 200 ug/L to 30 mLs with reagent water in a 70 mL polypropylene vessel for 2.0 ug/L or 0.6 ml to 30 mls for 4.0 ug/L.

c. Appropriate reagents are added as below in the sample preparation section.

d. It is necessary to digest the CCV standards for Method 7470A, Method 245.1 does not require digestion of standards.

f. Digestion standards

i. Laboratory control sample

a. Prepared from the secondary source standard.

b. Prepared by diluting 0.3 mL of the second source standard to 30 mL with reagent water in a 70 mL polypropylene vessel.

c. Appropriate reagents are added as below in the sample preparation section.

d. This solution should be given a unique identifier in the digestion log.

ii.. Matrix Spikes

a. Prepared from the secondary source working standard.

b. Prepared by diluting 0.3 mL of the second source standard to 30 mL with sample in a 70 mL polypropylene vessel. Project specific or method specific requirements may over-ride the spiking level.

c. Appropriate reagents are added as below in the sample preparation section.

VIII. CALIBRATION

A. Set up the instrument with proper operating parameters.

1. Perkin Elmer Flow Injection Mercury System (FIMS).

i. Replace any old tubing that is around the pump cylinder. The sample transfer tubing connected to the seperator cover must not have any moisture in it. If it does replace it. **(Perkin-Elmer tygon**

tubing, waste and carrier 1.52mm I.D., waste only 3.17mm I.D., stannous chloride 1.14mm I.D.)

ii. Also replace the filter membrane with the rough side up. (for instructions refer to page 1-22 in maintenance manual.)

iii. Turn on PE 100 spectrophotmeter; (Note: this must be on in order to start up the software on the computer.)

iv. Turn on computer and go to icon "AA Win LAB Analyst".

v. Go to method; select "Hg CAL 2" then OK.

vi. Wavelength = 253.7; smoothing points =9; measurement = peak height; read time =18sec.; BCC time = 2 sec.

vii. Go to "Sample Info" and enter the order of the samples and other information that may be needed.

viii. Save entered sample list under "Save ….sample info file" Note: description and batch ID are normally the date of analysis.

ix. Go to "auto"; then to set-up. Select Browse in both spaces. One is to bring up your saved "Sample Information" File. The other is to select a results library. Double click on heading and choose.

x. Turn the printer on.

xi. Connect all tubing to the pump and blocks.

xii. Start the pump by going to "FIAS" and click the pump 1 Icon (120).

xiii. The pump will start, then lock down and tighten the tubes onto the pump.

xiv. Turn on the nitrogen tank, it should be above 500 psi on the guage. Replace the nitrogen tank when it is at 500 psi.

xv. The pressure gauge on the PE100 should be just below 100.

xvi. Use the tension adjuster to press down the tubing magazine to the pump head on the top and bottom. Start the pump and then lock them down. This technique needs to be demonstrated so that a new user will be able to understand what is needed here and how to do it.

xvii. Adjust the spring tension tubing until there is a constant "bubble of low rate" coming out to the waste tube.

xviii. Place carrier tubes into carrier and stannous chloride tube into SnCl2. (click valve fill inject and make sure flow is correct and the line is rinsed).

xix. Make sure the permanganate waste bottle is bubbling in order to absorb any Hg vapors which could be vented into the room.

xx. Allow a few minutes for reagents to flow through the system before starting analysis.

xxi. Calibrate: Go to "Auto" click on "Analyze", click on "calibrate".

xxii. "Select Location" enter #'s to be ran, and then press "OK". Samples are done in increments of 10 samples

B. Analyze the calibration standards as below.

1. New calibration points must be analyzed when the ICV analysis is not within ± 5%. **A curve must be analyzed daily for all projects especially USACE and CLP projects.**

2. The curve should be linear with a calculated intercept with a minimum correlation coefficient (r) of $\geq$0.995 (USACE) or 0.998 (other). If not, a new curve must be analyzed.

IX. PROCEDURE

A. Glassware preparation

1. After use, samples are nuetralized and disposed down an acid sink with running water and rinsed with tap water. Or the sample may be discarded into the Mercury waste drum.

2. Acid clean the glassware used for mercury prep as follows:

a. Rinse with low Hg content 1:1 HCl.

b. Rinse with D.I. water.

B. Label the vessels indicating which sample will be in each..

C. Prepare calibration standards as detailed above. Add all reagents to the standards which are added to the samples as outlined below. Record the standard preparation in the digestion log.

D. Sample preparation

1. Transfer 30 mL, or an aliquot diluted to 30 mL of sample to the 30 mL mark on a 50 mL digestion vessel previously marked for this sample.

   NOTE: Normally, **a**n automatic dilution of 10X to 100X is performed for all TCLP extracts. All TCLP samples get one matrix spike unless several come in at one time from the same client with the same matrix. Then one in ten of the same matrix get spiked. Check with your manager.

2. Add 1.5 mL of concentrated sulfuric acid to each vessel and mix.

3. Add 0.75 mL of concentrated nitric acid to each bottle and mix.

4. Add 4.5 mL potassium permanganate solution to each vessel and mix. For sewage samples additional permanganate may be required. Shake and add additional portions of potassium permanganate to the solution if necessary, until the purple color persists for at least 15 minutes (not more than 7.5 mL). If the purple color does not persist after the addition of 7.5 mL $KMnO_4$ the sample must be diluted prior to digestion. Inform your manager that the minimum detection limit cannot be reached for that particular matrix.

   NOTE: The same amount of $KMnO_4$ added to the samples should be present in the standards and blanks.

5. Add 2.4 mL of potassium persulfate to each vessel and mix. Cover.

6. Heat for 2 hours in the block digester at 95$\pm$2°C (the block temperature must be monitored and documented. Record observed temperature, correction factor, and the corrected temperature), cool.

6. Samples may be saved at this point if there is not time to run the whole set that day.

NOTE: Stannous Chloride (VII. A 5.) and 3% HCl (VII. A 8.) are added by the instrument during analysis.

E. Sample analysis

1. Set up the instrument as described in the calibration section above.

2. When ready to run samples, add 1.8 mL of sodium chloride-hydroxylamine chloride to reduce the excess permanganate. Sample analysis must be preceded by the analysis of an ICV with control limits of ±10% for SW846-7470 and ±5 % for 245.1. Followed by the ICB (< ±MDL for USACE or ±RL/CRDL for others and CLP).

3. Each set of ten samples and at the end of the analytical run must be followed by a CCV with control limits of ±20% for SW846-7470 and ±10% for 245.1

4. CCB must always follow the CCV. Control limits are (<±MDL for USACE or ±RL/CRDL for others and CLP). CCB must be run at the beginning and end of a sequence and after every 10 samples. **No analyte must be detected >2xMDL for DOD QSM Ver. 3.**

5. The autosampler log is set up to analyze 106 samples at a time.

Instrument Run Log example:

AS LOC	**Sample ID**
0	Wash
1	0.0
2	0.02
3	0.05
4	0.1
5	0.2
6	0.4
7	0.6
8	1.0
9	ICV
10	ICB
11	LCSW
AS LOC	**Sample ID**
12	PBW
13	Sample
14	Sample
15	Sample
16	Sample
17	Sample
18	Sample
19	Sample
20	Sample
21	CCV

22	CCB
23	Sample
24	Sample
25	Sample
26	Sample
27	Sample
28	Sample
29	Sample
30	Sample
31	MS
32	MSD
33	FCV
34	FCB

F. Data reporting

1. Reduce data to result which will be reported.

2. Complete the data review checklist (attached). Must be completed and attached to each set of USACE data.

X. CALCULATIONS

A. Apply a least squares fit to the calibration standards plotting µg Hg/L versus the absorbance. For the concentration of the standards, assume 30 mL of solution volume (the 0.1 µg Hg standard will be input as 1.0 µg Hg/L) (0.1µg Hg / 0.030 L solution).

B. Input the sample absorbance into the mercury spreadsheet making sure that you are using the correct spreadsheet for the matrix of the sample.

C. Also make sure that the appropriate dilution factor is inputted in the correct space on the spreadsheet.

D. Report the data as µg Hg/L of sample.

XI. QUALITY CONTROL (Reference SW-846, 7470A Update III, USEPA CLP ILMO 4.1 or 245.1, Rev 3.0, 5/94 for further clarification)

A. Daily

1. **The instrument must be calibrated daily for all projects.**

2. Begin each analysis with an ICV(QCS) second source. The control limits are ±10% and IPC(CCV) for 245.1, limits are ± 5% and subsequent analyses are ± 10%.

3. Analyze ICB. Control limits (<±MDL for USACE or ±RL/CRDL for others and CLP)., depending on method. **No analyte detected >2xMDL for DOD QSM Ver. 3.**

4. If the ICV(QCS) is not in control a new curve must be analyzed prior to sample analysis.

5. If the IPC(initial CCV) for 245.1 is not within the limits of ±5%, try preparing another undigested CCV and reanalyzing before recalibrating. If this fails then a recalibration is necessary.

6. Follow each set of 10 samples with a CCV and also must end up with a CCV after the last sample. The control limits are ±20% for SW846-7470 and ± 10% for 245.1.

7. A CCB must always follow a CCV, the control limit is (<±MDL for USACE or ±RL/CRDL for others and CLP). CCB must be run at the beginning and end of a sequence and after every 10 samples. **No analyte detected >2xMDL for DOD QSM Ver. 3.**

B. Quarterly or as needed when doing straight CLP work.

1. IDL's for CLP 4.1.

C. Digestion

1. LCS data should be maintained and available for easy reference or inspection.

2. Preparation blank (<1/2 ±RL or ±RL/CRDL for common contaminates (DOD) and ±RL/CRDL for others and CLP).

   a. Employ a minimum of one preparation blank per sample batch to determine if contamination or any memory effects are occurring. The preparation blank is taken through the same digestion/preparation steps as the samples being tested. The result for the preparation blank must be below the method detection limit. If not, the analyst must use good judgment to evaluate the impact upon the associated samples. There is no impact if an associated sample is below the method detection limit nor if the level in the sample is greater than 10X the level found in the

preparation blank. If the level of mercury in a sample is above the method detection limit but less than 10X the level found in the preparation blank, the sample must be redigested and reanalyzed or the data must be qualified on the final report. The project manager or QA manager will make this determination.

3. Laboratory control sample (LCS)

   a. Employ a minimum of one laboratory control sample (LCS) per sample batch to verify the digestion procedure. The LCS is taken through the same digestion/preparation steps as the samples being tested. The minimum control limits are ±20% for SW846-7470 and ±15% for 245.1. If the LCS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be either redigested or the data should be qualified. The project manager or QA Officer will make this determination.

D. Sample matrix

1. Analyze one replicate sample for every twenty samples. 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. CLP does not allow this. Project specific requirements will take precedence in these situations.

2. Analyze one spiked sample and spiked sample duplicate for every twenty samples. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. Project specific requirements will take precedence in these situations. CLP requires 1 duplicate and 1 spike per batch. If the analyte level in the sample is not greater than four times the spiking level, the spike recoveries should be within ±25% of the true value (**± 20% for DOD projects**). If not, check with supervisor to determine appropriate action. The final analytical report must document this situation.

   NOTE: For TCLP extracts, a matrix spike must be performed for each different matrix. The method of standard additions must be used if the sample spike recovery is not at least 50% and the concentration of Hg does not exceed the regulatory level and if the concentration of Hg measured in the extract is within 20% of the regulatory level.

3. The relative percent difference (RPD) between replicate determinations is to be calculated as referenced in the laboratory QA manual. (A control limit of ± 20% RPD shall be used for sample values greater than ten times

the instrument detection limit.) Supervisor must be notified if the control limit is not met. Supervisor will determine corrective action if required. The final analytical report must document this situation.

4. For 245.1 analyze one serial dilution (1 to 5 dilution) for every 20 samples or per analytical batch, whichever is more frequent. Percent recovery should be ±10%. The concentration of the original sample should be a minimum of 50X the IDL in order to apply the recovery criterion; if not, the serial dilution approach is not used.

5. When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6 of SW846-7000A provides tests to evaluate the need for using the MSA.

E. Method Detection Limit (MDL), Empirical Laboratories' Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength:

TABLE I

Aqueous Method Detection Limits(MDL), Empirical Laboratories' Reporting Limits(ERL), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)				
Mercury by EPA 245.1, 7470A, SOW 4.1 & 5.2	**AQUEOUS MDL(ug/L)**	**AQUEOUS ERL(ug/L)**	**AQUEOUS CRQL ILMO 4.1 (ug/L)**	**AQUEOUS CRQL ILMO 5.2 (ug/L)**
Mercury	0.08	0.20	0.2	0.2

TABLE 2

ANALYTE	**WAVELENGTH**
Mercury	**253.7**

XII. CORRECTIVE ACTIONS

A. INSTRUMENT RELATED

1. ICV(**QCS for 245.1**)- second source not within ± 10%.
   a. If the problem is with the solution.
      i. Reprepare, obtain new stock if necessary.
   b. If the problem is with the calibration.
      i. Recalibrate through analysis of appropriate standards and recheck ICV.
2. CCV not within ± 20% for SW846 and ±10% for (**245.1, ± 5% for initial IPC and + 10% for subsequent IPCs**)
   a. If the problem is with the solution.
      i. Reprepare, obtain new stock if necessary.
   b. If the problem is with the calibration.
      i. Recalibrate through analysis of appropriate standards and reprepare/reanalyze the previous ten sample according the following guidelines.
         a. If the CCV was biased high, any of the previous ten samples which were below the detection limit do not require reanalysis.
         b. If the CCV was biased low, the previous ten samples must be reanalyzed.

B. DIGESTION RELATED

1. The preparation blank less than <1/2 RL or ±RL/CRDL for common contaminates (DOD) and ±RL/CRDL for others and CLP.

   a. If the problem is with the instrument or stannous chloride.
      i. Analyze a reagent blank to determine the stannous chloride and the instrument are behaving properly. If this check has detectable mercury, reprepare the stannous chloride or determine if there are any problems with the instrument. Contact supervisor immediately.

   b. If the problem is with the digestion.

i. All associated samples which are below the RL, CRDL or have a level of mercury greater than 5X the level found in the preparation blank can be reported. If the level of mercury in an associated sample is not BMDL nor greater than 5X the level found in the preparation blank, the sample must be redigested/reanalyzed or reported as qualified. The project manager or QA manager will make this determination.

2. LCS not within control limits (or ±20%, ± 15% for **245.1**).
   a. If the problem is with the instrument.
      i. Reanalyze when instrument is in control if further sample bottles are available.
   b. Is the problem is with the digestion.
      i. If biased low, associated samples must be redigested.
      ii. If biased high, the impact upon the data user must be evaluated. The samples will be redigested or the data will be qualified on the final report.

C. SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within ±20%
   i. The associated sample data must be qualified on the final report.

2. Spike analysis recovery not within ±25%(**± 20% for DOD projects**)
   i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.
   ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. TCLP extracts must be evaluated as in section XI.D.2 above. The associated sample data must be qualified on the final report.

3. When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6 of SW846-7000A provides tests to evaluate the need for using the MSA.

XIII. WASTE DISPOSAL and POLLUTION PREVENTION

Please see Waste Disposal SOP-405, for instruction of proper 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.

XIV. REFERENCES

1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III)*; Method 7470A

2. USEPA *Code of Federal Requlations, 40, CH 1,PT 136;* Method 245.1; APX-B

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

XV. DEFINITIONS

1. Refer to SOP-431 for common definitions.

ADDENDUM FOR USEPA SOW ILM05.2

1. The CCV concentration must be different from the ICV.

2. The same CCV shall be used throughout analysis for an SDG.

3. Calibration standards must be within 5% of the standard concentration.

4. A CRA must be analyzed after the ICV/ICB and after each batch of 20 samples, but before the final CCV/CCB. The control limit is ±30%.

5. Spike samples at 1 ug/L for water.

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):	
Batch Number(s):	
Method:	**7470A (Mercury)**

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 LCS or blank spike meet control limits?				
6. Did MS/MSD meet control limits?				
7. Was the preparation (Method) Blank below the project required detection limits?				
8. Did you return samples back to cold storage immediately after use?				
9. Was water bath 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: _____

Second-Level Review: _____ Date: _____

MERCURY ANALYSIS IN SOIL/SEDIMENT BY MANUAL COLD VAPOR TECHNIQUE

METHODS SW846 7471A 7471B, EPA 245.5 and CLPILM 04.1 (NJDEP does not accept CLPILM 04.1 after June, 2003), Addendum for USEPA CLP ILM 05.2

SOP NUMBER: **SOP-104**

REVISION NUMBER: **17**

APPROVED BY:

SECTION MANAGER

TECHNICAL DIRECTOR

EFFECTIVE DATE: **01/29/09**

DATE OF LAST REVIEW **01/29/09**

MERCURY ANALYSIS IN SOIL/SEDIMENT BY MANUAL COLD VAPOR TECHNIQUE

References:
SW846 Method 7471A, 7471B
USEPA Method 245.5, CLP SOW ILM04.1
See Addendum for CLP SOW ILM05.2

I. SCOPE AND APPLICATION:

A This procedure measures total mercury (organic and inorganic) in soils, sediments, bottom deposits and sludge type materials.

B. The range of the method is 0.2 to 2 µg/g. The range may be extended above or below the normal range by increasing or decreasing sample size or through instrument and recorder control.

II. SUMMARY OF METHOD:

A. A weighed portion of the sample is acid digested for 2 minutes at 95±2°C, followed by oxidation with potassium permanganate and with a secondary digestion at 95°C for 30 minutes. Mercury in the digested sample is then measured by the conventional cold vapor technique.

III. SAMPLE HANDLING AND PRESERVATION:

A. Because of the extreme sensitivity of the analytical procedure and the omnipresence of mercury, care must be taken to avoid extraneous contamination. Sampling devices and sample containers should be ascertained to be free of mercury; the sample should not be exposed to any condition in the lab that may result in contact with solid, liquid or airborne mercury.

B. Refrigerate solid samples at 4°C (±2°C) upon receipt until digestion and analysis.

C. The sample should be analyzed without drying. A separate percent solids determination is required

D. The holding time for digestion of mercury samples is 28 days.

IV. INTERFERENCES:

A. Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/Kg of sulfide, as sodium sulfide, do not interfere with the recovery of added inorganic mercury in reagent water.

B. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/Kg had no effect on recovery of mercury from spiked samples.

C. Samples high in chlorides require additional permanganate (as much as 12.5 mLs) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell.

D. Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

V. SAFETY

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

B. The toxicity and carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices. Normal accepted laboratory safety practices should be followed during reagent preparation and instrument operation. Always wear safety glasses or full-face shield for eye protection when working with these reagents. Each laboratory is responsible for maintaining a current safety plan, a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

C. Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Analyses should be conducted in a laboratory exhaust hood. The analyst should use chemical resistant gloves when handling concentrated mercury standards.

VI. EQUIPMENT/APPARATUS:

A. Perkin Elmer Flow Injection Mercury System (FIMS)

B. Perkin Elmer AS 90

C. Mercury lamp

D. Environmental Express Mod-Block digestion block capable of holding 95±2°C for 2 hours

E. A scale or balance capable of weighing to 0.01 ± 0.02 gram.

F. Snap cap digestion polypropylene vessels for use with the mod block digester. **Five vessels of each lot must be taken through analysis to check for mercury**

G. Polypropylene watch glasses suitable for use with the above vessels in F above.

H. Manual Sample Mill

I. Wiley Sample Mill

J. Clippers for cutting vegetation

VII. REAGENTS AND STANDARD PREPARATION:

A. REAGENTS

1. Reagent Water: Reagent water will be interference free. All references to water in this method refer to reagent water unless otherwise specified.

2. Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3. Both HNO_3 and HCl must be of the reagent grade suitable for mercury determinations.

   NOTE: This reagent is required for use when USACE project samples are being digested.

3. Concentrated HCl.

4. Concentrated HNO_3.

5. Stannous chloride in a one liter volumetric flask add ~500 mL D.I. H_2O, 30 mL concentrated HCl, and 11g stannous chloride crystals. Swirl to mix and dilute to 1 L.

6. Sodium chloride-hydroxylamine chloride solution: Dissolve 120 g of sodium chloride and 120 g of hydroxylamine sulfate in reagent water and dilute to 1 L. Note : this is normally made up 2 liters at a time.

7. Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 200 g of potassium permanganate in 4 L of reagent water.

8. 3 % HCl carrier solution: 30 mL HCl – 1 L DI H20; Prepare fresh daily.

9. Potassium persulfate 5% solution: Dissolve 100g in 2 liters of D.I. water. Used with digestion of CLP soils.

B. STANDARDS

1. Traceability

a. A bound logbook 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 logbook 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. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in a bound logbook. 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 a logbook. Each analyst must be sure to "Z" out the unused area of each logbook page.

2. Preparation

NOTE: All standard solutions should be prepared using class A volumetric flasks, class A volumetric pipettes (or calibrated Eppendorfs). All Standards, blanks, and samples are taken through the digestion process.

a. Stock mercury solution: (100 µg/mL). Order from manufacturer already prepared. This solution is given a unique identifier.

b. Primary source and secondary source mercury standard solutions: dilute 2 mL of stock solution to 1000 mL in a 1000 mL volumetric flask, with 1.5 mL concentrated HNO_3 (200 ug/L).

c. Calibration standards

   i. Prepared from the primary source standard. The preparation of the calibration standards, etc. is described below.

      a. Dilute the volumes below to 5 mLs in a 70 mL polypropylene vessel. (Note: The standards are diluted to 5 mLs for the initial step of the digestion.)

ug/L Hg	mLs of 200 ug/L standard in 50 mL
0.20	0.050
0.50	0.125
1.0	0.25
2.0	0.50
4.0	1.0
6.0	1.5
10.0	2.5

   ii. Appropriate reagents are added as below in the sample preparation section.

   iii. Prepare one vessel of each.

   iv. It is necessary to digest the calibration standards when following all mercury methods.

e. Calibration verification standards

   i. Initial calibration verification (ICV) solution – 4.0 ug/L

      a. Prepared from the secondary source mercury standard (200 ug/L).

      b. Prepared by diluting 1.0 mL of the second source mercury standard to 5 mLs in a polypropylene digestion vessel.

      c. Appropriate reagents are added as below in the sample preparation section.

d. It is necessary to digest the ICV standards when using all mercury methods for soil.

ii. Continuing calibration verification (CCV) solution

a. Prepared from the primary or secondary source mercury standard. The concentration is alternated from 2.0 ug/L to 4.0 ug/L every 20 samples.

b. Prepared by diluting 0.50 for a 2.0 ug/L and 1.0 mL for a 4.0 ug/L of the secondary 200 ug/L standard to 5.0 mL with reagent water in a polypropylene digestion vessel.

c. Appropriate reagents are added as below in the sample preparation section.

d. It is necessary to digest the CCV standards when following all mercury methods for soil.

f. Digestion standards

1. Laboratory control sample

a. The Laboratory Control Sample (LCS) is prepared from the secondary source mercury standard (200 ug/L) and added to ~ 0.3 grams of teflon chips.

b. Prepared by diluting 0.50 mL of the secondary mercury standard (200 ug/L) to 5 mLs in a polypropylene digestion vessel with 0.30 grams of teflon chips.

c. Appropriate reagents are added as below in the sample preparation section.

d. This solution is given a unique identifier in the digestion log.

2. Matrix Spikes

a. Prepared from the primary or secondary source mercury standard (200 ug/L).

b. Prepared by adding 0.50 mL of the mercury standard (200 ug/L) to the sample in a polypropylene digestion vessel. Project specific requirements may over-ride the spiking level.

c. Appropriate reagents are added as below in the sample preparation section.

VIII. CALIBRATION:

A. Set up the instrument with proper operating parameters.

1. Perkin Elmer Flow Injection Mercury System (FIMS)

a. Prepare the instrument for calibration by the following steps:

i. Replace any old tubing that is around the pump cylinder. The sample transfer tubing connected to the seperator cover must not have any moisture in it. If it does replace it. **(Perkin-Elmer tygon tubing, waste and carrier 1.52mm I.D.,waste only 3.17mm I.D., stannous chloride 1.14mm I.D.)**

ii. Also replace the filter membrane with the rough side up. (for instructions refer to page 1-22 in maintenance manual.)

iii. Turn on PE 100 spectrophotometer; (Note: this must be on in order to start up the software on the computer.)

iv. Turn on computer and go to icon "AA Win LAB Analyst"

v. Go to method; select "Hg CAL 2" then OK.

vi. Wavelength = 253.7; smoothing points =9; measurement = peak height; read time = 18 sec.; BCC time = 2 sec.

vii. Go to "Sample Info" and enter the order of the samples and other information that may be needed.

viii. Save entered sample list under "Save …sample info file" Note: description and batch ID are normally the date of analysis.

ix. Go to "auto"; then to set-up. Select Browse in both spaces. One is to bring up your saved "Sample Information." File. The other is to select a results library. Double click on heading and choose.

x. Turn the printer on.

xi. Connect all tubing to the pump and blocks.

xii. Start the pump by going to "FIAS" and click the pump 1 Icon (120).

xiii. The pump witll start, then lock down and tighten the tubes onto the pump.

xiv. Turn on the nitrogen tank, it should be >500 psi on the guage. Replace the nitrogen tank when it is at 500 psi.

xv. The pressure gauge on the PE100 should be just below 100.

xvi. Use the tension adjuster to press down the tubing magazine to the pump head on the top and bottom. Start the pump and then lock them down. This technique needs to be demonstrated so

that a new user will be able to understand what is needed here and how to do it.

xvii. Adjust the spring tension tubing until there is a constant "bubble of low rate" coming out to the waste tube.

xviii. Place carrier tubes into carrier and stannous chloride tube into SnCl2. (click valve fill inject and make sure flow is correct and the line is rinsed)

xix. Make sure the permanganate waste bottle is bubbling in order to absorb any Hg vapors which could be vented into the room.

xx. Allow a few minutes for reagents to flow through the system before starting analysis.

xxi. Calibrate: Go to "Auto" click on "Analyze", click on "calibrate".

xxii. "Select location" enter the #'s of the samples to be analyzed, then "OK".

B. Analyze the calibration standards as below.

1. A curve must be analyzed daily for all projects. A new curve must be analyzed when the ICV analysis is not within ±10% for SW846 7471A and 245.5 methods, or ±20% for 7471B.

2. The curve should be linear with a calculated intercept with a minimum correlation coefficient(r) of ≥0.995 (USACE) or 0.998 (other). If not, a new curve must be analyzed.

3. **CLP requires a blank + 5 calibration standards (0, .02, .05, .1, .5 and 1.0 µg). (One standard must be at CRDL or IDL whichever is greater.)**

IX. PROCEDURE:

A. Prepare calibration standards as detailed above. Add all reagents to the standards which are added to the samples as outlined below. Record the standard preparation in the standard log.

B. 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 must 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. Transfer 0.30 g (for USACE work use anywhere from 0.20 to 1.0 g and record the weight in the digestion log) of sample to a polypropylene digestion vessel previously marked for this sample. Record the exact sample mass on the bottle and in the digestion log. (Note: the balance must be calibrated for the specific task. Calibrate by weighing a 0.5 and a 0.1g weight on the balance along with a digestion vessel. Record in specific balance calibration log.)

2. Add 2.5 mL of reagent water, and 2.5 mL of aqua regia and mix for samples. Add 2.5 mL of aqua regia to standards and mix.

3. Cover samples and standards with watch glasses and heat for 2 minutes in the hot block at 95$\pm$2°C (The hot block temperature must be monitored and documented. Record observed temperature, correction factor, and the corrected temperature).

4. Cool, bring to 30 ml with D.I. water

5. Add 7.5 mL potassium permanganate solution to each vessel and mix. For sewage samples additional permanganate may be required. Shake and add additional portions of potassium permanganate to the solution if necessary, until the purple color persists for at least 15 minutes (not more than 12.5 mL).

NOTE: The same amount of $KMnO_4$ added to the samples should be present in the standards and blanks.

6. Heat for 30 minutes on the hot block at 95±2°C (The temperature must be monitored and documented. Record observed temperature, correction factor, and the corrected temperature), cool. Samples may be saved at this point if there is not time to run the whole set that day.

7. Add 3 mLs of sodium chloride-hydroxylamine chloride solution to each vessel.

8. Bring to 50 ml with D.I. water both standards and samples. Cap mix and vent to decolor and release Cl gas. The samples are now ready for analysis.

NOTE: Stannous Chloride (VII. A 5.) and 3% HCl (VII. A 8.) are added by the instrument during analysis.

C. Sample analysis

1. Set up the instrument as described in the calibration section above.

2. When ready to run samples, transfer samples and standards to autosampler tubes and load the auto sampler according to the sample information sheet set up previously. If chlorides are suspected, purge the head space in the polyethylene tube for at least 1 minute to get rid of any chlorine gas collected there. After a delay of at least 30 seconds the sample is ready for step "3". **NOTE:** When aqua-regia is added assume that all samples and standards have chlorine and treat accordingly. Purging the samples of chlorine is accomplished by putting a pasteur pipette on the end of some air tubing hooked to a fish pump. The pasteur pipette is then placed at an angle into the top of the polyethylene vessel without breaking the surface of the sample. It takes about one minute to purge the air above the sample of chlorine.

3. Analysis must be preceded by the analysis of an ICV (concentration at or near mid range) with control limits of ±10% for SW846-7471A or ±20% for 7471B and ±5% for 245.5 methods.

4. The ICB must follow the calibration standards (<±MDL (USACE) or ±RL/CRDL for other or CLP), but not before the ICV. **No analyte must be detected > 2xMDL for DOD QSM Ver. 3.**

5. Each set of ten samples must be followed by a CCV with control limits of ±20% for SW846-7471A and B and ±10% for 245.5 method. The run must also end with a CCV, then CCB.

6. Analyze CCB after calibration and each CCV. The CCB frequency is 10% or every 2 hours whichever is more frequent. (control limit is < ±MDL or ±RL/CRDL for other or CLP). **For DOD QSM Ver.3 CCB at beginning and end of sequence and after every 10 samples. No analyte detected > 2xMDL.**

7. Instrument Run Log example:

AS LOC	Sample ID
0	Wash
1	0.0
2	0.O2
3	0.05
4	0.1
AS LOC	**Sample ID**
5	0.2
6	0.4
7	0.6
8	1.0
9	ICV
10	ICB
11	LCSW
12	PBW
13	Sample
14	Sample
15	Sample
16	Sample
17	Sample
18	Sample
19	Sample
20	Sample
21	CCV
22	CCB
23	Sample
24	Sample
25	Sample
26	Sample
27	Sample
28	Sample
29	Sample
30	Sample
31	MS
32	MSD
33	FCV
34	FCB

8. Sample analysis:

   i. Go to "Analyze", "select location" and type in the range of numbers needed to complete analysis. (ie. 9-54). Press enter and the autosampler will proceed to enter the selected range. NOTE: Check standards are loaded as part of the tray.

   ii. Make sure that the sample wash beaker is filled with 3% HCl.

   iii. Dilute and reanalyze samples that are more concentrated than within 10% of the high standard. Soil sample dilutions are made from the digested aliquot. Sample concentration results that are below the calibration curve but above the MDL are reported flagged as estimated, ("B" flag).

D. Data reporting

1. Reduce data to result which will be reported using the soil spreadsheet found on the network..

2. Complete the data review checklist (attached). Must be completed and attached to each set of USACE data.

X. CALCULATIONS:

A. Pull up the blank spreadsheet at K:\wcm\tests\mercury and fill in all the information pertinent to the current analysis. Save as the date of analysis. This information can be obtained from your mercury digestion log.

B. Input the sample absorbance into the excel spreadsheet in the appropriate cell. The spreadsheet uses the current calibration to calculate the Hg results.

C. Make sure that the appropriate dilution factors are entered into the spreadsheet in the correct cells.

D. The spreadsheet should divide the result which is the µg Hg obtained from the sample mass by the sample mass in grams. This will yield a result of µg Hg/g sample on a wet weight basis. Calculations in the spreadsheet should be checked occassionally to make sure that they are working correctly.

E. If available, divide the result by the %solids to obtain the result on a dry weight basis.

F. Report the data as µg Hg/g of sample (mg/kg wet or mg/kg dry when % solids are available).

XI. QUALITY CONTROL (Reference SW-846, 7471A Update III, 7471B Revision 2 February 2007, USEPA CLP ILMO 4.1 or EPA 245.5 for further clarification)

A. Daily

1. **The instrument must be calibrated daily for all projects.**

2. Begin each analysis with an ICV. The control limits are ±10% for 7471A and 245.5, ±20% for 7471B.

3. Analyze ICB. Control limit is <± **MDL** or ±RL/CRDL for other or CLP. **For DOD QSM Ver. 3, no analyte detected > 2x MDL.**

4. If the ICV is not in control a new curve must be analyzed prior to sample analysis.

5. Follow each set of 10 samples with a CCV and also must end up with CCV after last sample. The control limits are ±20% for SW846-7471A, SW846 7471B and for 245.5. If an exceedance occurs, analyze another CCV, if the second CCV fails, then a new calibration curve should be generated and all affected samples should be reanalyzed.

6. Follow each CCV with a CCB. Control limit is **< ±MDL** or ±RL/CRDL for others or CLP. **For DOD QSM Ver. 3, no analyte detected > 2x MDL.**

B. Quarterly

1. IDLs for CLP (Follow SOP - 414).

C. Annually

1. MDLs must be analyzed for all matrixes (Follow SOP - 414).

D. Digestion

1. LCS data should be maintained and available for easy reference or inspection.

2. Preparation blank (< ±½ RL or ±RL for common contaminates or ±RL/CRDL for others or CLP)

a. Employ a minimum of one preparation blank per sample batch to determine if contamination or any memory effects are occurring. The preparation blank is taken through the same digestion/preparation steps as the samples being tested. The result for the preparation blank must be < ± ½ RL for USACE or ±RL/CRDL for others or CLP. If not, the analyst must use good judgment to evaluate the impact upon the associated samples. There is no impact if an associated sample is below the method detection limit or if the level in the sample is greater than 10X the level found in the preparation blank. If the level of mercury in a sample is above the method detection limit, but less than 10X the level found in the preparation blank, the sample must be redigested and reanalyzed or the data must be qualified on the final report. The project manager or QA officer will make this determination.

3. Laboratory control sample (LCS).

a. Employ a minimum of one LCS per sample batch to verify the digestion procedure.. The LCS is taken through the same digestion/preparation steps as the samples being tested. The minimum control limits are ±20% for SW846-7471A, 7471B and 245.5 solid samples. An LCS will accompany each batch of soil samples. If the LCS is not in control, the Inorganic Manager and QA Officer must be notified immediately. Several possibilities exist at this point and a thorough investigation and data evaluation is essential. The first question is to evaluate the impact upon the data. All samples may need to be retested or flagged with the appropriate qualifier. The next question is to find out why it occurred and to proceed with a corrective action plan to prevent reoccurrence. This corrective action is documented in a CAR.

E. Sample matrix

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.

2. Analyze one spiked sample and spiked sample duplicate 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. Project specific requirements will take precedence in these situations. CLP requires 1 duplicate and 1 spike per

batch. If the analyte level in the sample is not greater than four times the spiking level, the spike recoveries should be within ±25% for 7471A and ±20% for 7471B of the true value(**±20% for DOD projects**). If results do not fall within the control limit-redigestion/reanalysis may be required. If reanalysis is not required, the associated batch of samples will be flagged accordingly. Discuss the situation with your supervisor. A Corrective Action Report (CAR) must be filled out and attached to the data as well as emailed or sent to the supervisor when the control limits are exceeded.

3. The relative percent difference (RPD) between replicate determinations is to be calculated as referenced in the laboratory QA manual. (A control limit of ± 20% RPD (non-aqueous samples may routinely exceed this amount) shall be used for sample values greater than ten times the instrument detection limit.) Supervisor must be notified if the control limit is not met. Supervisor will determine corrective action if required. The final analytical report must document this situation. A Corrective Action Report (CAR) must be filled out and attached to the data as well as emailed or sent to the supervisor when the control limits are exceeded.

4. For 245.5 analyze one serial dilution (1 to 5 dilution) for every 20 samples or per analytical batch, whichever is more frequent. Percent recovery should be 10%. The concentration of the original sample should be a minimum of 50X the IDL in order to apply the recovery criterion; if not, the serial dilution approach is not used.

5. When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6 of SW846-7000A provides tests to evaluate the need for using the MSA.

F. Method Detection Limit (MDL), Empirical Laboratories' Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength:

TABLE I

Solid/Soil Method Detection Limits(MDL), Empirical Laboratories' Reporting Limits(ERL), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)				
Mercury by EPA 245.1, 245.5, 7471A, SOW 4.1 & 5.2	**SOLID/SOIL MDL (mg/Kg)**	**SOLID/SOIL ERL (mg/Kg)**	**SOLID/SOIL CRQL ILMO 4.1 (mg/Kg)**	**SOLID/SOIL CRQL ILMO** 5.2 (mg/Kg)

Mercury	0.0157	0.033	0.1	0.1

TABLE 2

ANALYTE	WAVELENGTH
Mercury	**253.7**

XII. CORRECTIVE ACTIONS

A. INSTRUMENT RELATED

1. ICV not within ± 10% (SW846) and (245.5)
   a. If the problem is with the solution.
      i. Reprepare, obtain new stock if necessary.
   b. If the problem is with the calibration.
      i. Recalibrate thru analysis of appropriate standards and recheck ICV.

2. CCV not within ± 20% (SW846) and (245.5)
   a. If the problem is with the solution.
      i. Reprepare, obtain new stock if necessary.
   b. If the problem is with the calibration.
      i. Recalibrate thru analysis of appropriate standards and reprepare/reanalyze the previous ten sample according the following guidelines.
         a. If the CCV was biased high, any of the previous ten samples which were below the minimum detection limit do not require reanalysis.
         b. If the CCV was biased low, the previous ten samples must be reanalysed.

B. DIGESTION RELATED

1. The preparation blank less than ± ½ RL for DOD or ±RL/CRDL for others or CLP.
   a. If the problem is with the instrument or stannous chloride.
      i. Analyze a reagent blank to determine the stannous chloride and the instrument are behaving properly. If this check has detectable mercury, reprepare the stannous chloride or determine if there are any problems with the instrument.
      ii. If the problem was with the instrument or the stannous chloride and the situation is corrected continue analysis with a second aliquot of the preparation blank.
   b. If the problem is with the digestion.

i. All associated samples which are below the method detection limit (MDL) or have a level of mercury greater than 10X the level found in the preparation blank can be reported. If the level of mercury in an associated sample is not <MDL nor greater than 10X the level found in the preparation blank, the sample must be redigested/reanalyzed or reported as qualified. The project manager or QA manager will make this determination.

2. LCS not within control limits.
   a. If the problem is with the instrument.
      i. Reanalyze when instrument is in control with another aliquot of the sample.
   b. If the problem is with the digestion.
      i. If biased low, associated samples must be redigested.
      ii. If biased high, the impact upon the data user must be evaluated. The samples will be redigested or the data will be qualified on the final report.

C. SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within ±20%
   i. The associated sample data must be qualified on the final report.
2. Spike analysis recovery not within ±25% 7471A and ±20% 7471B (**±20% for DOD projects**)
   i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.
   ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. A corrective action report must accompany the data and be emailed or given to the superviser.

XIII. WASTE DISPOSAL and POLLUTION PREVENTION

Please see Waste Disposal SOP-405, for instruction of proper 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.

XIV. REFERENCES

1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III)*; Method 7471A

2. USEPA *Code of Federal Requlations, 40, CH 1,PT 136;* Method 245.1; APX-B

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

XV. DEFINITIONS

1. Refer to SOP-431 for common definitions.

Addendum for USEPA CLP ILM 05.2

1. CCV concentration must be different from ICV.

2. The same CCV shall be used throughout analysis for a sample delivery group.

3. Calibration standards must be within 5% of the standard concentration.

4. 0.2 grams of sample must be used for the sample aliquot, add enough reagent water to each sample to make a total volume of 10 mL. Proceed with method as in the water method SOP 103.0 Revision 9.

5. The ICV and CCV must be at ±20% recovery.

6. A CRA must be analyzed at the beginning and end of each batch of 20 samples. Right after the ICV/ICB and right before the final CCV/CCB. The control limit is ±30%.

7. The matrix spike must be analyzed at the concentration of 0.5 mg/Kg.

Sample Number(s):
Batch Number(s):
Method: 7471A (Mercury)

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 LCS or blank spike meet control limits?				
6. Did MS/MSD meet control limits?				
7. Was the preparation (Method) Blank below the project required detection limits?				
8. Did you return samples back to cold storage immediately after use?				
9. Was water bath 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.				

ANALYST DATA REVIEW CHECKLIST
7471A (Mercury)

Comments on any "No" response:

_____
_____
_____
_____
_____
_____

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

METALS ANALYSIS

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

METHODS 200.7, (SW846) 6010B, (SM 19th Edition 2340B) Hardness Calculation, (USEPA CLP) ILMO 4.1 (NJDEP does not accept CLPILM 04.1 after June, 2003)

Addendum for USEPA CLPILM 05.2

SOP NUMBER: **SOP-105**

REVISION NUMBER: **14**

APPROVED BY:

SECTION MANAGER

TECHNICAL DIRECTOR

EFFECTIVE DATE: **09/09/08**

DATE OF LAST REVIEW: **09/09/08**

ICP METHOD
SOP
References: SW-846, Method 6010B, December 1996
USEPA, Method 200.7, June 1991, Standard Methods 19th Edition 2340B, 1995
USEPA CLP, ILM 04.1. See Addendum for USEPA CLPILM 05.2

I. SCOPE AND APPLICATION

A. Inductively Coupled Argon Plasma (ICP) determines trace elements in solution. **We use the ICP to determine the concentration of the following metals: Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Sr, Ti, Tl, V and Zn.** All matrices, including ground water, aqueous samples, TCLP, SPLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis.

B. **Detection limits, sensitivity, and optimum ranges of the metals may be found in the ICP method file.** Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

II. SUMMARY OF METHOD

A. 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.

B. 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 resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by photomultiplier tubes. 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.

C. ICP's 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.

D. It is standard procedure to use an internal standard (Yttrium) with samples to increase the stability of the instrument as recommended by the manufacturer (TJA). (When samples are suspected of containing Yttrium internal standard cannot be used.)

III. SAMPLE HANDLING AND PRESERVATION

A. Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples which have been prefiltered 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).

B. 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. Since the most concentrated acid matrix allowed for direct disposal down an acid sink is a ratio of 20:1, the samples must be diluted with 1 part water to 2 parts sample prior to pouring down the sink while the tap water is running.

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

- 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 HNO_3 prior to metal determination.

- 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.

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- Method 3031 prepares oily waste samples for total metal determination by ICP. The samples are mixed with permanganate, H_2SO_4 , HNO_3, and HCl, then filtered. The filter is then added back to the filtrate and heated with more HNO_3 and filtered once again. If manganese is needed a manganese removal step is necessary.

- Method 3050B prepares waste 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.

IV. INTERFERENCES

A. 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.

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 (interelement 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.

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 interelement 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 intereference effects from all method anlytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using anlyte concentrations that will adequtetely describe the interfernce. 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 concentration near the upper analytical range limit.

3. Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for interelement contributions. Instruments that use equations for interelement 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 interelement 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.

4. When using interelement 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 As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. According to Table 2 from the method, 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximatley 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.

5. Interelement 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. Interelement corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an intefering emission line may appear should be avoided when practical. Interelement 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.

6. The interference effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and agron flow rate). When using the recommended wavelengths, the anlaylst 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 of matrix. The analyst is encouraged to utilize a computer routine for automatic correction on all analyses.

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. Adquate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.

8. When interelement corrections are applied, their accuracy should be verified, daily, by analyzing spectral interference check solutions. 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 ± one reporting limit from zero, daily verification is not required. All interelement 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 e

inspected to ensure that there is no contamination that may be perceived as a spectral interference.

B. 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 is 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.

C. 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 within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range. The aspiration time for this sample should be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of two of the method detection limit should be noted. Until the required rinse time is established, this method suggests a rinse period of at least 60 seconds between samples and standards. If a memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Alternate rinse times may be established by the analyst based upon their DQOs.

D. Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration accordingly. Concentrations should be within 20% of the true spiked concentration or dilution of the samples will be necessary. In the absence of measurable analyte, overcorrection could go undetected if a negative value is reported as zero.

V. SAFETY

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

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.
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.
3. MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves in the Quality Assurance Officers office.

VI. EQUIPMENT/APPARATUS

A. Inductively coupled argon plasma emission spectrometer: Thermo Jarrell Ash (TJA) 61E trace or equivalent.

B. Computer-controlled emission spectrometer with background correction: Thermo Jarrell Ashe (TJA) 61E trace or equivalent.

C. Radio frequency generator compliant with FCC regulations: TJA 61E trace or equivalent.

D. Argon gas supply – Liquid Argon

E. Class A volumetric flasks

F. Class A volumetric pipettes

G. Analytical balance - capable of accurate measurement to a minimum of three significant figures (.001gm): Mettler model AE100

H. Variable Eppendorf Pipettes 1000µL; 5000µL

VII. REAGENTS AND STANDARD PREPARATION

A. Notes

1. Reagent Water. All references to water in the method refer to reagent grade water unless otherwise specified. Reagent water will be interference free.

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.

B. REAGENTS

1. Hydrochloric acid (concentrated), HCl.
2. Nitric acid (concentrated), HNO_3.

C. STANDARDS

1. Matrix

a. All standards contain 2% HNO_3 and 5% HCl.

2. Storage

a. The standards are stored at room temperature in 500 mL Teflon bottles.

3. Traceability

a. A bound logbook 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 logbook 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 logbook where information is recorded. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in a bound logbook. 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 a logbook. Each analyst must be sure to "Z" out the unused area of each logbook page.

4. Calibration standards

a. All standards have an acid matrix of 2% HNO_3 and 5% HCl and should be prepared using class A volumetric flasks, class A volumetric pipettes (or calibrated Eppendorfs).

b. STD-1 is the calibration blank: Reagent grade water **matrix matched as in (a) above. 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.**

c. Stock QC21 solution: (100 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier 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.

d. Stock Boron solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

e. Stock Tin solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

d. QC21 High (10 ug/mL). Dilute 50 mL of stock QC21 solution, 5 mL of stock Boron solution and 500 uL of stock Tin solution to 500 mL in a 500 mL volumetric flask, with 25 mL TRACE, Concentrated HCl and 10 mL TRACE, Concentrated HNO3. Prepare this solution fresh every three to six months.This standard is used to calibrate the following-As,B,Be,Cd,Co,Cr,Cu,Mn,Mo,Ni,Pb,Sb,Se,Sn,Ti,Tl,V,Zn.

e. Stock Silver solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

f. Stock Aluminum solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

g. Stock Calcium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

h. Stock Magnesium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

i. Stock Iron solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

j. Stock Potassium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

k. Stock Barium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

l. Stock Sodium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

m. NAK High solution: (500000 ug/L Al,Fe), (2000 ug/L Ag), (5000 ug/L Ba), (500 mg/L Mg). Dilute 25 mL stock Al, Fe, Mg; and 1 ml stock Ag solution; and 0.25 mL of stock Ba solution to 500 mL in a 500 mL volumetric flask with 25 mL TRACE, Concentrated HCl and 10 mL TRACE, Concentrated HNO3. Solution is stable for 6 months.

n. NAK 100ppm solution: (100 mg/L Ca, Na, K.) Dilute 5.0 mL stock Ca, Na, K solutions into a 500ml flask with 25 mL TRACE, Concentrated HCl and 10 mL TRACE, Concentrated HNO3. Solution is stable for 6 months.

o. Stock Arsenic solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

p. Stock Cobalt solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

q. Stock Chromium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

r. Stock Copper solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

s. Stock Manganese solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

t. Stock Nickel solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

u. Stock Lead solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

v. Stock Selenium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

w. Stock Thallium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

x. Stock Beryllium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

y. Stock Cadmium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

z. Stock Antimony solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

aa. Stock Molybdenum solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

bb. Stock Strontium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

cc. Stock Titanium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

dd. Stock Vanadium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

ee. Stock Zinc solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

ff. Stock Ultimate Low Standard: (200 µg/L Ag, As, Co, Cr, Cu, Mn, Ni, Pb, Se, Tl); (2500 µg/L Al); (2000 µg/L Fe); (50 µg/L Be, Cd); (10 mg/L B, Sn, Ca, Mg); (500 µg/L Sb, Zn); (1000 µg/L Mo, Sr, Ba, Ti); (200 µg/L V). Dilute 100 uL of stock Ag, As, Co, Cr, Cu, Mn, Ni, Pb, Se, V and Tl solutions; 125 µ/L of stock Al solution; 100 uL of stock

Fe solution; 5 mL of stock B solution; 500 uL of stock Sn, Ca, and Mg solutions; 250 uL of stock Sb and Zn solutions; 500 uL of stock Mo, Sr, Ba and Ti solutions; 1.0 mL of stock Cd and Be diluted to 100 mL and then use 2.5 mL of that solution diluted to 500 mL in a 500 mL volumetric flask with 25 mL TRACE, Concentrated HCl and 10 mL TRACE, Concentrated HNO3. Solution is stable for 6 months.

gg. Ultimate Low Standard: (20 µg/L Ag, As, Co, Cr, Cu, Mn, Ni, Pb, Se, Tl); (250 µg/L Al); (200 µg/L Fe); (5.0 µg/L Be, Cd); (1.0 mg/L B, Sn, Ca, Mg); (50 µg/L Sb, Zn); (100 µg/L Mn, Sr, Ba, Ti); (20 µg/L V). Dilute 10 mL of Stock Ultimate Low Standard to 100 mL in a 100 mL volumetric flask with 5 mL of TRACE, Concentrated HCl and 2 mL TRACE, Concentrated HNO3. This solution is prepared daily.

Stock Ultimate Low Standard and Ultimate Low Standard are only used when specified by client.

5. Calibration Verification standards

a. ICV Prep- 1.0 ml of QC-23, 1.0 ml of QC-7, 0.1 ml of 10,000 ppm Na, 0.09 ml of Fe at 10,000 ppm, and 0.09 ml of Al at 10,000 ppm diluted to 100 ml with 2% HNO3 and 5% HCL matrix. Mix well. CCV Prep- 1.0 ml of QC-21, 1.0 ml of QC-7, 1.0 ml of SN at 100 ppm, 0.1 ml of Na at 10,000 ppm, 0.09 ml of FE at 10,000 ppm,and 0.09 ml of Al at 10,000 ppm diluted to 100 ml with 2% HNO3 and 5% HCL matrix and mix well. This solution is prepared daily. These solutions are given a unique identifier and recorded in the standard log. ICV is prepared from a source independent from that used in the initial calibration.

b. The CRI solution is analyzed to check the accuracy of the instrument down near the contract required detection limits (CRDL). It is analyzed in conjunction with the interference check sample. The sample is prepared from a purchased solution which contains 120 µg/mL Sb, 100 µg/mL Co and V, 80 µg/mL Ni, 50 µg/mL Cu, 40 µg/mL Zn, 30 µg/mL Mn, 20 µg/mL As, Cr, Ag and Tl, 10 µg/mL Be, Cd and Se along with 6 µg/mL Pb. 500 µ/L of the solution is diluted to 500 mL. This solution is stable for 6 months.

b. The interference check solutions (ICSA and ICSAB) are prepared to contain known concentrations of interfering elements that will provide an adequate test of the IECs. A solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe is diluted 10x to prepare the ICSA. The ICSAB is prepared by diluting 100x a 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 solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe diluted 10x. These solutions are prepared as needed or monthly.

d. Reporting Limit Standard- Prepared 1.0 ml of RL Stock solution A and 1.0 ml of RL Stock Solution B diluted to 100 ml with 2% HNO3 and 5% HCL matrix , mix well. Solution stable for 3 months

6. Digestion standards

a. The Laboratory control sample (LCS) 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 is diluted to 500 mL with 0.125 mL of CLP-CAL-2 and CLP-CAL-3. 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 LCS 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. The LCS is prepared from a source independent from that used in the calibration standards. This solution is prepared daily or as needed. Note: The analysis of Molybdenum is not a routine procedure but a project-specific requirement. A customized LCSW mix must be prepared to contain this target analyte.

b. The solid Laboratory Control Sample (Soil) (LCSS) is prepared by weighing up 1.0 g of teflon chips and spiking using the same spiking solutions used to spike the sample matrix. This standard is given a unique identifier i.e. LCSS(date prepared)A,B,C etc.

c. The spiking solutions are prepared as follows:

1. Stock Multi-element Spiking Solutions: High Purity <u>CLP-CAL-1</u> <u>solution A</u>: 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; <u>solution B</u>: 250 ug/mL Ag; <u>CLP-CAL-2</u>: 1000 ug/L Sb; <u>CLP-CAL-3</u>: 1000 ug/mL As, Pb, Se, Tl; 500 ug/mL Cd. Order from the manufacturer already prepared. These solutions are given a unique identifier. Add 0.050 mL (0.20 mL for soil samples) of CLP-CAL-1 solutions A and B, and 0.0125 mL (0.05 mL for soil samples) of CLP-CAL-2 and 3 to 50 mL of sample (1gram of sample for 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.

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.

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 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.

VIII. CALIBRATION AND ASSOCIATED QA/QC

A. 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).

B. Operating conditions - **The instrument settings can be found in method file.** 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.

C. Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions. **(See SOP-106, *ICP Instrument Operation*)** Flush the system with 2% HNO_3 / 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 ($r \geq 0.995$). If a three point

calibration curve is not required for the client samples being analyzed Empirical Laboratories may use a blank and one standard as referenced in USEPA - CLP protocols.

D. Before beginning the sample run, analyze the 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. Then reanalyze the standard as a sample to check to see if the correction was made successfully. If the IEC correction overcompensated or under compensated for any one metal which is affected by it, the standard should be run again as a QC as before. 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 (± 10%) for 200.7 (± 5%) and ICB (< ±MDL or ±RL/CRDL for others or CLP, **for CCB, DOD QSM Ver. 3 no analytes detected >2xMDL**) first, 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.

E. 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: < ±MDL or ±RL/CRDL for others or CLP, **for CCB, DOD QSM Ver. 3 no analytes detected >2xMDL, beginning and end of sequence and after every 10 samples**) or twice during every 8-hour work shift, whichever is more frequent. Results should be within ±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)

F. Verify the interelement and background correction factors at the beginning and after each batch of 20 samples in the specific order of CRI, ICSA, ICSAB, CCV and CCB(CCB criteria: < ±MDL or ±RL/CRDL for others or CLP, **for CCB, DOD QSM Ver. 3 no analytes detected >2xMDL, beginning and end of sequence and after every 10 samples**) or twice during every 8-hour work shift, whichever is more frequent. Do this by analyzing the interference check solution A and AB. Results should be within ±20% of the true value for ICSAB. **For ICSA DOD QSM Ver 3. , absolute value of concentration for all non-spiked analytes < 2xMDL**.(ICSAB required at the end for CLP projects)

G. *When analyzing samples associated with North Carolina or with DOD QSM Ver. 3 work, a solution containing analytes at their reporting limit must be analyzed prior to sample analysis. The concentrations must be within 20% DOD(20 or 30% depending on project) of their true values to be acceptable.*

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.

H. The instrument must be calibrated once every 24 hours if performing straight CLP work.

I. Note: The instrument software is set up to go to the "L" rack for calibration standards, initial IEC standards and initial and continuing calibration verifications (ICV/CCV). This QC is inserted by the analyst when setting up the autosampler table which is what the instrument software uses for a RUN LOG (The software does not allow the analyst to print the actual run log but prints an autosampler report). The autosampler report is used to load the instrument and does not include ICV and CCVs in racks 2 through 5 because all calibration QC is loaded in the "L" rack and then samples and CCB's are loaded in Racks 2 through 5. When the samples are analyzed the software inserts ICVs and CCVs from the actual run log set up by the analyst. Since each CCV location is visited twice by the autosampler during the run, two locations will be called CCV1 and two locations will be CCV2 and so on. The time and date are printed on the instrument print out with the data so that the data user can distinguish between the two CCVs.

J. Instrument Autosampler Report example:

Calibration or "L" Rack(used by instrument software to insert QC)

1) STD 1-blank
2) NAK-100ppm
3) NAK-high
4) Fe-500,000ppb
5) Mn-10,000ppb
6) QC 21-high
7) Al IEC-QC(calculate IEC)
8) FE IEC-QC(calculate IEC)
9) ICV
10) CCV1
11) CCV2
12) CCV3

Sample Sequence RACK 1

1) Al IEC-(readback)
2) Fe IEC-(readback)
3) ICB-initial
4) RL-reporting limit standard

5) NAK 100ppm-(readback)

Sample Sequence RACK 1 (cont.')

6) NAK High-(readback)
7) QC 21 High-(readback)
8) Rinse
9) CRI-0
10) ICAS-0
11) ICASB-0
12) Rinse
13) CCB 1A
14) Preparation Blank
15) Laboratory Control Sample
16) Sample 1
17) Sample 2
18) Sample 3
19) Sample 4
20) Sample 5
21) Sample 6
22) Sample 7
23) Sample 8
24) CCB 1B
25) Sample 9
26) Sample 10
27) Sample 11
28) Sample 12
29) Sample 13
30) Sample 14
31) Sample 15
32) Sample 16
33) Sample 17
34) Sample 18
35) CCB2A
36) Sample 19
37) Sample 20
38) Sample serial dilution
39) Sample matrix spike
40) Sample matrix spike duplicate
41) Sample post digestion spike
42) CRI-1
43) ICSA-1
44) ICSAB-1
45) Rinse
46) CCB 2B
47) Preparation Blank
48) Laboratory Control Sample

RACK 2

1) Sample 1
2) Sample 2
Etcetera…

Each rack holds 48 samples and there are 4 racks that are used for samples and CCBs and run QC other then ICV/CCV's.

IX. PROCEDURE

A. Once the instrument has been calibrated, begin the analysis of samples.

B. 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. **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.**

C. Flush the system with 2% HNO_3 / 5% HCl for at least 1 minute before the analysis of each sample.

D. Dilute and reanalyze samples that are more concentrated than the linear calibration limit or, for 200.7, ± 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 RL are flagged as estimated with a "B" flag.**

E. 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.

- The results of the CCV are to agree within 10% for 6010 (5% for 200.7) on initial verification of the expected value, with relative standard deviation (RSD) < 5% from replicate (minimum of two 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.

- The results of the calibration blank (this is not the method/preparation blank) are to agree within < ±MDL(SW-846 Method 6010B), and 3 x IDL or CRDL for CLP, for **DOD QSM Ver. 3 no analytes detected**

>2xMDL. 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).

- Total hardness is reported from HNO_3 preserved sample. The final concentration is calculated from the calcium and magnesium results as follows:

  Ca mg/L x 2.5 + Mg mg/L x 4.1 = total Hardness in mg/L as $CaCO_3$

F. Documentation of Capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

X. CALCULATIONS

A. The instrument will generate data results in mg/L or µg/L (labeled appropriately). Each result represents an average of three individual readings per metal channel.

B. For aqueous samples, if a post/predigestion 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.

C. For solid samples, if a postdigestion 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 (ug/g or mg/kg) = IR*DF*FED/SM

SR = Sample result
IR = Instrument result (µg/L)
DF = Dilution factor (post digestion)
FED = Final volume of digestate (L)
SM = Sample mass digested(g)

XI. QUALITY CONTROL

A. Daily

1. See sections VIII and IX above.

B. Quarterly

1. Linear range standards must be analyzed at a frequency no less than once every three months. The linear range standard represents the second standard 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 +/-5% of true value.

2. The interelement correction factors (IEC) should be verified at the time the linear range standards are analyzed.

3. IDL's if CLP work required.

C. Digestion

1. All quality control data should be maintained and available for easy reference or inspection.

2. Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank, 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 ± ½ RL (USACE) or ±RL/CRDL for other or CLP. If exceeded, the impact upon the data should be evaluated and the associated sample(s) should be either redigested or the data should be qualified.

3. Employ a minimum of one laboratory control sample (LCS) for aqueous samples or one teflon chip spiked sample per sample batch to verify the digestion procedure. These LCSs are taken through the same digestion/preparation steps as the sample being tested. The control limits are ±15% method 200.7 - aqueous and soil samples or ±20% for all other methods aqueous and soil samples. If the LCS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be redigested. Consult your supervisor for further action. Qualifying the associated data may not be permissable for CLH.

D. Sample

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 20% RPD (if both are >5x CRDL) or ± the CRDL (if either are <5X CRDL).

2. Analyze one spiked sample and spiked sample duplicate 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. Project specific requirements will take precedence in these situations. If the analyte level in the sample is not greater than 4X the spiking level, the spike recoveries should be within ±25% of the true value (**± 20% for DOD projects**). If not, a post digestion spike should be analyzed.

3. The relative percent difference (RPD) between replicate determinations is to be calculated as referenced in the laboratory QA manual. (A control limit of ± 20% RPD (non-aqueous samples may routinely exceed this amount) shall be used for sample values greater than five times the contract required detection limit.) 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.

4. *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 ±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 85% to 115% of the known value and is required if the pre-digestion matrix spike (low-level only for CLH) 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. The analyst and or section manager must note this situation on the final analytical report.

E. Method Detection Limit (MDL), Empirical Laboratories Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength:

TABLE I

Aqueous and Soil Method Detection Limits(MDL), Empirical Laboratories Reporting Limits(ERL), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)

Analytes by EPA 200.7,3005A/3050A- 6010B SOW 4.1 & 5.2	AQUEOUS MDL (ug/L)	AQUEOUS ERL (ug/L)	AQUEOUS CRQL ILMO 4.1 (ug/L)	AQUEOUS CRQL ILMO 5.2 (ug/L)	SOLID/SOIL MDL (mg/Kg)	SOLID/SOIL ERL (mg/Kg)	SOLID/SOIL CRQL ILMO 4.1 (mg/Kg)	SOLID/SOIL CRQL ILMO 5.2 (mg/Kg
Silver	**1.0**	**10**	**10**	**10**	**0.20**	**2.0**	**2**	**2**
Aluminum	**50**	**200**	**200**	**200**	**10**	**40**	**40**	**40**
Arsenic	**3.0**	**10**	**10**	**15**	**0.6**	**2.0**	**2**	**3**
Barium	**5.0**	**200**	**200**	**200**	**1.0**	**40**	**40**	**40**
Beryllium	**1.0**	**5.0**	**5**	**5**	**0.20**	**1.0**	**1**	**1**
Calcium	**200**	**5000**	**5000**	**5000**	**20**	**1000**	**1000**	**1000**
Cadmium	**1.0**	**5.0**	**5**	**5**	**0.20**	**1.0**	**1**	**1**
Cobalt	**5.0**	**50**	**50**	**50**	**1.0**	**10**	**10**	**10**
Chromium	**2.0**	**10**	**10**	**10**	**0.40**	**2.0**	**2**	**2**
Copper	**4.0**	**25**	**25**	**25**	**0.40**	**5.0**	**5**	**5**
Iron	**30**	**100**	**100**	**100**	**1.0**	**20**	**20**	**20**
Potassium	**200**	**5000**	**5000**	**5000**	**40**	**1000**	**1000**	**1000**
Magnesium	**200**	**5000**	**5000**	**5000**	**40**	**1000**	**1000**	**1000**
Manganese	**1.0**	**15**	**15**	**15**	**0.20**	**3.0**	**3**	**3**
Sodium	**200**	**5000**	**5000**	**5000**	**40**	**1000**	**1000**	**1000**
Nickel	**3.0**	**40**	**40**	**40**	**1.0**	**8.0**	**8**	**8**
Lead	**1.5**	**5.0**	**3**	**10**	**0.60**	**2.0**	**0.6**	**2**
Selenium	**3.0**	**10**	**5**	**35**	**0.60**	**2.0**	**1**	**7**
Antimony	**5.0**	**60**	**60**	**60**	**1.0**	**12**	**12**	**12**
Thallium	**3.0**	**10**	**10**	**25**	**0.60**	**2.0**	**2**	**5**
Vanadium	**5.0**	**50**	**50**	**50**	**1.0**	**10**	**10**	**10**
Zinc	**5.0**	**20**	**20**	**60**	**1.0**	**4.0**	**4**	**12**

TABLE 2

METAL	WAVELENGTH
Aluminum	**308.2**
Antimony	**206.8**
Arsenic	**189.0**
Barium	**493.4**
Beryllium	**313.0**
Cadmium	**226.5**
Calcium	**317.9**
Chromium	**267.7**
Cobalt	**228.6**
Copper	**324.7**
Iron	**271.4**
Lead	**220.3**
Magnesium	**279.0**
Manganese	**257.6**
Nickel	**231.6**
Potassium	**766.4**
Selenium	**196.0**
Silver	**328.0**
Sodium	**330.2**
Thallium	**190.8**
Vanadium	**292.4**
Zinc	**206.2**

XII. CORRECTIVE ACTIONS

A. INSTRUMENT RELATED

1. ICV not within ± 10% or ± 5% for 200.7
   a. Is the problem with the solution?
      i. Reprepare or obtain new stock.
   b. Is the problem with the calibration?
      i. Recalibrate through analysis of appropriate standards and recheck ICV.

2. ICB not ±MDL or within ± 3X IDL or CRDL for CLP, **DOD QSM Ver. 3 no analytes detected >2xMDL**

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

3. Check standards not within ± 5%
   a. Is the problem with the solution?
      i. Repour, reprepare or obtain new stock.
   b. Is the problem with the calibration?
      i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

4. CRI not within ± 20% (Internal QC, only required for CLP work).
   a. Is the problem with the solution?
      i. Reprepare or obtain new stock.
   b. Is the problem with the calibration?
      i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.
5. ICSA metals not present are not less than the CRDL for that metal, **for ICSA DOD QSM Ver 3. , absolute value of concentration for all non-spiked analytes < 2xMDL.**
   a. Is the problem with the solution?
      i. Reprepare or obtain new stock.
   b. Is the problem with the calibration?
      i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

6. ICSAB not within ± 20%
   a. Is the problem with the solution?
      i. Reprepare or obtain new stock.
   b. Is the problem with the calibration?
      i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

7. CCV not within ± 10%
   a. Is the problem with the solution?
      i. Reprepare 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. Note: CLH data must always be reanalyzed back to the last compliant CCV and not qualified.

8. CCB not ±MDL or within ± 3X IDL or CRDL for CLP, **DOD QSM Ver. 3 no analytes detected >2xMDL**

a. Is the problem with the solution?
   i. Reprepare
b. Is the problem with the calibration?
   i. Apply SW846 guidance. (See Section IX-E for additional guidance). Note: CLH data must always be reanalyzed back to the last compliant CCB and not qualified.

B. DIGESTION RELATED

1. Preparation blank not within ± ½ RL and ± RL for common contaminants USACE 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 redigested or the data must be qualified on the final report.

2. LCS 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 redigested.
      ii. If biased high, the impact upon the data user must be evaluated. The samples will be redigested or the data will be qualified on the final report.

C. SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within ±20% (if both are >5X CRDL) or ± the CRDL (if either are <5X CRDL).
   a. The associated sample data must be qualified on the final report.

2. Spike analysis recovery not within ±25%(**± 20% for DOD projects**)
   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.

3. When required, post digestion spike analysis recovery not within ±15%.
   a. The associated sample data must be qualified on the final report.
   b. For USACE analysis by MSA is required.

4. Serial dilution analysis percent difference not within ±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.
      iii. 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.

XIII. WASTE DISPOSAL and POLLUTION PREVENTION

Please see Waste Disposal SOP-405 fot instruction of proper 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.

XIV. REFERENCES

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

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

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

Refer to SOP-431 for common environmental laboratory definitions.

Addendum for USEPA CLPILM 05.2

1. The control limit for the ICSA is at 20% or ±CRQL whichever is greater.
2. Preparation codes are required in the digestion log See SOW Exhibit B for a listing of these codes with definitions.

3. The CRQL check standard is run at the concentration of the respective CRQLs. For a listing of CRQL for this SOW see Exhibit C. Several of the metals concentration levels have changed.
4. The spiking level for CLP ILM 05.2 is at 50 ug/L for selenium. All other spike levels remain the same as in SOW ILM 04.1.
5. The CCV shall be analyzed at a different concentration then the ICV (at or near one-half of the calibration standard concentration.
6. The post digestion spike must be analyzed at 2x the indigenous level of the sample or two times the CRQL whichever is greater.
7. A Non-prepared MDL study must be analyzed and the results of this study used for MDL reporting when sample volumes are not digested.

CHANGES TO FORMS for SOWCLPILM 05.2

1. Forms must be double-sided
2. A photocopy of the instrument's direct sequential readout shall be included.
3. Undiluted samples must be reported as well as diluted samples.
4. J flags are used in place of B flags when a sample has a concentration less the CRQL but greater then or equal to the MDL.
5. A D flag is used for samples reported from a dilution.
6. All results are reported down to the MDL not the IDL.
7. Preparation codes are used on form 13.

   The form for method of standard additions (MSA) has been removed and all subsequent QC has move up one form number in other words form 8 is now serial dilution when it used to be the MSA form,etcetera.

ANALYST DATA REVIEW CHECKLIST Sample Number(s):
Batch Number(s):
Method: 6010B (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 LCS or blank spike meet control limits?				
6. Did MS/MSD meet control limits?				
7. Was the preparation (Method) Blank 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: _____

Second-Level Review: _____ Date: _____

DETERMINATION OF INORGANIC ANIONS IN WATER BY ION CHROMATOGRAPHY USING THE DIONEX DX-500 ION CHROMATOGRAPH WITH HYDROXIDE ELUENT AND DIONEX COLUMN AS18 BY METHOD 300.0/ SW846 9056 GUIDANCE

SOP NUMBER: SOP-145

REVISION NUMBER: 6

APPROVED BY:

SECTION MANAGER

QUALITY ASSURANCE OFFICER

EFFECTIVE DATE: 01/09/09

DATE OF LAST REVIEW: 01/09/09

DETERMINATION OF INORGANIC ANIONS IN WATER BY ION CHROMATOGRAPH USING THE DIONEX dx-500 ION CHROMATOGRAPH WITH HYDROXIDE ELUENT AND DIONEX AS18 COLUMN

References:
USEPA METHOD 300.0/ SW846 Method 9056

I. SCOPE AND APPLICATION:

1. This method covers the determination of the following inorganic common anions in reagent water, surface water, ground water, and other aqueous matrixes.

PART A.--Common Anions

Chloride	Nitrate	Fluoride	Sulfate
Nitrite	Bromide	Ortho Phosphate	

2. Single laboratory Method Detection Limit for the above analytes is listed in Tables 1A, 1B and 1C from method 300.0. The MDL for a specific matrix may differ from those listed, depending upon the nature of the sample and the specific instrumentation employed.

   A. In order to achieve comparable detection limits, an ion chromatographic system must utilize suppressed conductivity detection, be properly maintained and must be capable of yielding a baseline with no more then a 5 nS noise/drift per minute of monitored response over the background conductivity.

3. This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.

4. When the method is used to analyze unfamiliar samples for any of the above anions, anion identification should be supported by the use of a laboratory fortified matrix sample covering the anions of interest. The fortification procedure is described in the Quality Control section.

5. Users of the method data should state the data-quality objectives prior to analysis. Analyst using this method must demonstrate the ability to generate acceptable results with the method, using the procedures described in the Quality Control section.

II. SUMMARY OF METHOD

1. A small volume of sample, 50 uL for Part A is introduced into an ion chromatograph (IC). The anions of interest are separated and measured, using a

system comprised of a guard column, analytical column, suppressor device, and conductivity detector.

III. DEFINITIONS

1. **ANALYSIS BATCH** -- A group of no more than 20 field samples (Field sample analyses include only those samples derived from a field sample matrix. These include the initial and duplicate field samples as well as all Laboratory Fortified Sample Matrices). The analysis batch must include an Initial Calibration Check Standard, and End Calibration Check Standard, Laboratory Reagent Blank, and a Laboratory Fortified Blank. Within an ANALYSIS BATCH, for every group of **ten** field samples at least one Laboratory Fortified Matrix (LFM) and either a Field Duplicate, a Laboratory Duplicate or a duplicate of the LFM must be analyzed after the tenth field sample analysis.

2. **CALIBRATION STANDARD (CAL)** -- A solution prepared from the primary dilution standard solution or stock standard solutions and the surrogate analyte. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

   A. **INITIAL CALIBRATION STANDARDS --** A series of CAL solutions used to initially establish instrument calibration and develop calibration curves for individual target anions.

   B. **INITIAL CALIBRATION CHECK STANDARD** -- An individual CAL solution, analyzed initially, prior to any sample analysis, which verifies previously established calibration curves.

   C. **CONTINUING CALIBRATION CHECK STANDARD** -- An individual CAL solution which is analyzed after every tenth field sample analyses which verifies the previously established calibration curves and confirms accurate analyte quantitation for the previous ten field samples analyzed.

   D. **END CALIBRATION CHECK STANDARD** -- An individual CAL solution which is analyzed after the last field sample analyses which verifies the previously established calibration curves and confirms accurate analyte quantitation for all field samples analyzed since the last continuing calibration check.

3. **FIELD DUPLICATES (FD) --**Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout the field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

4. **INSTRUMENT PERFORMANCE CHECK SOLUTION (IPC) --** A solution of one or more method analytes, surrogates or other test substances used to

evaluate the performance of the instrument system with respect to a defined set of criteria.

5. **LABORATORY DUPLICATE --** Two sample aliquots, taken in the laboratory from a single sample bottle, and analyzed separately with identical procedures. Analyses of LD1 and LD2 indicate precision associated specifically with the laboratory procedures, removing any associated variables attributed by sample collection, preservation, or storage procedures.

6. **LABORATORY FORTIFIED BLANK (LFB) --**An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

7. **LABORATORY FORTIFIED SAMPLE MATRIX (LFM) --** An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

8. **LABORATORY REAGENT BLANK (LRB) --** An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

9. **LINEAR CALIBRATION RANGE (LCR) --** The concentration range over which the instrument response is linear.

10. **MATERIAL SAFETY DATA SHEET (MSDS) --** Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

11. **METHOD DETECTION LIMIT (MDL) --** The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

12. **MINIMUM REPORTING LEVEL (MRL) --** The minimum concentration that can be reported for an anion in a sample following analysis. This defined concentration can be no lower than the concentration of the lowest calibration standard and can only be used if acceptable quality control criteria for this standard are met.

13. **PERFORMANCE EVALUATION SAMPLE (PE) --** A certified solution of method analytes whose concentration is unknown to the analyst. Often, an aliquot of this solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of analyses are used to determine statistically the accuracy and precision that can be expected when a method is performed by a competent analyst.

14. **QUALITY CONTROL SAMPLE (QCS) --** A solution of method analytes of known concentrations that is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

15. **STOCK STANDARD SOLUTION (SSS) --** A concentrated solution containing one or more method analytes prepared in the laobratory using assayed reference materials or purchased from a reputable commercial source.

IV. INTERFERENCES

1. Interferences can be divided into three different categories: **direct chromatographic coelution**, where an analyte response is observed at very nearly the same retention time as the target anion; **concentration dependant coelution**, which is observed when the response of higher than typical concentrations of the neighboring peak overlap into the retention window of the target anion; and, **ionic character displacement**, where retention times may significantly shift due to the influence of high ionic strength matrices (high mineral content or hardness) overloading the exchange sites in the column and significantly shortening target analytes's retention times.

   A. A direct chromatographic coelution may be solved by changing columns, eluent strength, modifying the eluent with organic solvents (if compatible with IC columns), changing the detection systems, or selective removal of the interference with pretreatment. Sample dilution will have little to no effect. The analyst must verify that these changes do not negatively affect performance by repeating and passing all the criteria in the Quality Control Section.

   B. Sample dilution may resolve some of the difficulties if the interference is the result of either concentration dependant coelution or ionic character displacement, but it must be clarified that sample dilution will alter your Minimum Reporting Limit (MRL) by a proportion equivalent to that of the dilution. Therefore, careful consideration of project objectives should be given prior to performing such a dilution. An alternative to sample dilution, may be dilution of the eluent.

C. Pretreatment cartridges can be effective as a means to eliminate certain matrix interferences. Prior to using any pretreatment, the analyst should be aware that all **instrument calibration standards must be pretreated in exactly the same manner** as the pretreated unknown field samples. The need for these cartridges has been greatly reduced with recent advances in high capacity anion exchange columns.

   1. Extreme caution should be exercised in using these pretreatment cartridges. Artifacts are known to leach from certain cartridges, which can foul the guard, and analytical columns causing loss of column capacity indicated by shortened retention times and irreproducible results. Frequently compare your calibration standard chromatograms to those of the column test chromatogram (received when the column was purchased) to insure proper separation and similar response rations between the target analytes is observed.

D. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in an ion chromatogram. These interferences can lead to false positive results for target analytes as well as reduced detection limits as a consequence of elevated baseline noise.

E. Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

F. Any anion that is only weakly retained by the column may elute in the retention time window of fluoride and potentially interfere. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant; however, it is the responsibility of the analyst to generate precision and accuracy information in each sample matrix.

G. Close attention should be given to the potential for carry over peaks from one analysis which will effect the proper detection of analytes of interest in a second, subsequent analysis. The elution of nitrate (retention time of ~9.0 min.) indicates the end of a chromatographic run. A run time of 12 minutes is recommended to allow for the proper elution of any potentially interferrant late peaks. It is the responsibility of the analyst to confirm that no late eluting peaks have carried over into a subsequent analysis thereby compromising the integrity of the analytical results.

2. SAFETY

A. The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential heath hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

B. Your laboratory manager and/or Safety Officer is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. A formal safety plan is also available. Use proper personal protection equipment, PPE, such as safety glasses, gloves and laboratory coats should be warn when handling samples and chemicals.

V. EQUIPMENT AND SUPPLIES

1. Ion Chromatograph (IC) – Analytical system complete with eluent generator, an ion chromatographic pump, injection valves, both guard and analytical separator columns, suppressor, conductivity detector, and computer based data acquisition system. Dionex DX-500 or equivalent. (See letter from EPA to Dionex on discussion of alternate hydroxide eluent for anions. Also since hydroxide eluent cannot be run on the traditional column Dionex AS18, 4mm (P/N 060549) or equivalent should be used.

   a. Anion guard column--Dionex Ion Pac AG18 4mm (P/N 060551), or equivalent. This column functions as a protector of the separator column. If omitted from the system, the retention times will be shorter.

   b. Anion separator column--Dionex Ion Pac AS18, 4mm (P/N 060549), or equivalent. An optional column (2mm or 4 mm) may be used if comparable resolution of peaks is obtained, and the quality control requirements can be met.

      i. When a 4 mm column is employed, the injection volume should be 50 uL.

      ii. Comparable results can be attained using the Dionex, AS17, 4 mm column.

2. Anion suppressor device--The data presented in this method were generated using an Ultra 4 mm Dionex Anion Self Regenerating Suppressor (ASRS, P/N 53946). An equivalent suppressor device may be utilized provided comparable conductivity detection limits are achieved and adequate baseline stability is attained as measured by a combined baseline drift/noise of no more than 5 nS per minute over the background conductivity. Proper suppressor performance is essential to analytical data reproducibility and sensitivity of the conductivity detector.

   a. The ASRS was set to perform electrolytic suppression at a current setting of 300 ma using the external water mode. External water was delivered to the suppressor directly from a pressurized source at a flow rate of 5 mL/min. It should be noted that while Empirical Laboratories has the suppressor currently set at 300 mA, no external water is being used at this time.

3. Detector--Conductivity cell (Dionex CD20, or equivalent) capable of providing data as required in the Quality Control section of this SOP.

4. Data Acquisition System--The Dionex Peaknet Data Chromatography Software version 5.2 or equivalent is used by Empirical Laboratories.

5. Analytical balance--Mettler Used to accurately weigh target analyte salt for stock standard preparation (±0.1 mg sensitivity).

6. Micro beakers -- Plastic, disposable - used during sample preparation.

7. Syringes--Plastic, disposable, 10 mL - used during sample preparation.

8. Pipets -- Pasteur, plastic or glass, disposable, graduated, 5 mL and 10 mL.

9. Bottles -- High density polyethylene (HDPE) or glass, amber or clear, 30 mL, 125 mL, 250 mL. For sampling and storage of calibration solutions.

10. Particulate filters-- 0.45 micron syringe filters, specifically designed for IC applications (Gelman IC Acrodisc, PN 4485, or equivalent). These cartridges are used to remove particulates from the sample matrix while loading the sample manually or if the autosampler employed does not filter the sample during loading.

NOTE: See method for several types of pretreatment cartridges that are available and may be useful depending on the matrices of the samples normally processed.

11. Autosampler PolyVials 5-mL size, with filtercaps, 250 each --Dionex cat log # 38141.

12. Shaker for use when extracting soil samples.

13. Centrifuge to aid in separation after extraction.

14. Centrifuge tubes--50 mL capacity

VI. REAGENTS AND STANDARDS

1. Reagent water-- Distilled or deionized water 17.8 Mohm or better, free of anions of interest. Water should contain particles no larger than 0.20 microns.

2. A system or apparatus which automatically generates the hydroxide eluent (Dionex EG40, or equivalent) is an acceptable alternative to physically preparing the hydroxide eluent.

3. Stock standard solutions, 1000 mg/L (1mg/mL): Stock standard solutions are purchased as certified solutions from selected vendors.

NOTE: Stability of standards: Stock standards for most anions are stable for at least 6 months when stored at 4 °C. Dilute working standards should be prepared monthly.

VII. SAMPLE COLLECTION, PRESERVATION AND STORAGE

1. Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

2. Sample preservation and holding times for the anions that can be determined by this method are as follows:

PART A: Common Anions

Analyte	Preservation	Holding Time
Bromide	None required	28 days
Chloride	None required	28 days
Fluoride	None required	28 days
Nitrate-N	**Cool to 4 °C**	**48 hours**
Nitrite	**Cool to 4 °C**	**48 hours**
Ortho Phosphate	**Cool to 4 °C**	**48 hours**
Sulfate	Cool to 4 °C	28 days

3. When collecting a sample from a treatment plant employing chlorine dioxide, the sample must be sparged with an inert gas (helium, argon, nitrogen) prior to addition of the addition of the EDA preservative at time of sample collection.

VIII. QUALITY CONTROL

1. The laboratory is required to operate a formal quality control (QC) program. The requirements of this program consist of an initial demonstration of laboratory performance, and subsequent analysis in each analysis batch of a Laboratory Reagent Blank, Laboratory Fortified Blank, Instrument Performance Check Standard, calibration check standards, Laboratory Fortified Sample Matrices (LFM) and either Field, Laboratory or LFM duplicate sample analyses. This section details the specific requirements for each of these QC parameters. The laboratory is required to maintain performance records that define the quality of the data that are generated.

2. INITIAL DEMONSTRATION OF PERFORMANCE

   A. The initial demonstration of performance is used to characterize instrument performance (determination of accuracy through the analysis of the QCS) and laboratory performance (determination of MDLs) prior to performing analysis by this method.

   B. Quality Control Sample (QCS) – When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS. If the determined concentrations are not within ±10% of the stated values, performance of the determinative step of

the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.

C. Method Detection Limit (MDL)—MDLs are established for all analytes, using reagent water (blank) fortified at a concentration of three to five times the estimated instrument detection limit. To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method over at least three separate days. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

$$MDL = (t) \times (S)$$

Where,

t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.14 for seven replicates].

S = standard deviation of the replicate analyses.

D. MDLs should be determined every 6 months or at least annually, when a new operator begins work or whenever there is a significant change in the background, or instrument response. MDL check samples are used in connection with confirming that the MDL determined is legitiment and to monitor the instrument sensitivity periodically. MDL checks are analyzed whenever a new MDL is generated and at a minimum quarterly to monitor for any shifts in sensitivity.

3. ASSESSING LABORATORY PERFORMANCE

A. Laboratory Reagent Blank (LRB) – The laboratory must analyze at least one LRB with each analysis batch. Data produced are used to assess contamination from the laboratory environment. Values that exceed the MDL (**For DOD QSM Ver. 3 no analytes detected ≥ ½ RL or for common lab contaminants no analyte detected ≥ RL**) indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis.

B. Laboratory Fortified Blank (LFB)—The LFB should be prepared at concentrations similar to those expected in the field samples and ideally at the same concentration used to prepare the LFM. Calculate accuracy as percent recovery. If the recovery of any analyte falls outside the required concentration dependant control limits that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.

i. Control Limits for the LFB are 90 to 110%.

ii. The laboratory uses the LFB to assess laboratory performance against the required control limits listed in the QC section. When sufficient internal performance data becomes available (usually a minimum of 20-30 analyses), optional control limits can be developed from the percent mean recovery (x) and the standard deviation (S) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

UPPER CONTROL LIMIT = x + 3S
LOWER CONTROL LIMIT = x – 3S

The optional control limits must be equal to or better than those listed in the QC section (±10%). After each five to ten new recovery measurements, new control limits can be calculated using only the most recent 20-30 data points. Also, the standard deviation (S) data should be used to establish an on-going precision statement for the level of concentrations monitored. These data must be on file and be available for review.

i. Instrument Performance Check Solution (IPC) – The Initial Calibration Check Standard is to be evaluated as the instrument performance check solution in order to confirm proper instrument performance. The acceptable limits for this standard is 90 to 110%. Small variations in retention time can be anticipated when a new solution of eluent (or when the KOH cartridge is changed) is prepared but if shifts of more that 2% are observed in the IPC retention time, some type of instrument problem is present. Potential problems improperly prepared eluent, erroneous method parameters programmed such as flow rate or some other system problem. The chromatographic profile (elution order) of the target anions following an ion chromatographic analysis should closely replicate the profile displayed in the test chromatogram that was shipped when the column was purchased. As a column ages, it is normal to see a gradual shift and shortening of retention times, but if after several years of use, extensive use over less than a year, or use with harsh samples, this retention time has noticeably shifted to any less than 80% of the original recorded value, the column may require cleaning or replacement. Particularly if resolution problems are beginning to become common between previously resolved peaks. A laboratory must retain a historic record of retention times for all the target anions in the IPC to provide evidence of an analytical column's vitality.

4. ASSESSING ANALYTE RECOVERY AND DATA QUALITY

A. Laboratory Fortified Sample Matrix (LFM) – The laboratory adds a known amount of analyte to a minimum of 10% of the field samples within an analysis batch. The LFM sample is prepared from a sample matrix which has been analyzed prior to fortification. The analyte concentration must be high enough to be detected above the original sample and should adhere to the QC requirements. It is recommended that the solutions used to fortify the LFM be prepared from the same stocks used to prepare the calibration standards and not from external source stocks. This will remove the bias contributed by an externally prepared stock and focus on any potential bias introduced by the field sample matrix.

i. If the fortified concentration is less than the observed background concentration of the unfortified matrix, the recovery should not be calculated. This is due to the difficulty in calculating accurate recoveries of the fortified concentration when the native sample concentration is so high.

ii. The LFM should be prepared at concentrations no greater than five times the highest concentration observed in any field sample. If no analyte is observed in any field sample, the LFM must be fortified no greater than five times the lowest calibration level which as outlined in this method is the minimum reported level (MRL). For example, if chloride is not detected in any field samples above the lowest calibrations standard concentration of 5.00 ug/L, the highest LFM fortified concentration allowed is 25.0 ug/L.

iii. Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample. Percent recovery should be calculated using the following equation:

$$R = \frac{C_s - C}{s} \times 100$$

where, R = percent recovery.
C_s = fortified sample concentration
C = sample background concentration
S = concentration equivalent of analyte added to sample.

iv. Until sufficient data becomes available (usually a minimum of 20 to 30 analysis), assess laboratory performance against recovery limits of 80 to 120%. When sufficient internal performance data becomes available develop control limits from percent mean recovery and the standard deviation of the mean recovery. The optional control limits must be equal to or better than the required control limits of 80 –120%.

v. If the recovery of any analyte falls outside the designated LFM recovery range and the performance for that analyte is shown to be in control, the recovery problem encountered with the LFM is judged to be matrix induced and the results for that sample and the LFM are reported with a "matrix induced bias" qualifier.

B. FIELD OR LABORATORY DUPLICATES –Analyze either a field, matrix spike duplicate or a laboratory duplicate for a minimum of 10% of the collected field samples or at least one with every analysis batch, whichever is greater. The sample matrix selected for this duplicate analysis must contain measurable concentrations of the target anions in order to establish the precision of the analysis set and insure the quality of the data. If none of the samples within an analysis batch have measurable concentrations, the LFM should be employed as a laboratory duplicate.

i. Calculate the relative percent difference (RPD) of the initial quantitated concentration (I_c) and duplicate quantitated concentration (D_c) using the following formula,

$$RPD = \frac{(I_c - D_c)}{([I_c + D_c]/2)} \times 100$$

ii. Duplicate analysis acceptance criteria

Concentration range	RPD Limits
MRL to 10xMRL	± 20%
10xMRL to highest calibration level	± 10%

iii. If the RPD fails to meet these criteria, the samples must be reported with a qualifier identifying the sample analysis result as yielding a poor duplicate analysis RPD. This should not be a chronic problem and if it frequently recurs (>20% of duplicate analyses) it indicates a problem with the instrument or individual technique.

C. Where reference materials are available, they should be analyzed to provide additional performance data. The analysis of reference samples is a valuable tool for demonstrating the ability to perform the method acceptably.

D. In recognition of the rapid advances occurring in chromatography, the analyst is permitted certain options, such as the use of different columns, injection volumes, and/or eluents, to improve the separations or lower the cost of measurements. Each time such modifications to the method are made, the analyst is required to repeat the procedure in the QC section and adhere to the condition of baseline stability.

E. The laboratory adopts additonal quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the client and the nature of the samples. Whenever possible, the laboratory performs analysis of quality control check samples and participate in relevant performance evaluation sample studies.

5. CALIBRATION AND STANDARDIZATION

A. Establish ion chromatographic operating parameters equivalent to those indicated in Tables 1C for a 4-mm column.

   i. Estimate the Linear Calibration Range (LCR) – The LCR should cover the expected concentration range of the field samples.

   ii. For an individual calibration curve, a minimum of eight calibration standards is required for a curve.

B. Prepare the calibration standards by carefully adding measured volumes of one or more stock standards to a volumetric flask and diluting to volume with reagent water. Chloride and sulfate are calibrated from 0.5-200 mg/L; fluoride, nitrate, and nitrite from 0.05-20 mg/L.

C. Using a 4mm column, inject 50 uL (Part A) of each calibration standard. Tabulate peak area responses against the concentration. The results are used to prepare calibration curves using a linear least squares fit for each analyte. Acceptable calibration curves are confirmed after reviewing the curves for linearity and passing the criteria for the initial calibration check standard. Alternately, if the ratio of response to concentration (response factor) is constant over the LCR (indicated by < 15% relative standard deviation (RSD)), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

   i. Peak areas strongly recommended since they have been found to be more consistent, in terms of quantitation, than peak heights. Peak height can tend to be suppressed as a result of high levels of common anions in a given matrix which can compete for exchange sites. Using peak areas, it is the analyst responsibility to review all chromatograms to insure accurate baseline integration of target analyte peaks since poorly drawn baselines will more significantly influence peak areas than peak heights.

D. Once the calibration curves have been established they must be verified prior to conducting any sample analysis using an initial calibration check standard. This verification must be performed on each analysis day or whenever fresh eluent has been prepared. A continuing calibration check standard must be

analyzed after every tenth sample and at the end of the analysis set as an end calibration check standard. The response for the initial, continuing and end calibration check must satisfy the QC criteria of 90 to 110%. If during the analysis set, the response differs by more than the calibration verification criteria, or the retention times shift more than ±5% from the expected values for any analyte, the test must be repeated, using fresh calibration standards. If the results are still outside these criteria, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable calibration check standard must be reanalyzed.

i. Control limits for calibration verification are 90 to 110%.

F. After satisfying the requirements, the levels selected for the other calibration check standards should be varied between a middle calibration level and the highest calibration level.

6. **PROCEDURE**

A. Other columns, chromatographic conditions, or detectors may be used if the requirements of the QC section are met.

B. Check system calibration daily and, if required, recalibrate as necessarily.

C. Sample Preparation

i. For refrigerated or samples arriving to the laboratory cold, ensure the samples have come to room temperature prior to conducting sample analysis by allowing the samples to warm on the bench for at least 1 hour.

D. Using a Luer lock, plastic 5 to 10 mL syringe, withdraw the sample from the micro beaker and attach a 0.45 um particulate filter (demonstrated to be free of ionic contaminants) directly to the syringe. Filter the sample into an autosampler vial.

E. Using a 4 mm column, inject 50 uL of each sample. Tabulate peak area responses against the concentration. During this procedure, retention times must be recorded. Use the same size loop for standards and samples. Record the resulting peak size in area units. An automated constant volume injection system may also be used.

F. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte.

However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

G. If the response of a sample analyte exceeds the calibration range, the sample may be diluted with an appropriate amount of reagent water and reanalyzed. If this is not possible then three new calibration concentrations must be employed to create a separate high concentration curve, one standard near the estimated concentration and the other two bracketing around an interval equivalent to ±25% the estimated concentration. The latter procedure involves significantly more time than a simple sample dilution therefore; it is advisable to collect sufficient sample to allow for sample dilution or sample reanalysis, if required.

H. Shifts in retention time are inversely proportional to concentration. Nitrate, phosphate and sulfate will exhibit the greatest degree of change, although all anions can be affected. In some cases this peak migration may produce poor resolution or make peak identification difficult.

I. Should more complete resolution be needed between any two coeluting peaks, the eluent can be diluted. This will spread out the run, however, and will cause late eluting anions to be retained even longer. The analysts must verify that this dilution does not negatively affect performance by repeating and passing all the QC criteria.

   i. Eluent dilution will reduce the overall response of an anion due to chromatographic band broadening which will be evident by shortened and broadened peaks. This will adversely effect the MDLs for each analyte.

7. DATA ANALYSIS AND CALCULATIONS

A. Prepare a calibration curve for each analyte by plotting instrument response, as peak area, against standard concentration. Compute sample concentration by comparing sample response with the standard curve. If a sample has been diluted, multiply the response by the appropriate dilution factor.

B. Report ONLY those values that fall between the lowest and the highest calibration standards. Samples with target analyte responses exceeding the highest standard should be diluted and reanalyzed. Samples with target analytes identified but quantitated below the concentration established by the lowest calibration standard should be reported as below the minimum reporting limit (MRL).

C. Report results for Part A anions in mg/L.

D. Report NO_3^- as N

Traceability

A bound logbook 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 logbook as well as on the container's label.

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, date opened, and the logbook where information is recorded. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in a bound logbook. 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.

The analyst must initial and date each entry made in a logbook. Each analyst must be sure to "Z" out the unused area of each logbook page.

IX. INSTRUMENT INFORMATION

Analyst should confirm the following:

1. **Start Up routine for instrument is as follows:**

   a. **Turn Power on to autosampler, conductivity detector, eluent generator and pump in any order.**

   b. **Turn on the Helium gas supply~ 80 to 100. Ensure gas lines to bottles not in use are off. Ensure air supply to the injection valve is on.**

   c. **Close the vent valves on sparging bottles and allow head pressure to build for a few moments. DI H2O bottles should be 7 to 10 psi.**

   d. **Open the eluent supply valve(s) for the bottles in use. Check for sputter after eluent flow has started.**

   e. **Load the autosampler cartridges and put autosampler into RUN state.**

   f. **To vent airlocks, Run eluent with pump on and SRS off, open waste valve-> bottom door top black knob, just for a few seconds and close it.**

   g. **Turn on the SRS power. NOTE: NEVER TURN ON THE SRS POWER SUPPLY WITHOUT THE PUMP GOING FIRST!!! Use either LOCAL/DIRECT CONTROL to enter commands at keypads, or REMOTE/DIRECT CONTROL to use the direct control option from the RUN menu within PEAKNET.**

   h. **After System has come to equilibrium, load sequence and run.**

2. **Shut Down routine for instrument is as follows:**

   a. **If the instrument is not going to be operated for a period of time, run deionized water through the eluent lines for ~ 30 minutes to an hour to rinse the lines.**

   b. **Stop the OFF/ON pump and then select SRS-OFF.**

   c. **Close gas supply valves and eluent valves on the eluent bottles. Turn off He supply. Is not necessary to vent the eluent bottles.**

   d. **Power down the modules in any order.**

3. **General Sample Loading and Run Set-up.**

   a. **Enter Peak-net Software from Desktop.**

   b. **Loading a Run: Click on <u>Schedule</u>. The headings within the Schedule Editor are SAMPLE, SAMPLE TYPE, LEVEL, METHOD and DATA FILE.**

      i. **Name each sample under the SAMPLE heading column. (ICB, ICV, LCSW, ICCS, IPC, sample #'s, etc.)**

      ii. **SAMPLE TYPE is sample unless loading a calibration curve.**

      iii. **LEVEL designations are used only when assigned to a calibration curve.**

      iv. **Enter method name under METHOD heading. In most cases, date of most recent calibration in Anions Method file will be used.**

      v. **Enter the date under the DATA FILE heading. The program will then sequentially assign the data file names based on the date.**

      vi. **All other column headings are defaulted to enter "1". Samples requiring dilution should be left at "1" and manual calculation is required.**

      vii. **To include a command to Shut Down the pump at the end of the run: Name the row following the last sample, Pump Off under the sample heading. It is not necessary to include a vial in the corresponding position in the autosampler. Sampe type is Sample, and Method is entered as <pumpoff.met>.**

      Typical run-log:

      1 Blank
      2 RLS@ 0.05/0.05ppm

3 ICV @ 2.5/25ppm
4 ICB
5 RLS @0.5ppm SO_4
6 LCSW ISA…….
7 Sample (Cl, SO_4….)
8 Sample (Cl, SO_4….)
9 Sample (Cl, SO_4….)
10 Sample (Cl, SO_4….)
11 Sample @ 10X(Cl)
12 Sample @ 50X(Cl)
13 CCV @ 2.5/2ppm
14 CCB
15 Sample (Cl, SO_4….)
16 Sample MS
17 Sample MSD
18 Sample (Cl, SO_4….)
19 Sample @ 10X (Cl)
20 Sample (Cl)
21 Sample (Cl, SO_4….)
22 Sample (Cl, SO_4….)
23 CCV @ 2.5/25ppm
24 CCB
25 Sample (Cl, SO_4….)
26 Sample (Cl, SO_4….)
27 Sample (Cl, SO_4….)
28 Sample (Cl, SO_4….)
29 Sample (Cl, SO_4….)
30 Sample MS
31 Sample MSD
32 Sample (Cl, SO_4….)
33 CCV @5.0/50ppm
34 CCB
35 Sample (Cl, SO_4….)
36 Sample (Cl, SO_4….)
37 Sample (@ 2X (SO_4)
38 CCV @ 5.0/50ppm
39 CCB
40 pumpoff

c. Save a schedule under File/Save as, using the date as the title of the Schedule.

d. When saved, exit out of Schedule Editor.

e. Load autosample cartridges in the same order as the scheduled run. After putting the cartridges in the autosampler, switch the autosampler to RUN using the Hold/Run button.

f. Assuming that the Pump is equilibrated with steady eluent baseline/uniform conductivity, enter into the RUN page.

g. Go to File to Open Method. Open correct method <date> of most recent calibration. This will begin pumping eluent at 1.0 mL/minute and turn on the SRS pump at 300 *u*amps voltage.

h. Next, go to file to Open Schedule. Open newly created schedule for the day.

i. When Method and Schedule are opened, go to Run and click on Start. The autosampler will inject into the first sample and the run should continue until completion.

X. POLLUTION PREVENTION

A. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

B. 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.

C. For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

XI. WASTE MANAGEMENT

A. The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Section 14.3 from method 300.1.

XII. CORRECTIVE ACTIONS

A. INSTRUMENT RELATED

1. ICV not within ± 10%

   a. If the problem is with the solution.

      i. Reprepare, obtain new stock if necessary.

   b. If the problem is with the calibration. Recalibrate thru analysis of appropriate standards and recheck ICV.

2. CCV not within ± 10%

   a. If the problem is with the solution.

      i. Reprepare, obtain new stock if necessary.

   b. If the problem is with the calibration.

      i. Recalibrate thru analysis of appropriate standards and reprepare/reanalyze the previous ten sample according the following guidelines.

         a. If the CCV was biased high, any of the previous ten samples which were BMDL do not require reanalysis.

         b. If the CCV was biased low, the previous ten samples must be reanalysed.

3. CCB not >± MDL (USACE)(**For DOD QSM Ver. 3 no analyte detected >2xMDL, frequency- beginning and ending a run and every 10 samples**)or ± RL or CRDL for others and CLP

   a. If the CCB is biased high.

      i. Any samples BDL or greater than 10X the CCB bias need not be reanalyzed.
      ii. Any samples above the detection limit but less than 10X the CCB level must be reanalyzed after the problem is corrected.

   b. If the CCB is biased low.

      i. Any samples greater than 10X the absolute CCB bias need not be reanalyzed.
      ii. All other samples must be reanalyzed after the problem is corrected.

4. LCS not within our in-house generated control limits (or ±10%).

   a. If the problem is with the instrument.

      i. Reanalyze when instrument is in control.

      ii. If biased high, the impact upon the data user must be evaluated. The samples will be re-extracted or the data will be qualified on the final report.

C. SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within ±20%

   i. The associated sample data must be qualified on the final report.

2. Spike analysis recovery not within ±20%

   i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.

   ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report.

XIII. SOURCES/REFERENCES:

1. Standard Methods for the Examination of Water and Wastewater, Method 4110B, "Anions by Ion Chromatography", 18th Edition of Standard Methods (1992).

2. Dionex, System DX500 Operation and Maintenance Manual, Dionex Corporation, Sunnyvale, California 94086, 1996.

3. Method Detection Limit (MDL) as described in "Trace Analyses for Wastewater," J. Glaser, D. Foerst, G. McKee, S. Quave, W. Budde, Environmental Science and Technology, Vol. 15, Number 12, page 1426, Decemvber, 1981.

4. American Society for Testing and Materials. Test Method for Anions in Water by Chemically – Suppressed Ion Chromatography D4327-91. Annual Book of Standards, Vo. 11.01 (1993).

5. Code of Federal Regulations 40, Ch. 1, Pt. 136, Appendix B; MDL determination.

6. Hautman, D.P. & Bolyard, M. Analysis of Oxyhalide Disinfection By-products and other Anions of Interest in Drinking Water by Ion Chromatography. Jour. Of Chromatog., 602, (1992), 65-74.

7. USEPA Methods 300.0; *Method for Determination of Inorganic Substances*(EPA/600/R-93/100) / *Method for the Determination of Organic and Inorganic Compounds in Drinking Water* (Vol. 1, EPA 815-R-00-014).

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

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: EPA 300.0 Anions by Ion Chromatography
Instrument is a Dionex DX-500 system. Equipped with Guard Column, Analytical Column, Conductivity Suppressor, Conductivity Detector, and Eluent Generator.

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 RLS meet control limits?				
6. Did LCS, Laboratory Fortified Blank or blank spike meet control limits?				
7. Did MS/MSD meet control limits?				
8. Was the Blank below the project required detection limits?				
9. Did you return samples back to cold storage immediately after use?				
10. Were samples analyzed for Nitrate done within the 48-hr holding time?				
11. Sample preparation information is correct and complete.				
12. Were all samples filtered through a 0.45*u*m filter?				
13. Analytical results are correct and complete.				
14. The appropriate SOP's have been used and followed.				
15. Raw data" including all manual integration's have been correctly interpreted.				
16. "Special" sample preparation and analytical requirements have been met.				

ANALYST DATA REVIEW CHECKLIST
EPA 300.0 (Anions by IC)

17. 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: _____

Second-Level Review: _____ Date: _____

Empirical Laboratories, LLC

ELECTROMETRIC DETERMINATION OF pH

METHODS
(USEPA) 150.1, Standard Methods 4500-H^+B AND 9040B FOR WATERS, LIQUIDS AND LIQUID WASTES, 9045C FOR SOILS AND SOIL WASTES

SOP NUMBER: **SOP-187**

REVISION NUMBER: **6**

APPROVED BY:

SECTION MANAGER

TECHNICAL DIRECTOR

EFFECTIVE DATE: **09/05/06**

DATE OF LAST REVIEW: **02/08/08**

Empirical Laboratories, LLC

ELECTROMETRIC DETERMINATION OF pH

I. SCOPE AND APPLICATION:

A. This SOP is specifically for the Orion 420A pH meter with Orion Triode pH electrode.

B. USEPA Method 150.1/SM4500-H^+B is applicable to drinking, surface, and saline waters, domestic and industrial wastes and acid rain.

C. Method 9040B is applicable to aqueous wastes and those multiphase waste where the aqueous phase constitutes at lease 20% of the total volume of the waste. This method also fulfills the requirement for characterization of Corrosivity according to SW846.

D. Method 9045C is applicable to soils and waste samples. Wastes may be solids, sludges, or non-aqueous liquids. If water is present, it must constitute less than 20% of the total volume of the sample.

II. METHOD SUMMARY:

The pH of a sample is determined electrometrically using either a glass electrode in combination with a reference potential or combination electrode.

III. SAMPLE HANDLING AND PRESERVATION:

A. pH, when used for permit reporting on water samples, is intended to be a field test. It is often monitored on a continuous basis when 24 hour composites are collected. If the pH is to be analyzed on a discreet grab sample, most States require that analysis occur within 15 minutes of collection to be reported without qualification for regulatory purposes. Nevertheless, occasionally, samples will be transported to the laboratory for analysis. Frequently this is done for the information purposes only, to be used by the client or the laboratory for the purposes other than regulation. To avoid confusion or misuse of the data, these analyses will clearly be labeled as "laboratory pH" on the final report. They must be analyzed as soon as possible in the laboratory. Soil or solid samples are subject to the sample holding time restrictions, and should also be analyzed as soon as possible and qualified on the final report.

B. High-purity waters and waters not at equilibrium with the atmosphere are subject to changes when exposed to the atmosphere, therefore the sample containers should be filled completely and kept sealed prior to analysis.

C. Samples must be unpreserved.

IV. INTERFERENCES:

A. The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants, or high salinity.

B. Sodium error at pH levels greater than 10 can be reduced or eliminated by using a "low sodium error" electrode.

C. Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gently wiping the electrode or washing it with a detergent followed by rinsing with distilled water. An additional rinsing with dilute Hydrochloric Acid (1:10) may also be necessary to remove any remaining film.

D. Temperature fluctuations will cause measurement errors, however pH measurements made with the Orion 420A meter and the Orion Triode electrode have automatic temperature compensation features.

E. A dirty electrode may also cause problems. See the maintenance sections or the electrode manual.

V. EQUIPMENT/APPARATUS:

A. pH meter: This SOP is specifically for the Orion 420A. This meter has set-up features which have been pre-set. In order to change these, refer to the instruction manual, pages 8-9. This meter has an automatic temperature compensation feature.

B. Electrode: There are many different types of pH electrodes available. This SOP is for the Orion Triode pH electrode which has an epoxy outer body and contains an internal filling solution of Ag/AgCl.

NOTE: REFER TO THE INSTRUCTION MANUAL FOR ANY QUESTIONS OR PROBLEMS WITH EITHER THE METER OR THE ELECTRODE. ALSO REFER TO THE INSTRUCTION MANUAL FOR PROPER CARE AND MAINTENANCE OF THE pH METER AND PROBE.

C. Magnetic stirrer and Teflon-coated stirring bars. Put a piece of cardboard between the stirrer and the beaker containing the sample so that the heat from the stirrer will not affect the temperature of the sample.

D. Small beakers or cups.

Empirical Laboratories, LLC

VI. REAGENTS:

A. Standard buffers: 4, 7, and 10. The pH meter must be calibrated using standard buffers daily or, if not daily, with each use. Separate solutions of the three buffers should be kept in 120 ml. plastic bottles with the caps on when not in use. Pour up fresh buffers each use. (Record the date in the log book).
An additional pH 12 buffer is required for the Corrosivity test of alkaline wastes per Method 9040B.

B. Reference electrode filling solution.

1. Use Orion #900011 which contains AgCl. Make sure that the filling solution is the correct one for your electrode. If you use the wrong solution you may damage the electrode.

2. Never allow the electrode to dry out.

3. Change the filling solution at least once a month. (Record the date in the log book). When you change the filling solution you should soak the electrode in pH 7 buffer for at least one hour before analyzing samples.

C. Deionized water.

D. Hydrochloric acid (HCl): 1:3 mixture with reagent water.

VII. CALIBRATION:

A. Initial calibration (2 point)

This meter is capable of both autocalibration and manual calibration. We calibrate manually.

1. Turn the power on. It will display a quick check mode and then the reading will stabilize. The meter should be in the pH mode, if not, press the "mode" key until the pH mode indicator is displayed.

2. Uncover the hole on the upper part of the electrode. This allows a uniform flow of the filling solution. (It should be kept covered when not in use.)

3. Rinse the electrode with deionized water and blot dry with a kimwipe (do not wipe the electrode).

4. The meter must be calibrated prior to use with two pH buffer standards which bracket the expected value of the sample to be

measured (4 and 7, or 7 and 10). Place the electrode in pH 7 buffer first, with stirring bar turning. Make sure the electrode is not touching the bottom or sides of the beaker. Buffers must be at room temperature.

5. Press the "2nd" key, followed by the "cal" key. It will display the time and date of the last calibration and then "P1" will be displayed in the lower field -- this means it is ready to read the first calibration standard.

6. Wait for a stable pH display and the meter will say "ready". Press the "no" key. The first digit will start flashing. Scroll up or down using the arrow-head keys until the correct value appears in the first digit (it will be 7 for the 7.00 buffer) then press "yes". Continue in the same manner until all the digits have been correctly entered, then press "yes" to enter the new value. The display will remain frozen for a few seconds, then "P2" will be displayed which means that the meter is ready to read the second calibration standard.

NOTE: Due to the temperature compensation of the meter, at 20° C the 7 buffer should read 7.01.

7. Rinse the electrode and place in the pH 4 **or** 10 buffer (depending on the expected pH of the sample to be analyzed) with stirring bar turning. Wait for a stable pH reading and then enter the correct value as you did for the 7 buffer.

NOTE: As with the 7 buffer, due to the temperature compensation of the meter, at 20° C the 4 buffer should read 4.00 and the 10 buffer should read 10.05.

8. After both points have been calibrated, the electrode slope will be displayed in the main field. The slope should be between 92 and 102. (Record the slope on the data sheet.) If it is not within this boundary you should inspect the electrode and meter and recalibrate with fresh buffers. If the slope is still out of range, check the manual for troubleshooting.

9. The meter will automatically advance to the "measure" mode and you are now ready to check the calibration and read the samples.

B. Calibration for Corrosivity characterization by Method 9040B

1. For corrosivity characterization, the calibration of the pH meter should include a buffer of pH 2 for acidic wastes and a pH 12 buffer for caustic wastes; also, for corrosivity characterization, the sample must be measured at 25±1 °C if the pH of the waste is above 12.0. Repeat adjustments on successive portions of the two buffer solutions until

readings are within 0.05 pH units of the buffer solution value. The specific steps to follow match the explanations in Section A.

C. Calibration Check

1. Rinse the electrode and place it in the 7 buffer. Read this buffer as a sample and not as a calibration standard. Press "measure". The display will start to fluctuate as the pH is being measured. Once the reading stabilizes the meter will beep and the display will freeze.

2. Record the reading. (It should be 7.00± 0.09. If it is not within this range, inspect the electrode and meter and recalibrate with fresh buffers. If the reading is still out of range, check the manual for troubleshooting.)

3. If the 7 buffer reads correctly, read the 4 buffer and 10 buffer. Record these readings. (The reading should not deviate from the true values by more than 0.09 units. If they are not within this range, inspect the electrode and meter and recalibrate with fresh buffers. If the reading is still out of range, check the manual for troubleshooting.)

VIII. PROCEDURE:

A. Water and Liquid Waste Samples (Non Drinking Water) by Methods 150.1 or 9040B

1. Sample(s) should be at room temperature when analyzed. The Triode electrode is capable of adjusting for temperature inconsistencies, however, you should still adjust the samples to room temperature before analyzing. (Record the temperature of **each** sample on the data sheet.)

2. Rinse the electrode with deionized water and blot it dry with a kimwipe. Place it into the sample with the stirring bar turning. Make sure that the electrode does not touch the sides or the bottom of the beaker.

3. The display will start to change. Once the reading stabilizes the meter will beep and the number will freeze. Record this number on the data sheet.

4. If you calibrated with 7 and 4 buffers, the sample pH must fall between 7 and 4. If the pH falls out of this range, you must go back and recalibrate using the 7 and 10 buffers and rerun the sample.

5. If you are determining Corrosivity by 9040B, use a pH 12 buffer to bracket the alkaline upper range.

6. Be sure you rinse off the electrode with deionized water in between samples.

B. Drinking Water and Acid-rain Samples by Method 150.1

Treat these samples exactly as you would other water samples except for the stirring. It is imperative that the stir plate is set on its' lowest setting and that the sample is not stirred vigorously. You should use the smallest stir bar possible. Make sure that you do not aerate the sample.

C. Soil Samples by Method 9045C

1. To 20 g of soil in a 50-mL beaker, add 20 mL of reagent water, cover, and continuously stir the suspension for 5 minutes. Additional dilutions are allowed if working with hygroscopic soils and salts of other problematic matrices.

2. Let the soil suspension stand for about 1 hour to allow most of the suspended clay to settle out from the suspension or, if necessary, filter or centrifuge off the aqueous phase for pH measurement.

3. Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant solution to establish a good electrical contact through the ground-glass joint.

4. Solutions should be a room temperature when analyzed. The Triode electrode is capable of adjusting for temperature inconsistencies however, you should still adjust the samples to room temperature before analyzing. (Record the temperature of **each** solution on the data sheet.)

5. Rinse the electrode with deionized water and blot it dry with a kimwipe. Place it into the solution as described in step 3.

6. The display will start to change. Once the reading stabilizes the meter will beep and the number will freeze. Record this number on the data sheet.

7. If you calibrated with 7 and 4 buffers, the sample pH must fall between 7 and 4. If the pH falls out of this range, you must go back and recalibrate using the 7 and 10 buffers and rerun the sample.

8. Be sure you rinse off the electrode with deionized water in between samples.

B. Solid Waste Samples by Method 9045C

1. To 20 g of waste sample in a 50-mL beaker, add 20 mL of reagent water, cover and continuously stir the suspension for 5 minutes. Additional dilutions are allowed if working with hydroscopic wastes and salts or other problematic matrices.

2. Let the waste suspension stand for about 15 minutes to allow most of the suspended waste to settle out from the suspension or, if necessary, filter or centrifuge off aqueous phase for pH measurement.

NOTE: If the waste is hygroscopic and absorbs all the reagent water, begin the experiment again using 20 g of waste and 40 mL of reagent water.

NOTE: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous phase. The electrode may need to be cleaned (Step IV.C) if it becomes coated with an oily material.

3. Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant solution to establish a good electrical contact through the ground-glass joint.

4. Solutions should be a room temperature when analyzed. The Triode electrode is capable of adjusting for temperature inconsistencies, however, you should still adjust the samples to room temperature before analyzing. (Record the temperature of **each** solution on the data sheet.)

5. Rinse the electrode with deionized water and blot it dry with a kimwipe. Place it into the solution as described in step 3.

6. The display will start to change. Once the reading stabilizes the meter will beep and the number will freeze. Record this number on the data sheet.

7. If you calibrated with 7 and 4 buffers, the sample pH must fall between 7 and 4. If the pH falls out of this range, you must go back and recalibrate using the 7 and 10 buffers and rerun the sample.

8. Be sure you rinse off the electrode with deionized water in between samples.

IX. ELECTRODE STORAGE AND MAINTENANCE:

A. Storage

1. Store the electrode in a solution made up of 1 gram KCl or NaCl in 200 mL of pH 7 buffer. This solution may be purchased from Fisher.

2. The reference chamber hole should be covered overnight and the power to the pH meter should be turned off.

3. White crystals will probably form on the electrode. These are from the potassium chloride and will not damage the electrode, however, the electrode should be rinsed with distilled water to get rid of this build-up.

4. If storing the electrode for more than one week, the reference chamber should be filled and the fill hole should be securely covered. Cover the sensing element with its protective cap containing a few drops of storage solution. Before reusing it, prepare it as a new electrode. (See manual).

B. Maintenance

1. Drain the reference chamber, flush it with fresh filling solution and refill the chamber. Allow the electrode to set overnight before using it. This should be done on a monthly basis. Record the date in the log book.

2. Inspect the electrode for scratches, cracks, salt crystal build-up, and general physical integrity.

3. If there is build-up of salt or any grease or dirt on the electrode it may be necessary to soak it in dilute HCl (1:10) for half an hour. Then drain and refill the reference chamber and soak the electrode in storage solution for at least half an hour before using. Also record this in the log book.

For more information on storage and maintenance of the electrode, see the instruction manual.

X. QUALITY CONTROL:

A. Analyze a laboratory control sample (LCS) or second-source standard once a month or every 300 measurements, whichever is more frequent. The tolerance range is ± 0.25 units.

B. Run a duplicate every 10 samples (count frequence per method and matrix). The specification limit is ± 0.09 units. If exceeded, repeat the measurement once or twice more. If the specification is still not met, you must recalibrate and reanalyze the sample in question.

C. Read one of the buffers every 20 samples (all methods and matrices included to count frequency), or at the beginning and end of the

measurement session; whichever is more frequent. If it does not read its true value ± 0.09 units, then you must recalibrate.

D. Record the QC data (LCSs and duplicates) on the QC sheet. Make sure that this information is written legibly and is complete, including specification of the method number.

E. Electrodes must be rinsed thoroughly between samples.

F. Document any problem experience during the measurements. Complete a corrective action where necessary. This must be done within 24 hours of occurrence of the incident.

G. Refer to SOP-431 for common environmental laboratory definitions.

XI. REFERENCES

A. USEPA Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983, Method 150.1.
B. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Methods 9040B and 9045C.
C. Standard Methods 18th Edition 1992 p. 4-65.

XII. WASTE MANAGEMENT AND POLLUTION PREVENTION

A. Please see Waste Disposal SOP-405 for instruction of proper disposal of waste generated from this area.

B. 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.

Empirical Laboratories, LLC

TOXICITY CHARACTERISTIC LEACHING PROCEDURE

METHOD 1311

SOP NUMBER: **SOP-198**

REVISION NUMBER: **6**

APPROVED BY:

SECTION MANAGER

TECHNICAL DIRECTOR

EFFECTIVE DATE: **12/22/08**

DATE OF LAST REVIEW: **12/22/08**

Empirical Laboratories, LLC

METHOD 1311

TOXICITY CHARACTERISTIC LEACHING PROCEDURE

1.0 SCOPE AND APPLICATION

1.1 The TCLP is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

1.2 If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.

2.0 SUMMARY OF METHOD

2.1 For a highly liquid waste, determine if the percent dry solids is less than 0.5%. If so, the sample is filtered and the filtrate is treated as the extraction fluid. If not, the sample is reduced in size to pass through a 9.5 mm sieve. The percent wet solids are then determined. A 5 g and 100 g aliquot of wet solids are generated. The liquid phase for the 100 g aliquot is stored at 4 degrees Celsius. The 5 g aliquot is used to determine the appropriate extraction fluid. The 100 g aliquot is added to the extraction fluid and tumbled for about 18 hr's. The sample is filtered and the pH of the fluid is determined. An appropriate amount of the liquid phase is added to the extraction fluid and the sample is preserved according to the test, which will be performed.

2.2 The TCLP extract for volatiles is analyzed by method 8260B according to SOP ATSD-202. The TCLP extract for semivolatiles is subject to separatory funnel extraction by method 3510 according to SOP ATSD-300. The TCLP extract for metals is divided for two separate digestions and analyses: (a) mercury digestion and analysis by 7470A (ATSD-103), and (b) ICP-metals digestion by 3010A (ATSD-100) and analysis by 6010B (ATSD-116).

3.0 INTERFERENCES

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods and related SOPs. (See section 2.2)

4.0 APPARATUS AND MATERIALS

4.1 Agitation apparatus: The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 $\pm$ 2 rpm. This rate is measured and recorded with each extraction or at least once per quarter to ensure accuracy of the equipment.

4.2 Extraction Vessels

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4.2.1 Zero-Headspace Extraction Vessel (ZHE). The ZHE (depicted in Figure 2) allows for liquid/solid separation within the device, and effectively precludes headspace. This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel. The vessels shall have an internal volume of 500-600 mL, and be equipped to accommodate a 90-110 mm filter. The devices contain VITON®[1] O-rings which should be replaced frequently.

For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 30 to 40 psi or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses and the manufacturer should be contacted.

The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psi, allow it to stand unattended for 10 minutes, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psi, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted.

Some ZHEs use gas pressure to actuate the ZHE piston, while others use mechanical pressure. Whereas the volatiles procedure refers to pounds per square inch (psi), for the mechanically actuated piston, the pressure applied is measured in torque-inch-pounds.

4.2.2 Bottle Extraction Vessel. When the waste is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel.

The extraction bottles may be constructed from various materials, depending on the analytes to be analyzed and the nature of the waste. It is recommended that borosilicate glass bottles be used instead of other types of glass, especially when inorganics are of concern. Plastic bottles, other than polytetrafluoroethylene, shall not be used if organics are to be investigated.

4.3 Filtration Devices: It is recommended that all filtrations be performed in a hood or well ventilated area.

4.3.1 Zero-Headspace Extractor Vessel (ZHE): When the waste is evaluated for volatiles, the zero-headspace extraction vessel is used for filtration. The device shall be capable of supporting and keeping in place the glass fiber filter and be able to withstand the pressure needed to accomplish separation (50 psi).

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[1] VITON® is a trademark of Du Pont.

NOTE: When it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the material within the ZHE.

4.3.2 Filter Holder: When the waste is evaluated for other than volatile analytes, any filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation may be used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressures of up to 50 psi or more. Vacuum filtration can only be used for wastes with low solids content (<10%) and for highly granular, liquid-containing wastes. All other types of wastes should be filtered using positive pressure filtration.

4.3.3 Materials of Construction: Extraction vessels and filtration devices shall be made of inert materials which will not leach or absorb waste components. Glass, polytetrafluoroethylene (PTFE), or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components.

4.4 Filters: Filters shall be made of borosilicate glass fiber, shall contain no binder materials, and shall have an effective pore size of 0.6 to 0.8 µm (Whatman GF/F), or equivalent. **Pre-filters must not be used. When evaluating the mobility of metals**, filters shall be acid-washed prior to use by rinsing with 1N nitric acid followed by three consecutive rinses with deionized distilled water (a minimum of 1 L per rinse is recommended). Glass fiber filters are fragile and should be handled with care.

4.5 pH Meters: The meter should be accurate to ± 0.20.

4.6 ZHE Extract Collection Devices: TEDLAR®[2] bags are used to collect the initial liquid phase and the final extract of the waste when using the ZHE device, only when both liquid and solid phases exist. Collect final extract in a 40 mL vial when only a solid phase exist.

4.7 ZHE Extraction Fluid Transfer Devices: Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a positive displacement or peristaltic pump, a gas tight syringe, pressure filtration unit (see Section 4.3.2), or other ZHE device).

4.8 Laboratory Balance: Accurate to within ± 0.01 grams may be used (all weight measurements are to be within ± 0.1 grams).

4.9 Beaker or Erlenmeyer flask, glass, various sizes.

4.10 Watchglass

[2] TEDLAR® is a registered trademark of Du Pont.

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4.11 Magnetic stirrer.

5.0 REAGENTS

5.1 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.

5.2 Reagent Water. Reagent water is defined as water in which an interferant is not observed at or above the method's detection limit of the analyte(s) of interest. For nonvolatile extractions, ASTM Type II water or equivalent meets the definition of reagent water. For volatile extractions, it is recommended that reagent water be generated by any of the following methods.

5.2.1 A water purification system may also be used to generate reagent water for volatile extractions.

5.2.2 Reagent water for volatile extractions may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the water temperature at 90 $\pm$ 5 degrees C, bubble a contaminant-free inert gas (e.g. nitrogen) through the water for 1 hour. While still hot, transfer the water to a narrow mouth screw-cap bottle under zero-headspace and seal with a Teflon-lined septum and cap.

5.3 Hydrochloric acid (1N), HCl, made from ACS reagent grade.

5.4 Nitric acid (1N), HNO_3, made from ACS reagent grade.

5.5 Sodium hydroxide (15N), NaOH, made from ACS reagent grade.

5.6 Glacial acetic acid, CH_3CH_2COOH, ACS reagent grade.

5.7 Extraction fluid.

5.7.1 Extraction fluid #1: Add 11.4 mL glacial acetic acid to approximately 1800 mL DI water. Add 8.6 mL 15N sodium hydroxide and dilute to 2 L. Seal and invert four times. Determine the pH which should be 4.93 +/- 0.05. Record in appropriate reagent log (RL) notebook.

5.7.2 Extraction fluid #2: Add 11.4 mL glacial acetic acid to approximately 1800 mL DI water and dilute to 2 L. Seal and invert four times. Determine the pH which should be 2.88 +/- 0.05. Record in appropriate reagent log (RL) notebook.

NOTE: All extraction fluids are purchased "certified" or prepared fresh and in volume quantity that matches the number of TCLP extractions to be performed on a given day. The TCLP/SPLP log-book is used to record the fluid type of choice with indication of its traceability to the lot numbers of acetic acid and sodium hydroxide used.

5.8 Analytical standards shall be prepared according to the appropriate analytical method.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Extreme care must be taken when taking samples. Samples should be collected using an appropriate sampling plan.

6.2 The TCLP may place requirements on the minimal size of the field sample, depending upon the physical state or states of the waste and the analytes of concern. An aliquot is needed for preliminary evaluation of which extraction fluid is to be used for the nonvolatile analyte extraction procedure. Another aliquot may be needed to actually conduct the nonvolatile extraction. If volatile organics are of concern, another aliquot may be needed.

6.3 Preservatives will not be added to samples before TCLP extraction.

6.4 For volatiles, the samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon-lined septum capped vials and stored at 4°C. Samples should be opened only immediately prior to TCLP extraction).

6.5 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. **Extracts must be spiked according to their prospective analytes before preservation. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH < 2, unless precipitation occurs (see Section 7.2.14 if precipitation occurs).** Extracts or portions of extracts for volatile analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses. Preserve according to the analytical methods as referenced in 2.2.

7.0 PROCEDURE

7.1 Preliminary Evaluations

Perform preliminary evaluations on a minimum 100 gram aliquot of waste. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include: (1) determination of the percent solids; (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration; (3) determination of whether the solid portion of the waste requires particle size reduction; and (4) determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the waste.

7.1.1 Percent wet solids: Percent wet solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure.

7.1.1.1 If the sample will obviously yield no liquid when subjected to pressure filtration proceed to Section 7.1.3.

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7.1.1.2 If the sample is liquid or has more than one phase, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device described in Section 4.3.2 and is outlined in Sections 7.1.1.3 through 7.1.1.9.

7.1.1.3 Pre-weigh the filter.

7.1.1.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the weighed filter in the buchner funnel.

7.1.1.5 Mix the sample and weigh out a representative subsample of (100 gram minimum) and record exact weight.

7.1.1.6 Allow sludges to stand to permit the solid phase to settle.

7.1.1.7 Quantitatively transfer the sample to the filter holder (liquid and solid phases). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature then allow the sample to warm up to room temperature in the device before filtering.

NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Section 7.1.1.5 to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

7.1.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, this material may not filter. If this is the case, the material

within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances.

7.1.1.9 Remove filter carefully from the buchner funnel so as to get as much of the solid as possible.

Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{Percent wet solids} = \frac{(\text{Filter wt.} + \text{sample wt.}) - \text{filter wt.}}{\text{Total weight of waste}} \times 100$$

7.1.2 If the percent solids is equal to or greater than 0.5%, then proceed either to Section 7.1.3 to determine whether the solid material requires particle size reduction or to Section 7.1.2.1 if it is noticed that a small amount of the filtrate is entrained in wetting of the filter. If the percent solids is less than 0.5%, then proceed to Section 7.2.9 if the nonvolatile TCLP is to be performed and to Section 7.3 with a fresh portion of the waste if the volatile TCLP is to be performed.

7.1.2.1 Remove the solid material and filter from the filtration apparatus.

7.1.2.2 Dry the filter and solid material at 100 $\pm$ 20°C. Record the final weight.

NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

7.1.2.3 Calculate the percent dry solids as follows:

$$\text{Percent dry solids} = \frac{(\text{Wt. of dry waste} + \text{filter}) - \text{tared wt. of filter}}{\text{Initial wt. of waste}} \times 100$$

7.1.2.4 If the percent dry solids is less than 0.5%, then proceed to Section 7.2.9 if the nonvolatile TCLP is to be performed, and to Section 7.3 if the volatile TCLP is to be performed. If the percent dry solids is greater than or equal to 0.5%, and if the nonvolatile TCLP is to be performed, return to the beginning of this Section (7.1) and, with a fresh portion of waste, determine whether particle size reduction is necessary (Section 7.1.3) and determine the appropriate extraction fluid (Section 7.1.4). If only the volatile TCLP is to be performed, see the note in Section 7.1.4.

7.1.3 Determination of whether the waste requires particle size reduction: Using the solid portion of the sample, evaluate the solid for particle size. Particle size reduction is required, unless the solid passes through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the

waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above. If the solids are prepared for organic volatiles extraction, special precautions must be taken.

NOTE: Surface area criteria are meant for filamentous (e.g., paper, cloth, and similar) waste materials. Actual measurement of surface area is not required, nor is it recommended.

7.1.4 Determination of appropriate extraction fluid: If the solid content of the waste is greater than or equal to 0.5% and if the sample will be extracted for nonvolatile constituents, determine the appropriate fluid for the nonvolatiles extraction as follows:

NOTE: TCLP extraction for volatile constituents uses only extraction fluid #1. Therefore, if TCLP extraction for nonvolatiles is not required, proceed to Section 7.3.

7.1.4.1 Weigh out a small subsample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 250 mL beaker or Erlenmeyer flask.

7.1.4.2 Add 96.5 mL of reagent water to the beaker, cover with a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use extraction fluid #1. Proceed to Section 7.2.

7.1.4.3 If the pH is >5.0, add 3.5 mL 1N HCl, **slurry briefly**, cover with a watchglass, heat to 50°C, and hold at 50°C for 10 minutes. (Note: Do not mix the sample during heating). The hot block may be used for the purpose of heating and holding at 50°C for 10 minutes.

7.1.4.4 Let the solution cool to room temperature (use Hobart to aid in cooling, 30 to 60 minutes) and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2. Note: The pH must be measured as quickly as possible in the phase of the procedure. It is critical to measure the pH as soon as the sample has reached room temperature. Proceed to Section 7.2.

7.1.5 If the aliquot of the waste used for the preliminary evaluation was determined to be 100% solid, then it can be used for the Section 7.2 extraction (assuming at least 100 grams remain), and the Section 7.3 extraction (assuming at least 25 grams remain).

7.2 Procedure for Metals and Semi-Volatiles

A minimum sample size of 100 grams (solid and liquid phases) is recommended. If less than the specified amount is used, proportion extraction fluid accordingly and notify manager. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample, whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes

of concern. Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to support all of the analyses required.

All glassware and equipment must be thoroughly cleaned before use. A hot soapy water wash, rinse with tap water, then repeat following with a DI water rinse. Rinse with 1:1 HNO_3 , acetone, and a final rinse with DI water.

7.2.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solid), weigh out a subsample of the waste (100 gram minimum) and proceed to Section 7.2.9.

7.2.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device described in Section 4.3.2 and is outlined in Sections 7.2.4 to 7.2.8.

7.2.3 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (see Section 4.4).

7.2.4 Weigh out a subsample of the waste (100 gram minimum) and record the weight. If the waste contains <0.5% dry solids, the liquid portion, after filtration, is defined as the TCLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required. For wastes containing >0.5% dry solids use the percent solids information to determine the optimum sample size (100 gram minimum) for filtration.

7.2.5 Allow sludges to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If the waste is centrifuged, the liquid should be decanted and filtered, followed by filtration of the solid portion of the waste through the same filtration system.

7.2.6 Refer to Section 7.1.1.7.

7.2.7 Refer to Section 7.1.1.8.

7.2.8 If the waste contains <0.5% dry solids, proceed to Section 7.2.13. If the waste contains >0.5% dry solids, and if particle size reduction of the solid was needed in Section 7.1.3, proceed to Section 7.2.10. If the waste as received passes a 9.5 mm sieve, quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase, and proceed to Section 7.2.11.

7.2.9 Prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 7.1.3. When the surface area or particle size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

NOTE: Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon coated sieve should be used to avoid contamination of the sample.

7.2.10 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{20 \text{ x percent wet solids x weight of waste fltered}}{100}$$

Slowly add this amount of appropriate extraction fluid to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30 $\pm$ 2 rpm for 18 $\pm$ 2 hours. Ambient temperature shall be maintained at 23 $\pm$ 2 □C during the extraction period. Record both tumble rate and room temperature.

NOTE: As rotation continues, pressure may build up within the extractor bottle for some types of wastes. To relieve excess pressure, the extractor bottle should be periodically opened (e.g., after 15 minutes, and 30 minutes) and vented into a hood.

7.2.11 Following the 18 $\pm$ 2 hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in Section 7.2.7. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed.

7.2.12 Prepare the TCLP extract as follows:

7.2.12.1 If the sample contained no initial liquid phase, the filtered liquid material obtained is defined as the TCLP extract. Proceed to Section 7.2.13.

7.2.12.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Section 7.2.12 with the initial liquid phase of the waste obtained in Section 7.2.7. This combined liquid is defined as the TCLP extract. Proceed to Section 7.2.13.

7.2.12.3 If the initial liquid phase of the waste, as obtained from Section 7.2.7, is not or may not be compatible with the filtered liquid resulting from Section 7.2.11, do not combine these liquids. Analyze these liquids, collectively defined as the

TCLP extract, and combine the results mathematically, as described in Section 7.2.14.

7.2.1.3 Following collection of the TCLP extract, the pH of the extract must be recorded. Immediately split aliquots, give to appropriate analyst for spiking and preservation of the extract for analysis. Metal aliquots must be acidified with nitric acid to pH <2. (If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible.) All other aliquots must be stored under refrigeration (4°C) until analyzed. TCLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. Refer also to sections 2.2 and 6.5.

If the individual phases are to be analyzed separately, determine the volume of the individual phases (to $\pm$ 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phase (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = On next page
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.3 Volatiles Extraction Procedure

Use ZHE device to obtain TCLP extract for analysis of volatile compounds only.

The ZHE device has approximately a 500 mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase.

Charge the ZHE with sample only once and do not open the device until the final extract has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4°C) to minimize loss of volatiles.

All glassware and equipment must be thoroughly cleaned before use. A hot soapy water wash, rinse with tap water, then repeat following with a DI water rinse. Rinse with methanol and a final rinse with DI water.

7.3.1 Pre-weigh the filtrate collection container and set aside. If using a TEDLAR® bag, express all liquid from the ZHE device into the bag, whether for the initial or final liquid/solid separation, and take an aliquot from the liquid in the bag for analysis.

7.3.2 Place the ZHE piston within the body of the ZHE (it may be helpful first to moisten the piston O-rings slightly with extraction fluid). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample (based upon sample size requirements determined from Section 7.3, Section 7.1.1 and/or 7.1.2). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

7.3.3 If the waste is 100% solid, weigh out a subsample (25 gram maximum) of the waste, record weight, and proceed to Section 7.3.5.

7.3.4 If the waste contains < 0.5% dry solids, the liquid portion of waste, after filtration, is defined as the TCLP extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For wastes containing $\geq$ 0.5% dry solids, use the percent solids information determine the optimum sample size to charge into the ZHE.

The recommended sample size is as follows:

7.3.4.1 For wastes containing < 5% solids, weigh out a 500 gram subsample of waste and record the weight.

7.3.4.2 For wastes containing $\geq$ 5% solids, determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge ZHE} = \frac{25}{\text{percent wet solids}} \times 100$$

7.3.5 If particle size reduction of the solid portion of the waste was required in Section 7.1.3, proceed to Section 7.3.6. If particle size reduction was not required in Section 7.1.3, proceed to Section 7.3.7.

7.3.6 Prepare the waste for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle size as described in Section 7.1.3. The means used to effect particle size reduction must not generate heat in and of itself. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

NOTE: Sieving of the waste is not recommended due to the possibility that volatiles may be lost. The use of an appropriately graduated ruler is recommended as an acceptable alternative. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended.

When the surface area or particle size has been appropriately altered, proceed to Section 7.3.7.

7.3.7 Waste sludges need not be allowed to stand to permit the solid phase to settle. Do not centrifuge wastes prior to filtration.

7.3.8 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Section 7.3.4 to determine the weight of the waste sample that will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more if necessary) to force all headspace slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. If the waste is 100% solid (see Section 7.1.1), slowly increase the pressure to a maximum of 50 psi to force most of the headspace out of the device and proceed to Section 7.3.12.

7.3.9 Attach the pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10 psi to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2 minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

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7.3.10 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

(Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the TCLP extraction as a solid.)

If the original waste contained <0.5% dry solids, this filtrate is defined as the TCLP extract. Proceed to Section 7.3.15.

7.3.11 The liquid phase may now be either analyzed immediately or preserved and stored at 4°C under minimal headspace conditions until time of analysis. Determine the weight of extraction fluid #1 to add to the ZHE as follows:

$$\text{Weight of extraction fluid} = \frac{20 \text{ x percent wet solids x weight of waste filtered}}{100}$$

7.3.12 The following details how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #1 is used in all cases.

7.3.12.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be preflushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.

7.3.12.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10 psi (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psi and check all ZHE fittings to ensure that they are closed.

7.3.12.3 Place the ZHE in the rotary agitation apparatus and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction

occurs) shall be maintained at 23 ± 2°C during agitation. Record both tumble rate and temperature.

7.3.13 Following the 18 ± 2 hour period, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (i.e., no gas release observed), the device is leaking. Check the ZHE for leaking as specified in Section 4.2.1, and perform the extraction again with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (i.e., TEDLAR® bag or VOC vials) holding the initial liquid phase of the waste. A separate filtrate collection container must be used if combining would create multiple phases, or there is not enough volume left within the filtrate collection container. Filter through the glass fiber filter, using the ZHE device as discussed in Section 7.3.9. All extract shall be filtered and collected if the TEDLAR® bag is used, if the extract is multiphasic, or if the waste contained an initial liquid phase.

NOTE: An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

7.3.14 If the original waste contained no initial liquid phase, the filtered liquid material obtained from above is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained from above and the initial liquid phase (Section 7.3.9) are collectively defined as the TCLP extract.

7.3.15 Following collection of the TCLP extract, immediately prepare the extract for analysis (one of the vials will be spiked at this point) and store with minimal headspace at 4°C. Analyze the TCLP extract according to the appropriate analytical methods (refer to section 2.2). If the individual phases are to be analyzed separately (i.e., are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phases (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

8.0 QUALITY ASSURANCE

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8.1 A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel. No bias correction is to be taken into consideration (Fr 57, 227).

8.2 A matrix spike shall be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.). One matrix spike must be analyzed for each analytical batch. Follow the matrix spike addition guidance provided in each analytical method. (The method and SOPs are referenced in section 2.2.)

8.2.1 Matrix spikes are to be added after filtration of the TCLP extract and before preservation.

8.2.2 The matrix spikes should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be not less than ten times the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same volume of TCLP extract as that which was analyzed for the unspiked sample.

8.2.3 The matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. If interferences exist, an alternate approach may be required.

8.2.4 Matrix spike recoveries are calculated by the following formula:

$$\%R\ (\%Recovery) = 100\ (X_s - X_u)/K$$

where:

X_s = measured value for the spiked sample,
X_u = measured value for the unspiked sample, and
K = known value of the spike in the sample.

8.3 All quality control measures described in each analytical methods must be followed.

8.4 The use of method of standard addition (MSA) shall be employed for a metallic contaminant if: (1) Recovery of the contaminant from the TCLP extract is not at least 50% and the concentration does not exceed the regulatory level, and (2) The concentration of the contaminant measured in the extract is within 20% of the appropriate regulatory level.

8.4.1 The MSA requires preparing calibration standards in the sample matrix rather than reagent water. It requires taking four identical aliquots of the solution and adding known amounts of standard to three of these aliquots. The forth aliquot is the unknown. The first addition should be prepared so that the resulting concentration is approximately 50% of the expected concentration of the sample. The second and third additions should be prepared so that the concentrations are approximately 100% and 150% of the expected concentration of the sample. All four aliquots are maintained at the same final volume by adding reagent water. Analyze all four aliquots.

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8.4.3 Prepare a plot, or subject data to linear regression, of instrument signals or external-calibration-derived concentrations as the dependant variable (y-axis) versus concentrations of the additions of standard as the independent variable (x-axis). Solve for the intercept of the abscissa (the independent variable, x-axis) which is the concentration in the unknown.

8.5 TCLP extraction holding times.

SAMPLE MAXIMUM HOLDING TIMES [DAYS]				
	From: Field collection To: TCLP extraction	From: TCLP extraction To: Preparative extraction	From: Preparative extraction To: Determinative analysis	Total elapsed time
Volatiles	14	NA	14	28
Semi-volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except mercury	180	NA	180	360

NA = Not applicable

If holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Of course, exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

GC/MS SEMIVOLATILES
BY EPA METHOD 625 AND
SW846 METHOD 8270C AND 8270D
INCLUDING ADDITIONAL
APPENDIX IX COMPOUNDS

SOP NUMBER: **SOP-201**

REVISION NUMBER: **18**

APPROVED BY:

SECTION MANAGER

QUALITY ASSURANCE OFFICER

EFFECTIVE DATE: **09/16/08**

DATE OF LAST REVIEW **09/16/08**

GC/MS SEMIVOLATILES

BY EPA METHOD 625 AND SW846 METHOD 8270C and 8270D

1.0 SCOPE AND APPLICATION

Please see Appendix for definitions.
This SOP (based primarily on SW-846 Method 8000B/8270C/8270D) is used for the analysis of semi-volatile organic compounds in a variety of matrices (soils, sediments, waters, etc.). Methods *Federal Register* Method 625 and CLP Method for Semi-volatiles have also been used in the development of this SOP. The analyses by these various methods are clearly defined in the respective regulatory manuals. A good understanding of these different methods is essential to the performance of each method. A laboratory list of 625 and 8270 analytes with example limits is found attached in the appendix. Other compounds may be analyzed by this SOP as detailed in section 1.0 of SW-846 Method 8270C. Any questions left by this SOP should be answered by reading the methods, paying close attention to SW-846 8000B/8270C/8270D, EPA 625 and CLP. If questions still remain unanswered, check with the Organic Lab Manager, QA/QC Officer and/or Technical Director.

2.0 METHOD SUMMARY

After sample preparation using the appropriate extraction technique, the sample is introduced into the GC/MS using direct injection. The analytes are separated in the gas chromatograph by a combination of the temperature program, the pressure program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra from the sample. Analytes are quantitated relative to known standards using the internal standard method.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Section 3.0 and tables 3-1 and 3-2 of the Empirical Laboratories' Quality Assurance Manual include details concerning sample preservation, containers and handling of semi-volatile samples and extracts. All water and soil samples are stored in the appropriate walk-in coolers at a temperature of 4°C. All extracts are stored in the Hobart in the Extraction lab at a temperature of 4°C. Water samples have a holding time of 7 days from date of sampling. Soil samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project).Extracts have 40 days from date of extraction to be analyzed

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

4.1 All raw data (samples & QC) must be evaluated for interferences. If contamination occurs, determine whether the source of interference is in the preparation or clean-up of the samples and take corrective action to eliminate the problem.

4.2 Contamination by carryover can occur when samples of high-concentration and low-concentration are analyzed sequentially. To reduce carryover, the sample syringe must be rinsed with solvent between injections. If an unusually high sample is detected, a solvent blank should be analyzed for cross contamination.

5.0. EQUIPMENT AND APPARATUS

5.1 HP 5890/6890/7890GC complete with electronic pressure control and temperature programmable gas chromatograph suitable for splitless injection.

5.2 Column: RTX-5MS (or equivalent) 30 m x 0.25 mm I.D. x 0.25 µm film thickness fused silica capillary column or RTX-5 SIL-MS 30 m x 0.28 mm I.D. x 0.5µm film thickness.

5.3 HP 5971/5973/5975 mass spectrometer capable of scanning from 35 to 500 amu every second or less, using 70 volts electron energy in electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for decafluorotriphenylphosphine, DFTPP, which meets all the tuning criteria of the EPA methods.

5.4 HP 7673/7683 autosampler capable of reproducibility from one injection to another proven by meeting QC and calibration criteria.

5.5 HP GC/MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.

5.6 Acquisition Software: HP Chemstation system is interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.

5.7 Data Processing Software: Target DB on Windows NT server data system is interfaced to the HP Chemstation. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances in any EICP between specified time or scan-number limits. The most recent NBS mass spectral library is installed.

6.0 REAGENTS

6.1 Methylene chloride **(Please read SOP-336 before handling this solvent in our laboratory.)** – Trace analysis grade.

6.2 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label and recorded in the BNA standards log book. The date they are opened is noted on the label and recorded in the BNA standards log book along with their lot number and vendor. Each standard that is prepared is recorded in the BNA standards log book and given a sequential number. Each standards label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. All stocks and standards are stored in the freezer at a temperature of -15°C ± 5°C from the date they are received/prepared. Standards are brought to room temperature before being used to make standards. Sonication is used if precipitation is observed after bringing to room temperature. The refrigerator and freezer temperature is monitored daily with an annually calibrated thermometer and recorded with calibration correction in the Extraction temperature/calibration logbook. Makeup of common standards is detailed below. See the BNA standards log book for makeup of other standards.

6.2.1 The Decafluorotriphenylphosphine (DFTPP) tuning standard is prepared as follows (includes benzidine, pentachlorophenol and 4,4'-DDT): Using a 100µL syringe, 100µL (GCM-150, Ultra Scientific @ 1000µg/mL, or equivalent) is injected into a 2.0mL volumetric flask containing approximately 1.2mL methylene chloride (Trace Grade) and diluted to volume with same making a 50µg/mL standard. After capping and inverting several times, the solution is transferred into 2 labeled 2ml, teflon-lined, screw-capped vials and stored in the freezer at -15°C ± 5°C for up to 6 months. A direct injection of 1.0µL is used to tune the instrument.

6.2.2 Calibration standards are prepared from a 200µg/mL working standard at a minimum of five concentrations. Calibration standards are prepared semi-annually unless the initial calibration verification standard indicates a problem. To makeup the 200µg/mL working standard inject the indicated amount of the following standards (or equivalent) into a 10mL volumetric containing approximately 5mL methylene chloride (Trace Grade) and dilute to volume with the same. After capping and inverting several times, the solution is transferred into an appropriate labeled vial, teflon-lined, screw-capped vial and stored in the freezer at -15°C ± 5°C for up to 6 months. See the Appendix for analytes contained in these mixes.

Standard (Interm.A in 10mls)	Conc (µg/mL)	Amount(µL)	Final (ug/mL)
Base/Neutrals Mix 1 (Vendor,Lot#)	2000	1000	200
Benzidines Mix 2 (Vendor,Lot#)	2000	1000	200
Acids Mix (Vendor,Lot#)	2000	1000	200
SV Mix w/ Pyridine & Carbazole (Vendor,Lot#)	2000	1000	200
BN Surrogate (Vendor,Lot#)	5000	400	200
Acid Surrogate (Vendor,Lot#)	10,000	400	400
Catechol (Standard#)	10,000	200	200
Biphenyl (Standard#)	10,000	200	200
Caprolactum (Standard#)	10,000	200	200
Acetophenone (Standard#)	10,000	200	200
Standard (Interm.B in 5mls)	**Conc (µg/mL)**	**Amount(µL)**	**Final (ug/mL)**
Benzidine (Mix # 2)	2000	500	200
Standard (Interm.C in 5mls)			
Atrazine (Standard#)	10,310	97	200
Benzaldehyde (Standard#)	10,590	94	200

Additional Appendix IX compounds can be added using 3 additional mixes, AppIX A,B,C. Refer to standards log.

To makeup the calibration standards, using a 1ml syringe add the appropriate amount of methylene chloride (trace grade) to a 2ml vial. Add the indicated amount of each intermediate standard to the vial. Add 20ul of internal standard to each screw-capped vial and stored in the freezer at -15°C ± 5°C for up to 6 months.

IntmA,Bor C	Amt. of 200ppm std	Amount of 2000ppm Internal Std
2 µg/mL	10 µL	20 µL
5 µg/mL	25 µL	20 µL
10 µg/mL	50 µL	20 µL
20 µg/mL	100 µL	20 µL
30 µg/mL	150 µL	20 µL
40 µg/mL	200 µL	20 µL

50 µg/mL	250 µL	20 µL
60 µg/mL	300 µL	20 µL
70 µg/mL	350 µL	20 µL
80 µg/mL	400 µL	20 µL
90 µg/mL	450 µL	20 µL
100 µg/mL	500 µL	20 µL

The makeup of the 50µg/mL CCV standard is detailed below. Occasionally, unusual compounds are added to the mix so it is best to check the BNA standards log book for exact standard makeup. Note: MS list spikes and full list spikes for LCS and/or MS/MSD are prepared from an alternate source or lot number other than the calibration standards.

Example: 50µg/mL CCV standard preparation:

SV3640

using 1ml syringe add the appropriate amount of methylene chloride to a
2ml vial. Add the indicated amount of intermediate / stock standard to the vial.
Add 20ul of internal standard to the vial (**SV 3635**).Soultion stored in freezer at -15deg C+/-5degC for upto 1 week.

50 ug/mL CCV

	Stock Standard	Concentration		50 ug/mL	
SV3515	BNA intermediate mix	200 PPM		250 uL	
SV3517	Benzidine Int.	200 PPM		250 uL	
SV3516	Atrazine benzaldehyde Int.	200 PPM		250 uL	

6.2.3 The Initial Calibration Verification (ICV) standard is prepared from a vendor stock standard at a concentration of 200µg/mL as detailed below.

Example: 50µg/mL ICV standard preparation:

(SV2100) ~ 50ug/ml ICV

Into a 1 mL volumetric flask containing Methylene Chloride ~ about 0.6ml, add 250uls of
SV1995 (readystock 200ppm)
and dilute to volume.

Top off w/20uls of Internal Standard **(SV2079)**
Prep'd: 01/08/03 Exp: 03/16/03

(SV2396) ~ 50ug/ml ICV (benzaldehyde & atrazine)

Into a 1 mL volumetric flask containing Methylene Chloride ~ about 0.6ml, add 250uls of
SV2305 (intermediate stock 200ppm) and dilute to volume.
Top off w/20uls of Internal Standard **(SV2393)**

Prep'd: 09/18/03 Exp: 03/18/04

7.0 PROCEDURE

Prior to using Federal Register 625, SW-846 8270C/8270D, or CLP (semivolatile method) the samples are prepared for chromatography using the appropriate sample preparation and clean up methods (generally SW-846 methods 3510, 3520, 3540, 3550, 3580, EPA method 625 or CLP).

7.1 Chromatographic conditions: Refer to corresponding instrument maintenance log for current gas chromatograph and mass spectrometer conditions.

7.2 Tuning - Prior to any calibration or analysis, DFTPP tuning criteria must be met for a 50 ng injection of the tuning standard [see below]. Tune must be met every 12 hours sample analysis is to be performed (every 24 hours for *Federal Register* Method 625 except for South Carolina which only allows 12 hours). The injection port performance compounds (pentachlorophenol, benzidine and 4,4'-DDT) are also injected to verify the performance of the injection port and must meet the following criteria. Degradation of DDT to DDE and DDE should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, and no peak tailing should be visible for 8270C. **Tailing factor should be 2.0 for 8270D for benzidine and pentachlorophenol**. For NPDES samples, the benzidine (base/neutral) tailing factor must not exceed 3.0 while the pentachlorophenol (acid) tailing factor cannot exceed 5.0. The calculation for tailing factors is best illustrated in Figure 13 of the *Federal Register* Method 625 which has been placed in the appendix. If degradation is excessive and/or poor chromatography is seen, the injection port may require cleaning and maintenance. It may also be necessary to break off 15-30cm of the capillary column. The mass spectrum of DFTPP is acquired as follows: by using one scan at the apex peak, or by by using the mean of the apex and the preceding and following scans or mean of a symmetric pattern of scans about the apex, or using the average across the entire peak. Background subtraction is accomplished using a single scan or more than 20 scans prior to the elution of DFTPP.

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

8270D has different tuning citeria for meeting DFTPP. See page 44 Table 3 of Method 8270D for criteria.

7.3 Calibration

7.3.1 Initial Calibration - An initial calibration curve at no less than five concentration levels must be analyzed (only three concentration levels are required for *Federal Register* Method 625) and shown to meet the initial calibration criteria before any sample analysis may be performed. Method 625 requires that the %RSD be less than 35% to use the average response factor for

quantitation, the curve is to be used otherwise and should have a correlation coefficient (***r***) of ≥0.995 linear, 0.99 and six points for quadratic. Method 8270C requires that the %RSD be less than 15% to use the average response factor for quantitation, the curve is to be used otherwise as long as ***r*** is ≥0.995 linear, 0.99 and six points for quadratic. In addition, there are calibration check compounds (CCCs) which must have a %RSD less than 30% and system performance check compounds (SPCCs) which must meet a minimum avarage response factor of 0.050. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. Generally, levels for the curve are 2 ug/mL, 5 µg/mL, 10 µg/mL, 20 µg/mL, 30 µg/mL, 40 µg/mL, 50 µg/mL, 60 µg/mL, 70 µg/mL, 80 µg/mL, 90 ug/mL and 100 µg/mL. 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 SOP-224 for guidance. All integrations are checked for acceptability. Response factors of less than 0.050 must be supported by the mass spectrum of the lowest standard. Retention times are set using the midpoint of the curve. **No quadratic curves are used for SouthCarolina. For 8270D the RSD for each target analyte should be less than or equal to 20% and each calibration level should meet the minimum response factors listed in Table 4. If the 20% RSD is not met, then the minimum correlation coefficient for the curve must be 0.99. If more than 10% of the compounds do not meet the 20% RSD or minimum correlation coefficient of 0.99, then the chromatographic system is considered too reactive to begin analysis. Injector maintenance should be performed and repeat the calibration procedure.**

CCCs:	Base/Neutral	Acid
	Acenapthene	4-Chloro-3-methylphenol
	1,4-Dichlorobenzene	2,4-Dichlorophenol
	Hexachlorobutadiene	2-Nitrophenol
	N-Nitroso-di-phenylamine	Phenol
	Di-n-octyl-phthalate	Pentachlorophenol
	Fluoranthene	2,4,6-Trichlorophenol
	Benzo(a)pyrene	

SPCCs:	Base/Neutral	Acid
	N-Nitroso-di-n-propylamine	2,4-Dinitrophenol
	Hexachlorocyclopentadiene	4-Nitrophenol

7.3.2 Initial Calibration Verification (ICV) - A second source standard at the 50 µg/mL level is used to check the validity of the curve. The standard recovery for all analytes must be between 75 and 125% (**70-130% for 8270D**). If the second source recovery is above 125% or 130% for 8270D, it is possible that the main standard has deteriorated for that compound. That standard should be remade and reevaluated. If that does not correct the problem, the standard should probably be replaced and a new curve generated. If the second source recovery is below 75% or 70% for 8270D, the second source standard may have deteriorated for that compound. The standard should be remade and reanalyzed. If this does not correct the problem, the standard

should probably be replaced. If any compound in the ICV exceeds the criteria above, it may be evaluated and initialed by the organic section manager. If deemed acceptable, the analyst may continue analysis. Any manual integrations are documented by inclusion of the integrated signals with the quantitation report and chromatogram. All integrations are checked for acceptability. For ICV standard preparation refer to standard log book.

7.4 Continuing Calibration Verification (CCV)- Every 12 hours a CCV at 50 µg/mL must be analyzed and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analysis may be performed. Acceptance criteria for 8270C consists of the same SPCC requirements as the initial calibration. The CCCs must be less than or equal to 20% difference or drift (%D, calculations follow in section 7.9). If any of the CCCs do not meet the above limits, then all required analytes must be <20%D. Internal standard areas should be within 50 to 200 percent of the area of the curve midpoint or the previous CCV. Retention times for the internal standards should be within 30 seconds of the retention time of the curve midpoint or the previous CCV. Method 625 requires a %D of less than 20% for all required analytes. Any manual integrations are documented by inclusion of the integrated signals with the quantitation report and chromatogram. All integrations are checked for acceptability. Samples are then quantitated against the initial calibration curve. Note: If any compound in the continuing calibration not subject to the criteria above exceeds 30%D, it must be evaluated and initialed by the organic section manager. If deemed acceptable, the analyst may continue analysis. **For 8270D, the 20% difference criteria must be applied to all compounds. If the criterion is not met for more than 20% of the compounds included in the initial calibration, then the GC system should undergo maintenance. If this does not solve the problem, then the initial calibration should be repeated. Each of the most common target analytes should meet the minimum response factors listed in Table 4. In situations where the failed compound is present, the data must be flagged as estimated. If the compound fails high in the CCV and is not present, the result can be reported as non-detect.**

7.5 LCS - The LCS is extracted 1 per extraction batch of up to 20 samples. The LCS containing all regular full list calibrated compounds is spiked into deionized water or sodium sulfate for soil using an alternate source or lot number than the calibration standards. See the LCS report forms in the appendix for example laboratory generated limits and the NPDES limits for 625 samples. Recoveries for the MS/MSD spike analytes in LCSs are charted annually to generate control limits for samples analyzed by method 8270C. In all cases, the lowest upper limit would be 100% and the lowest lower limit would be 10%. If enough data points are not present to generate limits, the limits default to CLP spike limits for spike analytes or 10-100% for all other analytes. See Section 8.3 below for corrective action.
When analyzing samples for DOD QSM Version 3, DOD limits will be used.

7.6 Method Blank - Method blanks are extracted at a minimum of 1 per extraction batch up to 20 samples. See Section 8.4 below for criteria and corrective action.

7.7 Samples - Prior to analysis, 1.0 mL samples are prepared by verifying volume and spiking with 20uL of the internal standard solution.

7.8 Instrument sequence-The instrument sequence log is filled out prior to sample analyses.

An example of a typical instrument sequence log follows:
1-DFTPP Tune (12:00 am)
2-CCV
3-LCS
4-Method Blank (or sample)
5-Sample
6-Sample
7-Sample
8-Sample
9-Sample
10-Sample
11-Sample-MS
12-Sample-MSD
13-Sample
14-Sample
15-Sample
16-DFTPP (12:00pm - 12 hours since last DFTPP/CCV)
17-CCV
18-Sample
19-Sample
20-Sample

7.9 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the chemstation. This run log is signed, dated and paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through Target DB on the Windows NT data system. The following must be checked to determine if the sample will need any reanalysis or dilution. Formal data evaluation is detailed in SOP-200 and documented using the Analyst Data Review Checklist (see Appendix). **See SOP-224 for guidance on manual integrations.**

7.9.1 Internal Standards - Areas should be within 50 to 200 percent of the area of the curve midpoint. Retention time should be within 30 seconds of the retention time of the curve midpoint. If not, the sample and historical data should be evaluated to determine the cause of the problem. If matrix effect is confirmed by reextraction/reanalysis or historical data, complete a corrective action report and flag the affected compounds on the final report for matrix effect. Note: criteria applies to the continuing calibration, not samples, but is used as an indication of the sample analysis validity.

7.9.2 Surrogates – Control limits are determined annually by charting LCSs and method blanks. In all cases, the lowest upper limit would be 100% and the lowest lower limit would be 10%. All of the three surrogates for each fraction

must be within the control limits in order for the extraction batch to be in control. If a surrogate exceeds the limits, the reason for the malfunction must be determined and a corrective action report must be completed. The sample must be reanalyzed, reextracted or flagged for QC problems. *Federal Register* Method 625 contains no criteria for surrogate recovery. **When analyzing samples for DOD QSM Version 3, DOD limits will be used.**

Surrogate	Water	Soil/Sediment
Nitrobenzene-d5	30-110	30-110
2-Fluorobiphenyl	35-110	35-110
Terphenyl-d14	55-125	40-120
Phenol-d6	15-110	30-110
2-Fluorophenol	15-110	25-110
2,4,6-Tribromophenol	45-125	30-115

7.9.3 Analyte concentration must be within the range of the calibration curve after rounding to 2 significant figures. If an analyte exceeds the curve, a dilution must be performed and the next sample must be checked for carryover. Any dilution should keep the concentration of the analyte in question within the top half of the curve.

7.9.4 The qualitative identification of compounds is based on retention time and a comparison of the sample mass spectrum, after background subtraction, with characteristic ions in a reference mass spectrum from the NBS database (NBS75K.I). This database is used as it contains relatively uncontaminated mass spectra of each target compound which cannot be obtained from the daily calibrations during each 12 hour analytical period due to overlapping peaks in the mixes. Characteristic ions from the reference mass spectrum library are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. In addition, the following criteria must be met. The RRT of the sample analyte must be within 0.06 RRT units of the RRT of the standard analyte. The relative intensities of the characteristic ions must agree within 30% of the relative intensities of the same ions in the reference spectrum. Structural isomers that produce very similar mass spectra should be identified as individual isomers so long as their GC retention times differ substantially. A library search may be made for analytes not associated with the calibration for the purpose of tentative identification. NOTE: The GC/MS analyst uses intelligence guided by experience to make the identifications. In uncontaminated spectra where ions are missing due to low concentration, if the major ions are present in the correct ratios at the correct retention time, the identification will be considered positive. In contaminated spectra, special emphasis will be placed upon higher mass ions, and the major ions will usually need to be present as major components of the spectrum (either unsubtracted or subtracted) for the identification to be positive. All assessments of relative intensities of ions will be made by visual estimates from the spectra.

7.9.5 Quantitation - Once a compound has been identified qualitatively, the concentration must then be quantitated. If the RSD of the compound's response factor is 15%(20% 8270D) or less, then the concentration may be

determined using the average response factor ($\overline{RF}$) from the initial calibration data. Otherwise, the concentration must be determined from equations based on internal standard calibration using either linear or non-linear calibration. Calculations follow in Section 7.10.

7.10 Calculations:

7.10.1 The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.

A_{is} = Peak area (or height) of the internal standard.

C_s = Concentration of the analyte or surrogate.

C_{is} = Concentration of the internal standard.

7.10.2 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.

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

where CCV RF is the response factor from the analysis of the verification standard and Average RF is the average response factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within ±20% for each CCC analyte, or for all target analytes if the CCCs are not target analytes, before any sample analyses may take place. **20% difference for 8270D.**

7.10.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to µg/L.]

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(V_s)(1000)}$$

where:

A_s = Area (or height) of the peak for the analyte in the sample.

A_{is} = Area (or height) of the peak for the internal standard.

C_{is} = Concentration of the internal standard in the volume extracted in ug/L.

D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.

V_i = Volume of the extract injected (µL). The nominal injection volume for samples and calibration standards must be the same.

$\overline{RF}$ = Mean response factor from the initial calibration.

V_s = Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

7.10.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to µg/kg.]

7.10.5

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(W_s)(1000)}$$

where: A_s,

A_{is}, C_{is}, D, and $\overline{RF}$ are the same as for aqueous samples, and

W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

8.0 QUALITY ASSURANCE/QUALITY CONTROL/CORRECTIVE ACTIONS

8.1 Internals - All samples and QC are spiked with internal standards. See section 7.9.1 above for criteria and corrective action.

8.2 Surrogates - All samples and QC are spiked with surrogates. The surrogate recoveries from method blanks and LCS are used to generate control control limits for the surrogates. See section 7.9.2 above for criteria and corrective action. If any surrogate recoveries are below 10%, samples must be re-extracted if sample is available.

8.3 LCS Sample - The LCS is extracted 1 per extraction batch of up to 20 samples. The LCS is spiked using an alternate source or lot number than the calibration standards. If the LCS compound has a recovery above the upper limit, but the same compound is not detected in any of the batch samples, no corrective action is required. For all

other situations, the LCS should be reanalyzed for the failed analytes only. If the second analysis fails, all associated samples should be reextracted/reanalyzed for the failed analytes only or the data must be evaluated for flagging due to QC problems.

8.4 Method Blanks - The concentration of all method target analytes should be below the MDL for each method target analyte(**<RL for common lab contaminants and <1/2 RL for other targets for DOD QSM Ver.3 projects**). The first step of corrective action is to assess the effect on the samples. If an analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps should be taken to find/reduce/eliminate the source of this contamination in the method blank. If an analyte is found in the method blank and some, or all, of the other batch samples, then corrective action is required. The source of contamination must be investigated and appropriate action taken and documented to find/reduce/eliminate the source of this contamination. The method blank, and any samples containing the same contaminant, may need to be reextracted/reanalyzed. For the common laboratory contaminants, meeting the above requirements is not practical. Random cases of contamination are difficult to control, however, daily contamination is not acceptable and corrective action is essential. If a contaminant is found in the method blank and the samples, the compound concentration must be flagged with a 'B' on the final report unless the concentration is greater than 10x that found in the method blank.

8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD (for full list spikes, the full list spiking solution is used). Control limits for the MS/MSD recoveries are the same as those for the LCS found in the appendix. RPD limits are found on the LCS report forms in the appendix. Samples which do not meet these criteria due to matrix must be evaluated for flagging on the final report due to QC problems. Generally, batch control is not based on MS/MSD results unless general method failure is determined to be the problem. In that case, the samples and associated QC would be reanalyzed for the failed analytes only. MS data evaluation must include the consideration of the following factors. **When analyzing samples for DOD QSM Version 3, DOD limits will be used.**

8.5.1 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.

8.5.2 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.

8.5.3 MS vs. MSD - If a spiked compound has a similar problem in both the MS and MSD and is not traced to a method problem, no further action may be necessary other than the generation of a corrective action report to document the problem.

8.5.4 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.

8.5.5 Demonstration of Capability (DOC) – Each new analyst must complete a demonstration of capability by analyzing four LCSs with acceptable precision and accuracy. This also must be done when a new instrument is installed or a significant change to the method has been made.

9.0 HEALTH AND SAFETY

9.1 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of gloves and lab coats is highly recommended.

9.2 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.

9.3 MSDS sheets are available for all reagents and standards which have been purchased. These are located on the bookshelf outside the office supply storage room.

10.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

10.1 Please see Waste Disposal, SOP-210 and SOP-405 for proper disposal of waste coming from this area within our laboratory.

10.2 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.

11.0 TABLE OF DEFINITIONS, REFERENCES & EXAMPLE FORMS

Definitions and examples of the LCS report sheets (625 water limits, in-house water limits and in-house soil limits), the analyst data review checklist and figure 13 from *Federal Register* method 625 for the tailing factor calculation are located in the appendix. **When analyzing samples for DOD QSM Version 3, DOD limits will be used for surrogates, LCS, and MS/MSD.**

APPENDIX

TABLE OF DEFINITIONS

1. amu- atomic mass unit
2. BNA- Base neutral/acid
3. °C- degrees Centigrade
4. CCC- Calibration Check Compound
5. CCV- Continuing Calibration Verification
6. CLP- Contract Laboratory Program
7. %D- percent difference
8. DFTPP- Difluorotriphenyphosphine
9. EICP- extracted ion current profile
10. EPA- Environmental Protection Agency
11. g- gram or grams
12. GC- Gas Chromatograph
13. GC/MS- Gas Chromatograph/Mass Spectrometer
14. ICV- Initial Calibration Verification
15. I.D.- inner diameter
16. ISTD- internal standard
17. LSC- Laboratory Sample Concentrator
18. MDL- method detection limit
19. MS- Matrix Spike
20. MSD- Matrix Spike Duplicate
21. M.S.- Mass Spectrometer
22. µm- micometer
23. µL- microliter
24. mL- milliliter
25. mm- millimeter
26. ng- nanogram
27. NPDES- National Pollutant Discharge Elimination System
28. P&T- purge and trap
29. QC- quality control
30. %R- percent recovery
31. RPD- relative percent difference
32. RRT- relative retention time
33. %RSD- percent relative standard deviation
34. SOP- Standard Operating Procedure
35. Surr.- surrogate
36. SPCC- System Performance Check Compound
37. TCLP- Toxicity Characteristic Leaching Procedure
38. USACE- United States Army Corps Of Engineers
39. LCS – Lab Control Sample

Refer to SOP-431 for additional definitions

REFERENCES

1. *40 CFR, Part 136*; Appendix A
2. *Test Methods for Evaluating Solid Waste*, SW-846
3. *National Environmental Laboratory Accreditation Conference*; CH. 5, 2003
4. USACE, *EM 200-1-3; Appendix 1; Shell*, 2/2001
5. *DOD Quality Systems Manual for Environmental Laboratories*, Ver.3, 01/2006

FORM 3

WATER SEMIVOLATILE LAB CONTROL SAMPLE (625)

Lab Name: Empirical Laboratories Contract:

Lab Code: NA Batch No.: NA SAS No.: NA SDG No.:

Matrix Spike - Client Sample No.:

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	LCS CONCENTRATION (ug/L)	LCS % REC #	QC. LIMITS REC.
Acenaphthene	100.00	0.0000	100.00	100	47-145
Acenaphthylene	100.00	0.0000	100.00	100	33-145
Anthracene	100.00	0.0000	100.00	100	27-133
Benzidine	100.00	0.0000	100.00	100	D-110
Benzo(a)anthracene	100.00	0.0000	100.00	100	33-143
Benzo(b)fluoranthene	100.00	0.0000	100.00	100	24-159
Benzo(k)fluoranthene	100.00	0.0000	100.00	100	11-162
Benzo(g,h,i)perylene	100.00	0.0000	100.00	100	D-219
Benzo(a)pyrene	100.00	0.0000	100.00	100	17-163
bis(2-Chloroethoxy)meth	100.00	0.0000	100.00	100	33-184
bis(2-Chloroethyl)ether	100.00	0.0000	100.00	100	12-158
bis(2-Chloroisopropyl)e	100.00	0.0000	100.00	100	36-166
Bis(2-ethylhexyl)phthal	100.00	0.0000	100.00	100	8-158
4-Bromophenyl-phenyleth	100.00	0.0000	100.00	100	53-127
Butylbenzylphthalate	100.00	0.0000	100.00	100	D-152
4-Chloro-3-methylphenol	100.00	0.0000	100.00	100	22-147
2-Chloronaphthalene	100.00	0.0000	100.00	100	60-118
2-Chlorophenol	100.00	0.0000	100.00	100	23-134
4-Chlorophenyl-phenylet	100.00	0.0000	100.00	100	25-158
Chrysene	100.00	0.0000	100.00	100	17-168
Dibenz(a,h)anthracene	100.00	0.0000	100.00	100	D-227
1,2-Dichlorobenzene	100.00	0.0000	100.00	100	32-129
1,3-Dichlorobenzene	100.00	0.0000	100.00	100	D-172
1,4-Dichlorobenzene	100.00	0.0000	100.00	100	20-124
3,3'-Dichlorobenzidine	100.00	0.0000	100.00	100	D-262
2,4-Dichlorophenol	100.00	0.0000	100.00	100	39-135
Diethylphthalate	100.00	0.0000	100.00	100	D-114
2,4-Dimethylphenol	100.00	0.0000	100.00	100	32-119

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

COMMENTS: _____

FORM 3

WATER SEMIVOLATILE LAB CONTROL SAMPLE (625)

Lab Name: Empirical Laboratories Contract:

Lab Code: NA Batch No.: NA SAS No.: NA SDG No.:

Matrix Spike - Client Sample No.:

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	LCS CONCENTRATION (ug/L)	LCS % REC #	QC. LIMITS REC.
Dimethylphthalate	100.00	0.0000	100.00	100	D-112
Di-n-butylphthalate	100.00	0.0000	100.00	100	1-118
4,6-Dinitro-2-methylphe	100.00	0.0000	100.00	100	D-181
2,4-Dinitrophenol	100.00	0.0000	100.00	100	D-191
2,4-Dinitrotoluene	100.00	0.0000	100.00	100	39-139
2,6-Dinitrotoluene	100.00	0.0000	100.00	100	50-158
Di-n-octylphthalate	100.00	0.0000	100.00	100	4-146
Fluoranthene	100.00	0.0000	100.00	100	26-137
Fluorene	100.00	0.0000	100.00	100	59-121
Hexachlorobenzene	100.00	0.0000	100.00	100	D-152
Hexachlorobutadiene	100.00	0.0000	100.00	100	24-116
Hexachlorocyclopentadie	100.00	0.0000	100.00	100	15- 70
Hexachloroethane	100.00	0.0000	100.00	100	40-113
Indeno(1,2,3-cd)pyrene	100.00	0.0000	100.00	100	D-171
Isophorone	100.00	0.0000	100.00	100	21-196
Naphthalene	100.00	0.0000	100.00	100	21-133
Nitrobenzene	100.00	0.0000	100.00	100	35-180
2-Nitrophenol	100.00	0.0000	100.00	100	29-182
4-Nitrophenol	100.00	0.0000	100.00	100	D-132
N-Nitroso-di-methylamin	100.00	0.0000	100.00	100	29- 66
N-Nitrosodiphenylamine	100.00	0.0000	100.00	100	23-100
N-Nitroso-di-n-propylam	100.00	0.0000	100.00	100	D-230
Pentachlorophenol	100.00	0.0000	100.00	100	14-176
Phenanthrene	100.00	0.0000	100.00	100	54-120
Phenol	100.00	0.0000	100.00	100	5-112
Pyrene	100.00	0.0000	100.00	100	52-115
1,2,4-Trichlorobenzene	100.00	0.0000	100.00	100	44-142
2,4,6-Trichlorophenol	100.00	0.0000	100.00	100	37-144

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

RPD: 0 out of 0 outside limits
Spike Recovery: 0 out of 56 outside limits

COMMENTS: _____

FORM 3

WATER SEMIVOLATILE LAB CONTROL SAMPLE (In-House)

Lab Name: Empirical Laboratories Contract:

Lab Code: NA Batch No.: NA SAS No.: NA SDG No.:

Matrix Spike - Client Sample No.:

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	LCS CONCENTRATION (ug/L)	LCS % REC #	QC. LIMITS REC.
Acenaphthene	100.0	0.0000	100.00	100	40-112
4-Chloro-3-methylphenol	200.0	0.0000	200.00	100	32-110
2-Chlorophenol	200.0	0.0000	200.00	100	29-110
1,4-Dichlorobenzene	100.0	0.0000	100.00	100	26-110
2,4-Dinitrotoluene	100.0	0.0000	100.00	100	46-122
4-Nitrophenol	200.0	0.0000	200.00	100	21-110
N-Nitroso-di-n-prop.(1)	100.0	0.0000	100.00	100	39-110
Pentachlorophenol	200.0	0.0000	200.00	100	32-133
Phenol	200.0	0.0000	200.00	100	10-110
Pyrene	100.0	0.0000	100.00	100	51-137
1,2,4-Trichlorobenzene	100.0	0.0000	100.00	100	28-110

COMPOUND	SPIKE ADDED (UG/L)	MSD CONCENTRATION (UG/L)	MSD % REC #	% RPD #	QC LIMITS RPD	QC LIMITS REC.
Acenaphthene	100.0	100.00	100	0	31	40-112
4-Chloro-3-methylphenol	200.0	200.00	100	0	42	32-110
2-Chlorophenol	200.0	200.00	100	0	40	29-110
1,4-Dichlorobenzene	100.0	100.00	100	0	28	31-110
2,4-Dinitrotoluene	100.0	100.00	100	0	38	46-122
4-Nitrophenol	200.0	200.00	100	0	50	21-110
N-Nitroso-di-n-prop.(1)	100.0	100.00	100	0	38	39-110
Pentachlorophenol	200.0	200.00	100	0	50	32-133
Phenol	200.0	200.00	100	0	42	10-110
Pyrene	100.0	100.00	100	0	31	51-137
1,2,4-Trichlorobenzene	100.0	100.00	100	0	28	28-110

(1) N-Nitroso-di-n-propylamine
Column to be used to flag recovery and RPD values with an asterisk
* Values outside of QC limits

RPD: 0 out of 11 outside limits
Spike Recovery: 0 out of 22 outside limits

COMMENTS: _____

FORM 3

SOIL SEMIVOLATILE LAB CONTROL SAMPLE (In-House)

Lab Name: Empirical Laboratories Contract:

Lab Code: ELABN Case No.: NA SAS No.: NA SDG No.:

Matrix Spike - Client Sample No.: Level:(low/med) LOW

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	LCS CONCENTRATION (ug/Kg)	LCS % REC #	QC. LIMITS REC.
Acenaphthene	10000	0.0000	10000	100	27-116
4-Chloro-3-methylphenol	20000	0.0000	20000	100	18-111
2-Chlorophenol	20000	0.0000	20000	100	10-110
1,4-Dichlorobenzene	10000	0.0000	10000	100	14-110
2,4-Dinitrotoluene	10000	0.0000	10000	100	34-110
4-Nitrophenol	20000	0.0000	20000	100	24-120
N-Nitroso-di-n-prop.(1)	10000	0.0000	10000	100	24-112
Pentachlorophenol	20000	0.0000	20000	100	10-114
Phenol	20000	0.0000	20000	100	10-110
Pyrene	10000	0.0000	10000	100	33-150
1,2,4-Trichlorobenzene	10000	0.0000	10000	100	19-110

COMPOUND	SPIKE ADDED (ug/Kg)	LCSD CONCENTRATION (ug/Kg)	LCSD % REC #	% RPD #	QC LIMITS RPD	QC LIMITS REC.
Acenaphthene	10000	10000	100	0	19	27-116
4-Chloro-3-methylphenol	20000	20000	100	0	42	18-111
2-Chlorophenol	20000	20000	100	0	50	10-110
1,4-Dichlorobenzene	10000	10000	100	0	27	14-110
2,4-Dinitrotoluene	10000	10000	100	0	47	34-110
4-Nitrophenol	20000	20000	100	0	50	24-120
N-Nitroso-di-n-prop.(1)	10000	10000	100	0	38	24-112
Pentachlorophenol	20000	20000	100	0	47	40-110
Phenol	20000	20000	100	0	35	10-110
Pyrene	10000	10000	100	0	36	33-150
1,2,4-Trichlorobenzene	10000	10000	100	0	23	19-110

(1) N-Nitroso-di-n-propylamine
Column to be used to flag recovery and RPD values with an asterisk
* Values outside of QC limits
RPD: 0 out of 11 outside limits
Spike Recovery: 0 out of 22 outside limits

COMMENTS: _____

BNA STANDARDS USED

base/neutral mix (2000ppm)		**acids mix (2000ppm)**
bis(2-Chloroethyl)ether		2,4-Dinitrophenol
bis(2-Chloroisopropyl)ether		2-Methylphenol
1,3-Dichlorobenzene		4-Methylphenol
1,2-Dichlorobenzene		Benzoic acid
1,4-Dichlorobenzene		4,6-Dinitro-2-methylphenol
Hexachloroethane		4-Nitrophenol
N-Nitroso-di-methylamine		2,4,5-Trichlorophenol
N-Nitroso-di-n-propylamine		2,4,6-Trichlorophenol
2,4-Dinitrotoluene		Phenol
2,6-Dinitrotoluene		Pentachlorophenol
Fluorene		2-Nitrophenol
Dimethylphthalate		4-Chloro-3-methylphenol
Hexachlorocyclopentadiene		2,4-Dichlorophenol
Anthracene		2,4-Dimethylphenol
4-Bromophenyl-phenylether		Benzoic acid
Di-n-butylphthalate		
bis(2-Chloroethoxy)methane		
1,2-Diphenylhydrazine		**semivoa misc.mix(2000ppm)**
Fluoranthene		Aniline
Hexachlorobenzene		Benzyl alcohol
N-Nitrosodiphenylamine		Carbazole
Phenanthrene		4-Chloroaniline
Hexachlorobutadiene		Dibenzofuran
Isophorone		2-Methylnaphthalene
Naphthalene		2-Nitroaniline
Nitrobenzene		3-Nitroaniline
1,2,4-Trichlorobenzene		4-Nitroaniline
Acenaphthene		Pyridine
Acenaphthylene		
2-Chloronaphthalene		
4-Chlorophenyl-phenylether		**Benzidine mix (2000ppm)**
Diethylphthalate		Benzidine
Benzo(a)anthracene		3,3'-Dichlorobenzidine
Bis(2-ethylhexyl)phthalate		
Butylbenzylphthalate		
Chrysene		**individual mixes (10,000ppm)**
p-(Dimethylamino)azobenzene		Caprolactam
Pyrene		Benzaldehyde
Benzo(b)fluoranthene		Atrazine
Benzo(k)fluoranthene		1,1'-Biphenyl
Benzo(g,h,i)perylene		Catechol
Benzo(a)pyrene		
Dibenz(a,h)anthracene		
Di-n-octylphthalate		
Indeno(1,2,3-cd)pyrene		

INTERNAL STANDARD ASSOCIATION / QUANT ION TABLE					
COMPOUND	***I.S**	**Q.M**	**COMPOUND**	***I.S**	**Q.M**
1,4-Dichlorobenzene-d4 **(I.S)(1)**		152	Dimethylphthalate	59	163
Acetophenone	1	105	Hexachlorocyclopentadiene	59	237
Aniline	1	93	2,4-Dinitrophenol	59	184
Benzaldehyde	1	106	2,4-Dinitrotoluene	59	165
Benzyl alcohol	1	108	2,6-Dinitrotoluene	59	165
bis(2-Chloroethyl)ether	1	93	Fluorene	59	166
bis(2-Chloroisopropyl)ether	1	45	2-Nitroaniline	59	65
1,3-Dichlorobenzene	1	146	3-Nitroaniline	59	138
1,2-Dichlorobenzene	1	146	4-Nitroaniline	59	138
1,4-Dichlorobenzene	1	146	4-Nitrophenol	59	65
2-Methylphenol	1	108	2,4,5-Trichlorophenol	59	196
4-Methylphenol	1	108	2,4,6-Trichlorophenol	59	196
3-Methylphenol	1	108	2-Fluorobiphenyl (S)	59	172
Phenol	1	94	Phenanthrene-d10 **(I.S) (79)**		188
Pyridine	1	79	Anthracene	79	178
Hexachloroethane	1	117	Atrazine	79	200
N-Nitroso-di-methylamine	1	42	4-Bromophenyl-phenylether	79	248
N-Nitroso-di-n-propylamine	1	70	Carbazole	79	167
2-Fluorophenol (S)	1	112	Di-n-butylphthalate	79	149
Phenol-d6 (S)	1	99	4,6-Dinitro-2-methylphenol	79	198
Naphthalene-d8 **(I.S)(35)**		136	1,2-Diphenylhydrazine	79	77
Benzoic acid	35	105	Fluoranthene	79	202
bis(2-Chloroethoxy)methane	35	93	Hexachlorobenzene	79	284
Caprolactam	35	113	N-Nitrosodiphenylamine	79	169
4-Chloroaniline	35	127	Pentachlorophenol	79	266
4-Chloro-3-methylphenol	35	107	Phenanthrene	79	178
2,4-Dichlorophenol	35	162	2,4,6-Tribromophenol (S)	79	330
2,4-Dimethylphenol	35	107	Chrysene-d12 **(I.S) (92)**		240
Hexachlorobutadiene	35	225	Benzidine	92	184
Isophorone	35	82	Benzo(a)anthracene	92	228
2-Methylnaphthalene	35	141	Bis(2-ethylhexyl)phthalate	92	149
Naphthalene	35	128	Butylbenzylphthalate	92	149
Nitrobenzene	35	77	Chrysene	92	228
2-Nitrophenol	35	139	3,3'-Dichlorobenzidine	92	252
1,2,4-Trichlorobenzene	35	180	p-(Dimethylamino)azobenzene	92	225
Catechol	35	110	Pyrene	92	202
Nitrobenzene-d5 (S)	35	82	Terphenyl-d14 (S)	92	244
Acenaphthene-d10 **(I.S) (59)**		164	Perylene-d12 **(I.S) (101)**		264
Acenaphthene	59	153	Benzo(b)fluoranthene	101	252
Acenaphthylene	59	152	Benzo(k)fluoranthene	101	252
1,1'-Biphenyl	59	154	Benzo(g,h,i)perylene	101	276
2-Chloronaphthalene	59	162	Benzo(a)pyrene	101	252
4-Chlorophenyl-phenylether	59	204	Dibenz(a,h)anthracene	101	278
Dibenzofuran	59	168	Di-n-octylphthalate	101	149
Diethylphthalate	59	149	Indeno(1,2,3-cd)pyrene	101	276
I.S=internal standard, Q.M=quant mass, S=surrogate					

INTERNAL STANDARD ASSOCIATION / QUANT ION TABLE					
COMPOUND	***I.S**	**Q.M**	**COMPOUND**	***I.S**	**Q.M**
1,4-Dichlorobenzene-d4 **(I.S)(1)**		152	Diphenylamine	59	169
Pentachloroethane	1	167	Thionazin	59	107
2-Picoline	1	93		59	
N-Nitrosomethylethylamine	1	88		59	
Methyl methanesulfonate	1	80		59	
N-Nitrosodiethylamine	1	102		59	
Ethyl methanesulfonate	1	79		59	
N-Nitrosopyrrolodine	1	100		59	
N-Nitrosomorpholine	1	56		59	
0-Toluidine	1	106		59	
	1			59	
	1			59	
	1			59	
	1		Phenanthrene-d10 **(I.S) (79)**		188
	1		4-Nitroquinoline-1-oxide	79	190
	1		Phenacetin	79	108
	1		4-Aminobiphenyl	79	169
	1		Pentachloronitrobenzene	79	237
	1		Sulfotepp	79	97
	1		Phorate	79	75
Naphthalene-d8 **(I.S)(35)**		136	Diallate	79	86
1- Methylnaphthalene	35	141	Dimethoate	79	87
N-Nitrosopiperidine	35	114	Pronamide	79	173
a,a-Dimethylphenethylamine	35	58	Disulfoton	79	88
O,O,O-Triethylphosphorothioate	35	97	Dinoseb	79	211
Hexachloropropene	35	213		79	
2,6-Dichlorophenol	35	162		79	
p-Phenylenediamine	35	108	Chrysene-d12 **(I.S) (92)**		240
N-Nitrosodi-n-butylamine	35	84	Methapyrilene	92	97
Safrole	35	162	p-(Dimethylamino)azobenzene	92	225
1,2,4,5-Tetrachlorobenzene	35	216	Chlorobenzilate	92	251
	35		3,3'- Dimethylbenzidine	92	212
	35		2- Acetylaminofluorene	92	181
	35		7,12-Dimethylbenz[a]anthracene	92	256
	35		Aramite	92	185
	35		Methyl parathion	92	109
	35		Parathion	92	109
Acenaphthene-d10 **(I.S) (59)**		164	Isodrin	92	193
Isosafrole	59	162	Kepone	92	272
1,4-Naphthoquinone	59	158	Famphur	92	218
Pentachlorobenzene	59	250	Perylene-d12 **(I.S) (101)**	101	
2-Naphthylamine	59	143	3-Methylcholanthrene	101	268
1-Naphthylamine	59	143	Hexachlorophene	101	196
2,3,4,6-Tetrachlorophenol	59	232		101	
5-Nitro-o-toluidine	59	152		101	
I.S=internal standard, Q.M=quant mass, S=surrogate					

Internal Standards & Surrogates

BNA internals (2000ppm)		**Acid surrogate (7500ppm)**
1,4-Dichlorobenzene-d4 **(I.S)(1)**		2-Fluorophenol (S)
Naphthalene-d8 **(I.S)(35)**		Phenol-d6 (S)
Acenaphthene-d10 **(I.S) (59)**		2,4,6-Tribromophenol (S)
Phenanthrene-d10 **(I.S) (79)**		**2,-Chlorophenol-d4 (S)**
Chrysene-d12 **(I.S) (92)**		
Perylene-d12 **(I.S) (101)**		**BN surrogate (5000ppm)**
		Nitrobenzene-d5 (S)
		Terphenyl-d14 (S)
		2-Fluorobiphenyl (S)
		1,2-Dichlorobenzene-d4 (S)

Reporting Limits

	Soil ug/KG	Water ug/L		Soil ug/KG	Water ug/L
Pyridine	330	5.0	**Acenaphthylene**	330	5.0
N-Nitroso-di-methylamine	330	5.0	**2,6-Dinitrotoluene**	330	5.0
Aniline	330	5.0	**3-Nitroaniline**	1300	20
Phenol	330	5.0	**Acenaphthene**	330	5.0
bis(2-Chloroethyl)ether	330	5.0	**2,4-Dinitrophenol**	3300	50
2-Chlorophenol	330	5.0	**4-Nitrophenol**	1300	20
1,3-Dichlorobenzene	330	5.0	**Dibenzofuran**	330	5.0
1,4-Dichlorobenzene	330	5.0	**2,4-Dinitrotoluene**	330	5.0
Benzyl alcohol	330	5.0	**Diethylphthalate**	330	5.0
1,2-Dichlorobenzene	330	5.0	**4-Chlorophenyl-phenylether**	330	5.0
2-Methylphenol	330	5.0	**Fluorene**	330	5.0
bis(2-Chloroisopropyl)ether	330	5.0	**4-Nitroaniline**	1300	20
3-Methylphenol	330	5.0	**2,3,4,6-Tetrachlorophenol**	330	5.0
4-Methylphenol	330	5.0	**1,1'-Biphenyl**	330	5.0
N-Nitroso-di-n-propylamine	330	5.0	**1,2-Diphenylhydrazine**	330	5.0
Hexachloroethane	330	5.0	**Carbazole**	330	5.0
Benzaldehyde	330	5.0	**4,6-Dinitro-2-methyl phenol**	1300	20
Nitrobenzene	330	5.0	**N-Nitrosodiphenylamine**	330	5.0
Isophorone	330	5.0	**4-Bromophenyl-phenylether**	330	5.0
2-Nitrophenol	330	5.0	**Hexachlorobenzene**	330	5.0
2,4-Dimethylphenol	1300	20	**Pentachlorophenol**	1300	20
Benzoic acid	3300	50	**Phenanthrene**	330	5.0
bis(2-Chloroethoxy)methane	330	5.0	**Anthracene**	330	5.0
2,4-Dichlorophenol	330	5.0	**Di-n-butylphthalate**	330	5.0
1,2,4-Trichlorobenzene	330	5.0	**Fluoranthene**	330	5.0
Naphthalene	330	5.0	**Atrazine**	330	5.0
4-Chloroaniline	330	5.0	**Benzidine**	3300	50
Hexachlorobutadiene	330	5.0	**Pyrene**	330	5.0
4-Chloro-3-methylphenol	330	5.0	**Butylbenzylphthalate**	330	5.0
2-Methylnaphthalene	330	5.0	**3,3'-Dichlorobenzidine**	330	5.0
1-Methylnaphthalene	330	5.0	**Benzo(a)anthracene**	330	5.0
Acetophenone	330	5.0	**Chrysene**	330	5.0
1,2,4,5-Tetrachlorobenzene	330	5.0	**Bis(2-ethylhexyl)phthalate**	330	5.0
Caprolactam	330	5.0	**Di-n-octylphthalate**	330	5.0
Hexachlorocyclopentadiene	330	5.0	**Benzo(b)fluoranthene**	330	5.0
2,4,6-Trichlorophenol	330	5.0	**Benzo(k)fluoranthene**	330	5.0
2,4,5-Trichlorophenol	330	5.0	**Benzo(a)pyrene**	330	5.0
2-Chloronaphthalene	330	5.0	**Indeno(1,2,3-cd)pyrene**	330	5.0
2-Nitroaniline	1300	20	**Dibenz(a,h)anthracene**	330	5.0
Dimethylphthalate	330	5.0	**Benzo(g,h,i)perylene**	330	5.0

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):	
Batch Number(s):	
Method:	**8260B/8270C/8270D (Circle One)**

QA/QC Item	Yes	No	NA	Second Level Review
1. Is the BFB/DFTPP tune performed every 12 hours and is the tuning criteria met?				
2. Are the RRFs and % RSDs within QC limits for appropriate analytes for the initial calibration? Check the retention times for compounds with the same spectra. Check compounds with different conc.(eg. m/p-xylene, ketones,etc.).				
3. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?				
4. Does the Continuing Calibration Standard (CCV) meet the criteria for the CCCs and SPCCs.				
5. Is the Method Blank run at the desired frequency and is its concentration for target analytes less than the MDLs?				
6. Are the LCS, MS, MSD within control limits and run at the desired frequency?				
7. Are all sample holding times met, analytes within calibration range, IS areas and surrogate recoveries within QC limits?				
8. Was the Method Blank, LCS, MS, MSD and samples loaded to the GCMS_LFSYS Tablespace within the Target DB Database?				

Comments on any "No" response:

Primary-Level Review: _____ Date: _____

Second-Level Review: _____ Date: _____

GC/MS VOLATILES
BY EPA METHOD 624 AND
SW-846 METHOD 8260B
INCLUDING APPENDIX IX COMPOUNDS

SOP NUMBER: **SOP-202**

REVISION NUMBER: **21**

APPROVED BY:

SECTION MANAGER

QUALITY ASSURANCE OFFICER

EFFECTIVE DATE: **09/11/08**

DATE OF LAST REVIEW **09/11/08**

GC/MS VOLATILES
BY EPA METHOD 624 AND SW-846 METHOD 8260B

1.0 SCOPE AND APPLICATION

Please see Appendix for definitions.
This SOP (based primarily on SW-846 Method 8260B) is used for the analysis of volatile organic compounds in a variety of matrices (soils, sediments, waters, etc.). Methods SW-846 Method 8000B; *Federal Register* Method 624; and CLP Method for Volatiles have also been used in the development of this SOP. The analyses by these various methods are clearly defined in the respective regulatory manuals. A good understanding of these different methods is essential to the performance of each method. The normal laboratory list of analytes with their typical LCS limits is found attached in the appendix. Other compounds may be analyzed by this SOP as detailed in section 1.0 of SW-846 Method 8260B. Any questions left by this SOP should be answered by reading the methods, paying close attention to SW-846 8000B/8260B, EPA 624 and CLP. If questions still remain unanswered, check with the Organic Lab Manager, QA/QC Officer and/or the Technical Director.

2.0 METHOD SUMMARY

After sample preparation, the sample is introduced into the GC/MS generally using purge and trap but sometimes using direct injection (see SW-846 Methods 5030B, 5035 and 3585 for preparation). In purge and trap, the analytes are stripped from the sample using helium and trapped on an adsorbent tube. The tube is heated while being backflushed with helium to carry the analytes to the GC/MS system. The analytes are separated in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra of the sample. Analytes are quantitated relative to known standards using the internal standard method.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Section 3.0 and tables 3-1 and 3-2 of the Empirical Laboratories, LLC Quality Assurance Manual include details concerning sample preservation, containers and handling of volatile samples. All water and non-quarantined soil volatile samples are stored in the Hobart in the sample storage room at a temperature of 4°C. Quarantined soil volatile samples are stored in the dorm-size refrigerator in the sample receiving/log-in room at a temperature of 4°C. Non-preserved water volatile samples have a holding time of 7 days from date of sampling. Preserved water samples and soil volatile samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project).

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Section 3.0 of SW-846 Method 8260B details interferences and potential problems which may be encountered when dealing with volatile analyses.

5.0. EQUIPMENT AND APPARATUS

5.1 GC : HP 5890 or 6890, temperature programmable, suitable for split or splitless injection.

5.2 Column: DB-VRX 60 meter x 0.25 mm I.D. 1.4 µm film thickness or 20 meter x 0.18 mm ID 1.0 µm film thickness silicon coated fused silica capillary column or equivalent.

5.3 M.S. : HP 5971, 5972 or 5973 capable of scanning 35 to 500 amu every one second or less, using 70 volts electron energy in electron impact ionization mode. The MS is capable of producing a mass spectrum for p-Bromofluorobenzene, BFB, which meets all tuning criteria for EPA methods [when 1 µL (50 ng) of the GC/MS tuning standard are injected through the GC.]

5.4 Purge and Trap Unit

5.4.1 Concentrators: Tekmar LSC 2000 or Tekmar/Dohrmann 3000/3100 Sample Concentrator equipped with Supelco trap number 2-1066-U or 2-4920-U VOCARB 3000 providing good delivery for all target compounds.

5.4.2 Autosamplers: Varian Archon 51 position programmable autosampler with 5ml to 25ml water and heated soil capability.

5.5 Acquisition Software: HP chemstation system interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.

5.6 Data Processing Software: TargetDB on Windows NT data system interfaced to the HP Chemstation. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances of any EICP between specified time or scan-number limits. The most recent NBS mass spectral library is installed.

5.7 Microsyringes – 1.0, 5.0,10, 25, 100, 250, 500 and 1000 µL.

5.8 Syringes – 5, 25 and 50 mL, gas-tight with Luer end.

5.9 Balance - analytical, 0.0001 g; top-loading, 0.1 g.

5.10 Disposable pasteur pipets.

5.11 Volumetric flasks, Class A - 2 mL, 5 mL, 10 mL, 50 mL, 100 mL and 250 mL with ground-glass stoppers.

5.12 Spatula - stainless steel.

5.13 Glass scintillation vials - 20mL with screw caps.

5.14 Nitrile Gloves

5.15 pH paper (measures pH from 0-14).

6.0 REAGENTS AND SOLUTIONS

6.1 Organic-free reagent water - obtained from a modulab system.

6.2 Methanol - Purge and trap grade (EM-Omnisolv EM-0482-6 or equivalent)

6.3 Methanol - suitable for use in gas chromatography (B&J Omnisolv MX0484-1, or equivalent)

6.4 Sodium bisulfate, $NaHSO_4$ – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).

6.5 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label. The date they are opened is noted on the label and recorded in the VOA standards and reagents logbook along with their lot number and vendor. Each standard that is prepared is recorded in the VOA standards and reagents logbook and given a sequential number. Each standard label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. Stock standards, when opened, have an expiration date of 6 months, **except for gas standards for South Carolina samples which have a one week expiration date**. All stocks and standards are stored in the freezer at a temperature of -15°C ± 5°C or less from the date they are received/prepared. The freezer temperature is monitored daily with an annually calibrated thermometer and recorded with calibration correction in the VOA refrigerator/freezer logbook. Makeup of common standards is detailed below. See the VOA standards log book for makeup of other standards.

6.5.1 The Bromofluorobenzene (BFB) tuning standard is prepared as follows: Using a 50µL syringe, 40µL of standard (BFB @ 2500ng/µL) is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor,Lot) and diluted to volume with same making a 50ng/µL standard. After capping and inverting 3 times, the solution is transferred to a labeled 2ml, teflon-lined, screw-capped vial and stored in the freezer at -15°C ± 5°C or less for up to 6 months(1 week for South Carolina samples). A direct injection of 1µL is used to tune the instrument.

6.5.2 The internal and surrogate standard is prepared as follows: Using the indicated syringe, the indicated amount of standard is injected into a 50 mL volumetric flask containing P&T methanol (Vendor,Lot) and diluted to volume with same making a 150ng/µL standard. After capping and inverting 3 times, the solution is transferred to the Archon standard vial and stored under helium for 1 month or less. Each 8260/624 sample is automatically injected with 1µL of this standard. The internal standard/surrogate solution will be replaced if the –50%-200% criteria fails in the CCV.

Standard	Conc. (ng/µL)	Syringe (µL)	Amount (µL)
8260 ISTD/Surr. Mix (Vendor,Lot)	2500	1000	3000

6.5.3 Calibration standards are prepared from the vendor stock standards at appropriate concentrations as follows. Occasionally unusual compounds are added to the mix so it is best to check the VOA standards log book for exact standard makeup. Note: for laboratory control spikes (LCS), alternate sources or lot numbers from the main calibration standard are used to make the LCS standard. See the appendix for analytes in the main mixes.

6.5.3.1 Primary Standard: Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor,Lot) and diluted to volume with same to make a 100-500ng/µL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at -15°C ± 5°C for 1 week. A 50µg/L (5mL purge) standard is made using 25µL of this standard to 50mLof reagent water.

Stock Standard(CCV)	Conc (ng/µL)	Syringe(µL)	Amount(µL)	Final Conc (ng/µL)
2-CEVE **(Cat#30265)** (Vendor, Lot)	20000	25	20	200
Vinyl Acetate **(#3766)** (Vendor, Lot)	5000	100	80	200
Ketones **(cat#30006)** (Vendor, Lot)	5000	100	80	200
Liquid mix **(C-349H-07)** (Vendor, Lot)	2000	100	100	100
Custom mix **(CCS-1037)** (Vendor, Lot)	5000	50	40	100
Gases **(cat#30042)** (Vendor, Lot)	2000	100	100	100
Acrolein/Acrylonitrile **(CC2098.10)** (Vendor, Lot)	20,000	50	50	500

Additional compounds may be added such as Appendix IX. Refer to standards log.

6.5.4 ICV/LCS/MATRIX SPIKE MIX: A second source standard is to check the validity of the gas and primary calibration standards used in analyzing the calibration curve. Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor,Lot) and diluted to volume with same to make a 100-500ng/µL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at -15°C ± 5°C for 1 week. A 50µg/L (5mL purge) ICV/LCS/Matrix Spike is made using 25µL of this standard to 50mLof reagent water/Sample Matrix.

Stock Standard(ICV/LCS)	Conc (ng/µL)	Syringe(µL)	Amount(µL)	Final Conc (ng/µL)
2-CEVE(Vendor,Lot#)	20,000	25	20	200
Vinyl Acetate(Vendor,Lot#)	5000	100	80	200
Ketones(Vendor,Lot#)	5000	100	80	200
Liquid mix (Vendor,Lot#)	2000	100	100	100
Custom Mix(Vendor,Lot#)	5000	50	40	100
Gases (Vendor,Lot#)	2000	100	100	100
Acrolein/Acrylonitrile (Vendor,Lot#)	50,000	50	50	500

7.0 PROCEDURE

7.1 Chromatographic conditions – Refer to corresponding instrument maintenance log for current gas chromatograph, mass spectrometer, and concentrator conditions.

7.2 System Bakeout - Prior to analysis, the autosampler purge and trap unit is stepped to bake and an instrument blank is analyzed.

NOTE: Further cleaning may be accomplished by backflushing the lines with methanol and then analyzing blanks overnight.

7.3 Tuning - Prior to any calibration or analysis, BFB tuning criteria must be met for a 1.0µL injection of the tuning standard [see below]. Tune must be met every 12 hours sample analysis is to be performed (every 24 hours for *Federal Register* Method 624 except for South Carolina which only allows 12 hours). The mass spectrum of BFB is acquired as follows: by using one scan at the apex peak, or by using the mean of the apex and the preceding and following scans or mean of a symmetric pattern of scans about the apex, or using the average across the entire peak. Background subtraction is accomplished using a single scan no more than 20 scans prior to the elution of BFB.

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

7.4 Calibration: Calibration standards are made up in water using the appropriate amount of the methanol standard. Calibration for soils for South Carolina requires that 5mL of sodium bisulfate solution is added to each calibration standard made if the samples will be preserved with sodium bisulfate. All calibration standard integrations must be checked for acceptability.

7.4.1 Initial Calibration - An initial calibration curve at no less than five concentration levels must be analyzed and shown to meet the initial calibration criteria before any sample analysis may be performed. For Arizona samples the surrogates must also be calibrated at a minimum of five concentrations. Method 624 requires that the %RSD be less than 35% to use the average response factor for quantitation, the curve is to be used otherwise and should have a correlation coefficient (***r***) of $\geq$0.995. Method 8260B requires that the %RSD be less than 15% to use the average response factor for quantitation, the curve is to be used otherwise as long as ***r*** is $\geq$0.995 linear or $\geq$ 0.99 quadratic. In addition, there are calibration check compounds (CCCs) listed below which must have a %RSD less than 30% and five system performance check compounds (SPCCs) which must meet the average response factor criteria listed below. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. Any response factors less than 0.050 must be supported by the mass spectrum of the lowest standard. **No quadratic curves for South Carolina.**

CCCs:	1,1-Dichloroethene	Toluene
	Chloroform	Ethylbenzene
	1,2-Dichloropropane	Vinyl chloride

SPCCs:	Chloromethane	0.10
	1,1-Dichloroethane	0.10
	Bromoform	0.10
	Chlorobenzene	0.30
	1,1,2,2-Tetrachloroethane	0.30

7.4.2 Initial Calibration Verification - A second source standard at the 50 µg/L (5mL purge) level is used to check the validity of the curve. The standard recovery for all analytes must be between 75 and 125%. If the second source recovery is above 125%, the main standard has probably deteriorated for that compound. That standard must be replaced and a new curve generated. If the second source recovery is below 75%, the second source standard has probably deteriorated for that compound and must be replaced. Any manual integrations are documented by inclusion of the integrated signals with the quantitation report and chromatogram.

7.4.3 Continuing Calibration Verification (every 12 hours) - A midpoint calibration standard (generally 50 µg/L - 5mL purge) must be analyzed and calculated against the initial calibration curve, then shown to meet the

calibration check criteria before any sample analysis may be performed. Acceptance criteria for method 8260B consists of the same SPCC criteria as above and ≤20% drift or difference (calculations given in section 7.10) for the CCCs as listed above. The internal standards must also be evaluated as listed below. Any manual integrations are documented by inclusion of the integrated signals with the quantitation report and chromatogram. Samples are then quantitated against the initial calibration curve. Note: If any compound in the continuing calibration not subject to the criteria above exceeds 30% D, it must be evaluated and initialed by the organic section manager. If deemed acceptable, the analyst may continue analysis.
NOTE: Acceptance criteria for method 624 consists of meeting recovery limits found in table 5 of the method for a QC check sample. This QC check sample is made from a separate source or lot number than the calibration standard at a concentration of 20 µg/L.

Internal standard checks

7.4.3.1.1 Retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.4.3.1.2 Response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to + 100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

7.5 LCS - An LCS is analyzed every 12 hour tune.Using standards prepared from an alternate vendor or lot number, blank water is spiked at the 50 µg/L (5mL/soil) or 10 µg/L (25mL) level. See section 8.3 below for criteria and corrective action. Note: the concentration of the LCS will be 20 µg/L when analyzing 624 samples (QC Check Sample). **When analyzing samples for DOD QSM Version 3, DOD limits will be used.**

7.6 Method Blank - Prior to sample analysis, the system must be shown to be free of contamination through analysis of a method blank. See section 8.4 below for criteria and corrective action.

7.7 Sample Analysis - Prior to analysis, the samples are prepared for chromatography using the appropriate sample preparation method (5mL water, 25mL water, low soil,

high soil, etc.) See SOP-225 for preparation of a 5035 soil sample. For a 5mL/25mL water sample, use the following procedure:

7.7.1. Load the vial into the Archon autosampler in the expected position.

7.7.2. Program the Archon for the loaded vial range and necessary dilutions, making sure the programmed method is set for the same volume as the purge vessel on the front of the LSC 2000 or 3000/3100 and that the Chemstation sequence matches the Archon sequence. Note: TCLP samples are analyzed at a 10x dilution. One TCLP sample is spiked per matrix.

7.7.3. After analysis of the sample has been completed, check the pH of the sample using pH paper and verify it to be less than a pH of 2. If it is not, record the pH and generate a corrective action report. The sample report will have to be flagged for preservation if the analysis is being performed more than 7 days after sampling. [Note: TCLP samples do not require a pH check.]

7.8 Instrument sequence

An example of a typical instrument sequence log follows:
1-BFB Tune (12:00 am)
2-CCV
3-LCS
4-Method Blank
5-Sample
6-Sample
7-Sample
8-Sample
9-Sample
10-Sample
11-Sample
12-Sample
13-Sample
14-Sample
15-Sample
16-Sample
17-Sample MS
18-Sample MSD
19-BFB (12:00pm - 12 hours since last BFB/CCV)
20-CCV
21-LCS
22-Method Blank
23-Sample
24-Sample

7.9 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the chemstation. This run log is signed, dated and paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through the TargetDB on Windows NT data system. Quantitative measurements are performed using the calculations found in section 7.10 below. The following must be checked to determine if the sample will

need any reanalysis or dilution. Formal data evaluation is detailed in SOP-200 (documented using the USACE Analyst Data Review Checklist for USACE projects). **See SOP-224 for guidance on manual integrations.**

7.9.1 Internal Standards - Areas should be within 50 to 200 percent of the area of the continuing calibration. Retention time should be within 30 seconds of the retention time of the continuing calibration. Note: criteria applies to the continuing calibration, not samples, but is used as an indication of the sample analysis validity. If not, the sample and historical data should be evaluated to determine the cause of the problem. Reanalysis is expected if it appears to be from a leak. If matrix effect is confirmed by reanalysis or historical data, complete a corrective action report and flag the affected compounds on the final report for matrix effect.

7.9.2 Surrogates – Control limits are determined by charting LCSs and method blanks. All of the surrogates must be within these limits in order for the analysis to be in control. If not, the reason for the malfunction must be determined and reanalysis may be necessary. If historical data indicates matrix, the sample would be flagged appropriately. When the surrogates exceed either the control limits, a corrective action report must be completed. *Federal Register* Method 624 contains no criteria for surrogate recovery. **When analyzing samples for DOD QSM Version 3, DOD limits will be used.**

Surrogate	WATER	SOIL/SEDIMENT
Dibromofluoromethane	85-120	80-120
1,2-Dichloroethane-d4	85-130	75-140
Toluene-d8	85-115	80-120
Bromofluorobenzene	80-120	80-125

7.9.3 Analyte concentration must be within the range of the calibration curve after rounding to 2 significant figures. If an analyte exceeds the curve, a dilution must be performed, the next sample must be checked for carryover and the sparge position must be checked for contamination through the analysis of a system blank. Any dilution should keep the concentration of the analyte in question within the mid-range of the curve.

7.9.4 Qualitative identification is made as indicated below.

7.9.4.1 The mass spectra are compared to reference spectra in a user-created data base especially compiled to contain relatively uncontaminated mass spectra of each target compound. Note: Such a file cannot be obtained from the daily calibrations during each 12 hour analytical period due to overlapping peaks in the mixes.

7.9.4.2 The GC/MS analyst uses intelligence guided by experience to make the identifications.In uncontaminated spectra where ions are missing due to low concentration, if the major ions are present in the correct ratios at the correct retention time, the identification will be

considered positive. In contaminated spectra, special emphasis will be placed upon higher mass ions, and the major ions will usually need to be present as major components of the spectrum (either unsubtracted or subtracted) for the identification to be positive. All assessments of relative intensities of ions will be made by visual estimates from the spectra.

7.10 Calculations:

7.10.1 The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.

A_{is} = Peak area (or height) of the internal standard.

C_s = Concentration of the analyte or surrogate.

C_{is} = Concentration of the internal standard.

7.10.2 Calibration verification involves the calculation of the percent drift (linear) 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.

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

where CCV RF is the response factor from the analysis of the verification standard and Average RF is the average response factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within ±20% for each CCC analyte, or for all target analytes if the CCCs are not target analytes, before any sample analyses may take place.

7.10.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to ug/L.]

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(V_s)(1000)}$$

where:

A_s = Area (or height) of the peak for the analyte in the sample.

A_{is} = Area (or height) of the peak for the internal standard.

C_{is} = Concentration of the internal standard in the volume purged in ug/L.

D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.

V_i = For purge-and-trap analysis, V_i is not applicable and is set at 1.

$\overline{RF}$ = Mean response factor from the initial calibration.

V_s = Volume of the aqueous sample purged (mL). If units of liters are used for this term, multiply the results by 1000.

7.10.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to ug/kg.]

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(W_s)(1000)}$$

where: A_s,

A_{is}, C_{is}, D, and $\overline{RF}$ are the same as for aqueous samples, and

W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

8.0 QUALITY ASSURANCE/QUALITY CONTROL/CORRECTIVE ACTIONS

8.1 Internal Standards - All samples and QC are spiked with internals. See section 7.9.1 above for criteria and corrective action.

8.2 Surrogates - All samples and QC are spiked with surrogates. The surrogate recoveries from method blanks and LCS are used to generate control limits. See section 7.9.2 above for criteria and corrective action.

8.3 LCS Sample - An LCS is analyzed every 12 hour tune. To prepare the LCS, a blank is spiked with standards prepared from an alternate vendor or lot number from the calibration standards. Note: the concentration of the LCS will be 20 µg/L when analyzing 624 samples (QC Check Sample). The recoveries are used to generate control limits. See the LCS report forms in the appendix for the laboratory generated limits. The limits are in-house generated matrix spike limits or client specified limits for matrix spike analytes and 70-130% (or client specified limits) recovery for waters or soils for all other analytes if limits have not been generated. Limits for 624 LCSs are taken from table 5 of method 624. If the LCS compound has a recovery above the upper limit, but the same compound is not detected in any of the batch samples, no corrective action is required. For all other

situations, the LCS should be reanalyzed for the failed analytes only. If the second analysis fails, all associated samples should be reanalyzed for the failed analytes only. **When analyzing samples for DOD QSM Version 3, DOD limits will be used. South Carolina limits are 70-130% except for poor purgers which are 60-140%.**

8.4 Method Blanks - The concentration for most method target analytes must be < ½ the reporting limit. Common laboratory contaminants, such as acetone and methylene chloride, must be < the reporting limit. The first step of corrective action is to assess the affect on the samples. If an analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps should be taken to find/reduce/eliminate the source of this contamination in the method blank. If an analyte is found in the method blank and some, or all, of the other batch samples, then corrective action is required. The source of contamination must be investigated and appropriate action taken and documented to find/reduce/eliminate the source of this contamination. The method blank, and any samples containing the same contaminant, would likely be reanalyzed. For the common laboratory contaminants, meeting the above requirements is not practical. Random cases of contamination are difficult to control, however, daily contamination is not acceptable and corrective action is essential. If a contaminant is found in the method blank and the samples, the compound concentration must be flagged with a 'B' on the final report unless the concentration is greater than 10x that found in the method blank. A method blank is analyzed every 12 hour tune.

8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for an MS/MSD with the LCS standard. Criteria for the MS/MSD recoveries are the same as the LCS limits. Limits for the RPDs are 30% RPD for water and soil.. Samples that do not meet these criteria due to matrix must be flagged on the final report for QC problems. Generally, batch control is not based on MS/MSD results unless general method failure is determined to be the problem. In that case, the samples and associated QC would be reanalyzed for the failed analytes only. MS data evaluation must include the consideration of the following factors. **When analyzing samples for DOD QSM Version 3, DOD limits will be used.**

8.5.1 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. A water sample which was taken from the same VOA vial for the original sample and the MS/MSD should have very good RPDs unless there has been a method problem. Corrective action must be taken in the form of reanalysis if a method problem is indicated.

8.5.2 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.

8.5.3 MS vs. MSD - If a spiked compound has a problem in both the MS and MSD, review the LCS and if acceptable no further action may be necessary since it is attributable to matrix effect.

8.5.4 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.

8.6 Demonstration of Capability (DOC) – Each new analyst must complete a demonstration of capability by analyzing four LCSs with acceptable precision and accuracy. This also must be done when a new instrument is installed or a significant change to the method has been made.

CHROMATOGRAPHIC COND.	**VOA1**	**VOA3**	**VOA4**	**VOA5**
GC	**5890 SERIES II**	**6890**	**5890 SERIES II**	**5890 SERIES II**
MS	**5971**	**5973**	**5972**	**5972**
GC COLUMN	RTX-VRX-60mX0.25mmX1.4um	RTX-VRX-60mX0.25mmX1.4um	DBVRX-60mX0.25mmX1.4um	RTX-VRX-60mX0.25mmX1.4um
INJECTOR LINER	1.0MM	1.0MM	2.0MM	2.0MM
INJECTOR TEMPERATURE	150deg C	150deg C	150deg C	150deg C
TRANSFER LINE TEMP.	250deg C	**150deg C**	250deg C	250deg C
COLUMN PRESSURE	9.0PSI	X	X	X
COLUMN FLOW	X	1.1 ml/min	1.1 ml/min	1.2 ml/min
SPLIT FLOW/RATIO	19ml/min@50deg C	43.3ml/min (40:1)	20ml/min@50deg C	20ml/min@50deg C
INITIAL TEMPERATURE	**45deg C**	**50deg C**	**50deg C**	**50deg C**
INITIAL HOLD TIME	**5.0 min**	**10 min**	**10 min**	**10 min**
RAMP1	**9deg C / min**	**25deg C / min**	**25deg C / min**	**25deg C / min**
TEMPERATURE 1 / TIME 1	**240deg C/ 6.1 min**	**220deg C/ 6.0 min**	**220deg C/ 7.2 min**	**220deg C/ 6.0 min**
RAMP2	X	X	X	X
TEMPERATURE 2 / TIME 2	X	X	X	X
TOTAL RUN TIME	32.7 min	23.0 min	24.2 min	23.0 min
CONCENTRATOR	**(Tekmar 2000)**	**(Tekmar 3100)**	**(Tekmar 3000)**	**(Tekmar 3100)**
ARCHON PURGE COND.				
VALVE/LINE TEMP.	125deg / 125deg C	110deg / 110deg C	130deg / 130deg C	130deg / 130deg C
PURGE TIME	11.0 min	11.0 min	11.0 min	11.0 min
PURGE FLOW	45ml/min	40ml/min	40ml/min	40ml/min
DRY PURGE TIME	2.0 min	2.0 min	2.0 min	2.0 min
DESORB PREHEAT	245deg C	255deg C	255deg C	255deg C
DESORB TEMPERATURE	250deg C	260deg C	260deg C	260deg C
DESORB TIME	**2.0 min**	**1.0 min**	**1.0 min**	**1.0 min**
BAKE TEMPERATURE	270deg C	270deg C	270deg C	270deg C
BAKE TIME	9.0 min	10.0 min	10.0 min	10.0 min

RTX columns are from RESTEK & DBVRX columns are from J&W.

9.0 HEALTH AND SAFETY

9.1 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.

9.2 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.

9.3 MSDS sheets are available for all reagents and standards that have been purchased. These are located in the office next to the technical director.

10. WASTE MANAGEMENT AND POLLUTION PREVENTION

10.1 Please see Waste Disposal, SOP-210 and SOP-405 for proper disposal of waste coming from this area within our laboratory.

10.2 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.

11.0 EXAMPLE FORMS

Definitions and examples of the instrument run log, LCS report sheets (624, water and soil) and the USACE analyst data review checklist are located in the following Appendix.
When analyzing samples for DOD QSM Version 3, DOD limits will be used for surrogates, LCS, and MS/MSD.

APPENDIX

TABLE OF DEFINITIONS

1. amu- atomic mass unit
2. BFB- Bromofluorobenzene
3. °C- degrees Centigrade
4. CCC- Calibration Check Compound
5. CCV- Continuing Calibration Verification
6. CLP- Contract Laboratory Program
7. %D- percent difference
8. EICP- extracted ion current profile
9. EPA- Environmental Protection Agency
10. g- gram or grams
11. GC- Gas Chromatograph
12. GC/MS- Gas Chromatograph/Mass Spectrometer
13. ICV- Initial Calibration Verification
14. I.D.- inner diameter
15. ISTD- internal standard
16. LSC- Laboratory Sample Concentrator
17. MDL- method detection limit
18. MS- Matrix Spike
19. MSD- Matrix Spike Duplicate
20. M.S.- Mass Spectrometer
21. µm- micometer
22. µL- microliter
23. mL- milliliter
24. mm- millimeter
25. ng- nanogram
26. P&T- purge and trap
27. QC- quality control
28. %R- percent recovery
29. RPD- relative percent difference
30. %RSD- percent relative standard deviation
31. SOP- Standard Operating Procedure
32. Surr.- surrogate
33. SPCC- System Performance Check Compound
34. TCLP- Toxicity Characteristic Leaching Procedure
35. USACE- United States Army Corps Of Engineers
36. VOA- volatile organic analysis
37. LCS- Lab Control Sample

REFERENCES

1. *40 CFR, Part 136*; Appendix A
2. *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition
3. *National Environmental Laboratory Accreditation Conference*; CH. 5, 2001
4. USACE, *EM 200-1-3; Appendix 1; Shell*, 2/2001
5. *DOD Quality Systems Manual for Environmental Laboratories*, 6/2002

FORM 3

WATER VOLATILE LAB CONTROL SAMPLE (624)

Lab Name: Empirical Laboratories, LLC Contract: ELAB

Lab Code: NA Batch No.: NA SAS No.: NA SDG No.:

Matrix Spike - Client Sample No.:

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	LCS CONCENTRATION (ug/L)	LCS % REC #	QC. LIMITS REC.
Acrolein	100.00	0.0000	100.000	100	NML
Acrylonitrile	100.00	0.0000	100.000	100	NML
Benzene	20.00	0.0000	20.000	100	64-136
Bromodichloromethane	20.00	0.0000	20.000	100	66-134
Bromoform	20.00	0.0000	20.000	100	71-129
Bromomethane	20.00	0.0000	20.000	100	14-186
Carbon tetrachloride	20.00	0.0000	20.000	100	73-127
Chlorobenzene	20.00	0.0000	20.000	100	66-134
Chloroethane	20.00	0.0000	20.000	100	38-162
2-Chloroethyl vinyl eth	40.00	0.0000	40.000	100	D-224
Chloroform	20.00	0.0000	20.000	100	68-132
Chloromethane	20.00	0.0000	20.000	100	D-204
Dibromochloromethane	20.00	0.0000	20.000	100	68-132
1,2-Dichlorobenzene	20.00	0.0000	20.000	100	63-137
1,3-Dichlorobenzene	20.00	0.0000	20.000	100	73-127
1,4-Dichlorobenzene	20.00	0.0000	20.000	100	63-137
1,1-Dichloroethane	20.00	0.0000	20.000	100	73-127
1,2-Dichloroethane	20.00	0.0000	20.000	100	68-132
1,1-Dichloroethene	20.00	0.0000	20.000	100	51-149
trans-1,2-Dichloroethen	20.00	0.0000	20.000	100	70-130
1,2-Dichloropropane	20.00	0.0000	20.000	100	34-166
cis-1,3-Dichloropropene	20.00	0.0000	20.000	100	24-176
trans-1,3-Dichloroprope	20.00	0.0000	20.000	100	50-150
Ethylbenzene	20.00	0.0000	20.000	100	59-141
Methylene chloride	20.00	0.0000	20.000	100	61-139
1,1,2,2-Tetrachloroetha	20.00	0.0000	20.000	100	61-139
Tetrachloroethene	20.00	0.0000	20.000	100	74-126
Toluene	20.00	0.0000	20.000	100	75-125
1,1,1-Trichloroethane	20.00	0.0000	20.000	100	75-125
1,1,2-Trichloroethane	20.00	0.0000	20.000	100	71-129
Trichloroethene	20.00	0.0000	20.000	100	67-133
Trichlorofluoromethane	20.00	0.0000	20.000	100	48-152
Vinyl chloride	20.00	0.0000	20.000	100	4-196

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

NML = No Method Limits

COMMENTS: _____

VOA Water Limits

	In-House	DOD Limits		South Carolina
1,1,1 Trichloroethane	80-125	65-130		70-130
1,1,1,2-Tetrachlorethane	70-140	80-130		70-130
1,1,2,2-Tetrachloroethane	70-130	65-130		70-130
1,1,2-Trichloroethane	80-130	75-125		70-130
1,1-Dichloroethane	75-130	70-135		70-130
1,1-Dichloroethene	70-125	70-130		70-130
1,2,4 Trichlorobenzene	45-135	65-135		70-130
1,2-Dibromo-3-chloropropane	70-130	50-130		70-130
1,2-Dibromoethane	75-130	80-120		70-130
1,2-Dichlorobenzene	70-130	70-120		70-130
1,2-Dichloroethane	70-135	70-130		70-130
1,2-Dichloropropane	75-130	75-125		70-130
1,3-Dichlorobenzene	65-125	75-125		70-130
1,4-Dichlorobenzene	70-125	75-125		70-130
2-Butanone	65-145	30-150		60-140*
2-Hexanone	70-140	55-130		60-140*
4-Methyl-2-pentanone	75-135	60-135		60-140*
Acetone	35-175	40-140		60-140*
Benzene	75-125	80-120		70-130
Bromochloromethane	80-125	65-130		70-130
Bromodichloromethane	85-135	75-130		70-130
Bromoform	70-140	70-130		70-130
Bromomethane	45-150	30-145		70-130
Carbon disulfide	65-130	35-160		70-130
Carbon tetrachloride	75-135	65-140		70-130
Chlorobenzene	75-120	80-120		70-130
Chloroethane	65-145	60-135		70-130
Chloroform	75-125	65-135		70-130
Chloromethane	45-145	40-125		70-130
Cis-1,2-Dichloroethene	80-120	70-125		70-130
Cis-1,3-Dichloropropene	75-130	70-130		70-130
Dibromochloromethane	80-140	60-135		70-130
Dibromomethane	65-140	75-125		70-130
Dichlorodifluoromethane	40-160	30-155		70-130
Ethylbenzene	75-130	75-125		70-130
Methylene chloride	70-130	55-140		70-130
M,p-Xylene	75-125	75-130		70-130
o-Xylene	75-130	80-120		70-130
Styrene	75-125	65-135		70-130
Tetrachloroethene	70-125	45-150		70-130
Toluene	75-125	75-120		70-130
Trans-1-2 Dichlorethene	70-125	60-140		70-130
Trans-1-3-Dichloropropene	70-130	55-140		70-130
Trichloroethene	80-125	70-125		70-130
Trichloroflurormethane	70-140	60-145		70-130
Vinyl chloride	65-140	50-145		70-130
MTBE	75-125	65-125		70-130

Naphthalene	45-135	55-140		70-130

*** - Poor Purger**

VOA Soil Limits

	Soil	DOD Limits		
1-1-1-2 Tetrachloroethane	75-125	75-125		
1-1-1-Trichloroethane	65-140	70-135		
1-1-2-2-Tetrachloroethane	65-130	55-130		
1-1-2-Trichloroethane	70-130	60-125		
1-1-Dichloroethane	70-135	75-125		
1-1-Dichloroethene	65-135	65-135		
1-2-4-Trichlorobenzene	55-135	65-130		
1-2-Dibromo-3-chloropropane	55-145	40-135		
1-2-Dibromoethane	70-135	70-125		
1-2-Dichlorobenzene	75-125	75-120		
1-2-Dichloroethane	65-140	70-135		
1-2-Dichloropropane	70-135	70-120		
1-3-Dichlorobenzene	70-125	70-125		
1-4-Dichlorobenzene	70-125	70-125		
2-Butanone	50-145	30-160		
2-Hexanone	50-140	45-145		
4-Methyl-2-pentanone	55-145	45-145		
Acetone	30-155	20-160		
Benzene	70-130	75-125		
Bromochloromethane	75-130	70-125		
Bromodichloromethane	70-145	70-130		
Bromoform	60-130	55-135		
Bromomethane	50-160	30-160		
Carbon disulfide	65-145	45-160		
Carbon tetrachloride	65-140	65-135		
Chlorobenzene	70-120	75-125		
Chloroethane	60-145	40-155		
Chloroform	65-135	70-125		
Chloromethane	50-150	50-130		
Dibromochloromethane	70-140	65-130		
Dibromomethane	75-125	75-130		
Dichlorodifluoromethane	35-155	35-135		
Ethylbenzene	75-125	75-125		
Methylene chloride	55-155	55-140		
Styrene	70-125	75-125		
Tetrachloroethene	65-130	65-140		
Toluene	55-140	70-125		
Trichloroethene	60-145	75-125		
Trichlorofluoromethane	60-140	25-185		
Vinyl Acetate	50-135			
Vinyl chloride	60-145	60-125		
cis-1-2-Dichlorethene	70-135	65-125		
cis-1-3-Dichloropropene	75-135	70-125		
m-p-Xylene	70-125	80-125		

o-Xylene	70-125	75-125		
trans-1-2-Dichloroethene	65-140	65-135		
trans-1-3--Dichloropropene	65-125	65-125		
MTBE	55-150			
Naphthalene	55-145	40-125		

VOA MIXES

LIQUID MIX (2000PPM)
C-610H (PRIMARY STANDARD)
M-502A-R-10X (ICV/LCS/MATRIX SPIKE)
Benzene
Bromobenzene
Bromochloromethane
Bromodichloromethane
Bromoform
n-Butylbenzene
sec-Butylbenzene
tert-butylbenzene
Carbon tetrachloride
Chlorobenzene
Chloroform
2- & 4-Chlorotoluene
Trichloroethene
Dibromochloromethane
1,2-Dibromo-3-chloropropane
1,2-Dibromoethane
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,1- & 1,2-Dichloroethane
1,1,2-Trichloroethene
1,1-Dichloroethene
cis-1,2-Dichloroethene
trans-1,2-Dichloroethene
1,2- & 1,3-Dichloropropane
1,2,3-Trichloropropane
2,2-Dichloropropane
1,1-Dichloropropene
cis-1,3-Dichloropropene
trans-1,3-Dichloropropene
Ethylbenzene
Hexachlorobutadiene
Isopropylbenzene
Isopropyltoluene
Methylene Chloride
Naphthalene
n-Propylbenzene
Styrene
1,1,1,2-Tetrachloroethane
1,1,2,2-Tetrachloroethane
Tetrachloroethene
Toluene
1,2,3-Trichlorobenzene

GASES MIX (2000PPM)
#30042 (PRIMARY STANDARD)
M-502B-R-10X (ICV/LCS/MATRIX SPIKE)
Bromomethane
Chloroethane
Vinyl chloride
Chloromethane
Dichlorodifluoromethane
Trichlorofluoromethane

C610H-PRIMARY STANDARD
M60310X (ICV/LCS/MATRIX SPIKE)
Acrolein (10,000PPM)
Acrylonitrile (10,000PPM)

CCS1037-PRIMARY CUSTOM MIX (5000PPM)
#557102- ICV/LCS CUSTOM MIX (5000PPM)
Tetrahydrofuran
MTBE
Methyl metacrylate
Ethyl methacrylate
1,1,2-Trichlorotrifluoroethane
Cyclohexane
Methylcyclohexane
Methyl acetate
Carbon disulfide
Iodomethane

PRIMARY and ICV/LCS STANDARD-neat
Vinyl acetate (5000PPM)

PRIMARYand ICV/LCS STANDARD-neat
2-Chloroethyl vinyl ether (20000PPM)

KETONES MIX (5000PPM)
#30006 (PRIMARY STANDARD and ICV/LCS) different lots
Acetone
2-butanone
2-hexanone
4-methyl-2-pentanone

1,2,3-Trimethylbenzene
1,2,4-Trimethylbenzene
1,3,5-Trimethylbenzene
m-,p- & o-Xylene
1,1,1-Trichloroethene

VOA CALIBRATIONS RANGE FOR EPA8260B/624(AQUEOUS)

Liquid mix, Gases mix & primary custom mix is calibrated at these levels - 0.5ppb, 1.0ppb, 2.0ppb, 10ppb, 20ppb, 50ppb, 100ppb & 200ppb. The ketones mix, 2-chloroethyl vinyl ether & vinyl acetate are calibrated at these levels - 1.0ppb, 2.0ppb, 4.0ppb, 20ppb, 40ppb, 100ppb, 200ppb & 400ppb. Acrolein & Acrylonitrile are calibrated at these levels – 2.5ppb, 5.0ppb, 10ppb, 50ppb, 100ppb, 250ppb, 500ppb, 1000ppb.

VOA CALIBRATIONS RANGE FOR EPA8260B(SOLIDS)

Liquid mix, Gases mix & primary custom mix is calibrated at these levels – 2.0ppb, 5.0ppb, 10ppb, 20ppb, 50ppb, 100ppb & 200ppb. The ketones mix, 2-chloroethyl vinyl ether & vinyl acetate are calibrated at these levels - 2.0ppb, 10ppb, 20ppb, 40ppb, 100ppb, 200ppb & 400ppb. Acrolein & Acrylonitrile are calibrated at these levels – 5.0ppb, 10ppb, 50ppb, 100ppb, 250ppb, 500ppb, 1000ppb.

VOA LCS/ICV/MATRIX SPIKE LEVEL FOR EPA8260B

Liquid mix, Gases mix, Ethyl methacrlate, Metyl methacrylate, Tetrahydrofuran, MTBE, Trichlorotrifluoroethane, Cyclohexane, Methylcyclohexane & Methyl acetate are spiked at 50ppb level. Acrolein & Acrylonitrile spiked at 250ppb level. Acetone, 2-Butanone, 2-Hexanone, Carbon disulfide, 2-Chloroethyl vinyl ether, 4-Methyl-2-pentanone, Vinyl acetate & Iodomethane are spiked at 100ppb level.

INTERNAL STANDARD ASSOCIATION / QUANT ION TABLE

COMPOUND	QUANT MASS	* I.S.	COMPOUND	QUANT MASS	* I.S.
			Dibromomethane	93	1
*Fluorobenzene **(1)**	96		1,1,2-Trichloroethane	83	2
*Chlorobenzene-d5 **(2)**	117		1,2,3-Trichloropropane	110	2
*1,4-Dichlorobenzene-d4 **(3)**	152		Hexachlorobutadiene	225	3
			Isopropylbenzene	105	2
Bromomethane	94	1	Isopropyltoluene	119	3
Chloroethane	64	1	Methylene Chloride	84	1
Vinyl chloride	62	1	Naphthalene	128	3
Chloromethane	50	1	Propionitrile	54	1
Dichlorodifluoromethane	85	1	n-Propylbenzene	91	3
Acetonitrile	41	1	Styrene	104	2
Allyl chloride	41	1	1,1,1,2-Tetrachloroethane	131	2
Trichlorofluoromethane	101	1	1,1,2,2-Tetrachloroethane	83	3
Benzene	78	1	Tetrachloroethene	166	2
Bromobenzene	156	3	Toluene	92	2
Bromochloromethane	128	1	1,2,3-Trichlorobenzene	180	3
Bromodichloromethane	83	2	1,2,4-Trichlorobenzene	180	3
Bromoform	173	2	1,2,4-Trimethylbenzene	105	3
n-Butylbenzene	91	3	1,3,5-Trimethylbenzene	105	3
sec-Butylbenzene	105	3	m-Xylene	91	2
tert-butylbenzene	119	3	p-Xylene	91	2
Carbon tetrachloride	117	1	o-Xylene	91	2
Chlorobenzene	112	2	Acrolein	56	1
Chloroform	83	1	Acrylonitrile	53	1
Chloroprene	53	1	Tetrahydrofuran	42	1
2-Chlorotoluene	91	3	MTBE	73	1
4-Chlorotoluene	91	3	Methacrylonitrile	41	1
Dibromochloromethane	129	2	Methyl methacrylate	41	1
1,2-Dibromo-3-chloropropane	157	3	Ethyl methacrylate	69	2
1,2-Dibromoethane	107	2	1,1,2-Trichlorotrifluoroethane	101	1
1,2-Dichlorobenzene	146	3	Cyclohexane	56	1
1,3-Dichlorobenzene	146	3	Methylcyclohexane	83	1
1,4-Dichlorobenzene	146	3	Methyl acetate	43	1
1,1-Dichloroethane	63	1	Carbon disulfide	76	1
1,2-Dichloroethane	62	1	Iodomethane	142	1
1,1-Dichloroethene	96	1	Vinyl acetate	43	1
cis-1,2-Dichloroethene	96	1	2-Chloroethyl vinyl ether	63	1
trans-1,2-Dichloroethene	96	1	Acetone	43	1
trans-1,4-Dichloro-2-butene	53	3	2-butanone	43	1
1,2-Dichloropropane	63	1	2-hexanone	43	2
1,3-Dichloropropane	76	2	Isobutyl alcohol	43	1
2,2-Dichloropropane	77	1	1,4-Dioxane	88	1
1,1-Dichloropropene	75	1	4-methyl-2-pentanone	43	1
cis-1,3-Dichloropropene	75	1	Dibromofluoromethane (S)	111	1
trans-1,3-Dichloropropene	75	2	1,2-Dichloroethane-d4 (S)	102	1
Ethylbenzene	91	2	Toluene-d8 (S)	98	2
1,1,1-Trichloroethane	97	1	Bromofluorobenzene (S)	95	2
Trichloroethene	95	1			

***I.S=internal Standard.**
S=surrogate.

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):	
Batch Number(s):	
Method:	**8260B/8270C (Circle One)**

QA/QC Item	Yes	No	NA	Second Level Review
1. Is the BFB/DFTPP tune performed every 12 hours and is the tuning criteria met?				
2. Are the RRFs and % RSDs within QC limits for appropriate analytes for the initial calibration? Check the retention times for compounds with the same spectra. Check compounds with different conc.(eg. m/p-xylene, ketones,etc.).				
3. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?				
4. Does the Continuing Calibration Standard (CCV) meet the criteria for the CCCs and SPCCs.				
5. Is the Method Blank run at the desired frequency and is its concentration for target analytes less than the MDLs?				
6. Are the LCS, MS, MSD within control limits and run at the desired frequency?				
7. Are all sample holding times met, analytes within calibration range, IS areas and surrogate recoveries within QC limits?				
8. Was the Method Blank, LCS, MS, MSD and samples loaded to the GCMS_LFSYS Tablespace within the Target DB Database?				

Comments on any "No" response:

Primary-Level Review: _____ Date: _____

Second-Level Review: _____ Date: _____

GC/ECD CHLORINATED ACID HERBICIDES BY EPA METHOD SW-846 8151A

SOP NUMBER: **SOP-208**

REVISION NUMBER: **13**

APPROVED BY:

SECTION MANAGER

QUALITY ASSURANCE MANAGER

EFFECTIVE DATE: **09/19/08**

DATE OF LAST REVIEW: **09/19/08**

GC/ECD
CHLORINATED ACID HERBICIDES
BY EPA METHOD SW-846 8151A

1.0 SCOPE AND APPLICATION

This SOP (based primarily on SW-846 Method 8000B/8151A) is used for the analysis of herbicide organic compounds in a variety of matrices (soils, sediments, waters, etc.). The analyses by these methods are clearly defined in the respective regulatory manuals. A good understanding of these methods is essential to the performance of each method. The normal laboratory list of analytes with their matrix (aqueous/non-aqueous) LCS limits are found attached in the appendix (page 10). Other compounds may be analyzed by this SOP as detailed in section 1.0 of SW-846 Method 8151A. Any questions left by this SOP should be answered by reading the methods, paying close attention to SW-846 8000B/8151A. If questions still remain unanswered, check with the Organic Manager, QA/QC Officer and/or Technical Director.

2.0 METHOD SUMMARY

After sample preparation using the appropriate extraction technique, the sample is introduced into the GC using direct injection. The analytes are separated in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the ECD. Analytes are identified and confirmed based on the retention time of known standards then quantitated relative to known standards using the external standard method.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Section 3.0 and tables 3-1 and 3-2 of the Empirical Laboratories Quality Assurance Manual include details concerning sample preservation, containers and handling of samples and extracts. All water and soil samples are stored in the appropriate walk-in cooler at a temperature of 1°C – 4.4°C. All extracts are stored in the Hobart in the Extraction lab at a temperature of 1°C – 4.4°C. Water samples have a holding time of 7 days from date of sampling for extraction. Soil samples have a holding time of 14 days from date of sampling for extraction (unless otherwise specified for the project). Extracts have a holding time of 40 days from extraction for analysis.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Section 3.0 of SW-846 Method 8151A details interferences and potential problems which may be encountered when dealing with herbicide analyses.

5.0. EQUIPMENT AND APPARATUS

5.1 GC's :

- 5.1.1 HP 5890/HP 5890 Series II/ Dual ECD - complete with temperature programmable gas chromatograph suitable for split/splitless injection.
- 5.1.2 Agilent 6890N/ Dual ECD - complete with temperature programmable gas chromatograph suitable for split/splitless injection.

5.2 Columns:
- 5.2.2 RTX-CLP(or equivalent): 30 m x 0.32 mm ID 0.5µm film thickness fused silica column.
- 5.2.3 RTX CLP II(or equivalent): 30 m x 0.32 mm ID 0.25 µm film thickness fused silica column.

5.3 Autosamplers:
- 5.3.1 HP 7673A/HP7673 autosamplers capable of reproducibility from one injection to another, proven by meeting QC and calibration criteria.
- 5.3.2 HP 7683/7683 autosamplers capable of reproducibility from one injection to another, proven by meeting QC and calibration criteria.

5.4 Acquisition Software: HP Chemstation system is interfaced to the GC. The system acquires and stores data throughout the chromatographic program.

5.5 Data Processing Software: Target DB Windows NT data system is interfaced to the HP Chemstation. The system accepts, processes, and stores acquired data.

6.0 REAGENTS

6.1 Hexane - pesticide quality or equivalent.

6.2 Ether - ultra-resi analyzed for organic residual analysis (Baker #9259-02 or equivalent)

6.3 Iso-Octane - for pesticide residual analysis (Fisher #0-297 or equivalent)

6.4 Methanol - suitable for use in gas chromatography (Omnisolv MX0484-1 or equivalent)

6.5 Diazomethane – as prepared in SOP-328.

6.6 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label and recorded in the GC standards logbook. The date they are opened is noted on the label and recorded in the GC standards logbook along with their lot number and vendor and each is given a sequential number. Each standard that is prepared is recorded in the GC standards logbook and given a sequential number. The following are noted in the logbook: standard makeup, solvent used, date received, date opened, date prepared, expiration date and analyst. Each standard label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. All stocks and standards are stored in the refrigerator at a temperature of 1°C – 4.4°C 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 temperature logbook. See the GC standards log book for makeup of standards. NOTE: If standards are not purchased in the methyl ester form, they must be esterified before use **(See SOP-328)**. If purchased as methyl esters, the indicated conversion factor must be applied to convert methyl ester concentrations to acid concentrations.

6.7 The Surrogate Standard, 2,4-Dichlorophenylacetic acid, DCAA is purchased in a solution in acetone. The date that it is received is noted on the label and recorded in the GC standards logbook. The date opened is noted on the label and recorded in the GC standards logbook along with the lot number and vendor and is given a sequential number.

6.8 Calibration Standard Preparation

6.8.1 Herbicide intermediate standard mix – See the GC Standards Logbook for preparation of standards. The solution is transferred into a labeled, teflon-lined, screw-capped vial and stored in the refrigerator at a temperature of 1°C - 4.4°C for up to 6 months.

Analyte (Methyl Ester)	Conversion Factor
2, 4-D	0.9407
2, 4-DB	0.9470
2, 4, 5-TP (Silvex)	0.9506
2, 4, 5-T	0.9479
Dalapon	0.9107
Dicamba	0.9407
Dichloroprop	0.9434
Dinoseb	0.9452
MCPA	0.9346
MCPP	0.9390

6.8.2 Calibration standards are prepared from the above intermediate standard at a minimum of five concentrations. Standards are prepared fresh each time they are needed and discarded after use. Note: standards prepared from an alternate source or lot than the calibration stock standards are used for all LCS spikes and the MS/MSD for full list spikes. See the GC Standards Logbook for preparation of calibration curve standards.

6.8.3 The Initial Calibration Verification (ICV) intermediate standard is prepared from vendor stock standards in the same manner as the Calibration intermediate standard above and is stored in the refrigerator at a temperature of 1°C – 4.4°C for up to 6 months. The ICV standard is then prepared at a concentration near the midpoint in the same manner as the Calibration standards above.

7.0 PROCEDURE

The GC/ECD should be primed by injecting a herbicide standard at the highest concentration of the calibration curve. Inject this prior to beginning initial or daily calibration.

7.1 Chromatographic conditions:

7.1.1 GC	ECD1 or 3
Purge on	0.50 min.
Injector temperature	210°C
Column flow	5-6 mL/min
Initial column temperature	50°C for 7.0 minutes
Initial temperature ramp	20°C/min
Second column temperature	150°C for 1.0 minute
Second temperature ramp	5°C/min
Third column temperature	230°C for 0.5 min
Final temperature ramp	20°C/min
Final column temperature	270°C for 0.0 minutes

7.2 Calibration - (See SW-846 Method 8000B Section 7.4.2).

7.2.1 Initial Calibration - A five point calibration curve must be injected and analyzed for each analyte of interest. For most analytes a six point curve is used. Injection volume for standards and samples is equal to 2 µL using the same injection technique to introduce both standards and samples (use of auto-injectors makes this a constant). All calibration integrations must be evaluated and any manual integrations are documented by the inclusion of the chromatogram (which includes peak integrations) with the quantitation report. The percent relative standard deviation (RSD) of the calibration factor must be < 20% over the working range for each analyte of interest. When the 20% criteria is exceeded for an analyte, a linear calibration may be used if the correlation coefficient (***r***) is ≥0.995. Quadratic can be used if correlation coefficient is ≥0.99 Otherwise, a new standard curve should be prepared for each analyte that exceeded the criteria.

7.2.2 Initial Calibration Verification - A second source standard at the midpoint level concentration is used to check the validity of the curve. The standard recovery for all analytes must be between 85 and 115% (**80-120% for DOD QSM**). If the second source recovery is above 115%, it is possible that the main standard has deteriorated for that compound. That standard should be remade and reevaluated. If that does not correct the problem, the standard should probably be replaced and a new curve generated. If the second source recovery is below 85%, the second source standard may have deteriorated for that compound. The standard should be remade and reanalyzed. If this does not correct the problem, the standard should probably be replaced. All calibration integrations must be evaluated and any manual integrations are documented by the inclusion of the chromatogram (which includes peak integrations) with the quantitation report.

7.2.3 Continuing Calibration Verification (CCV) - A mid-level standard must be analyzed every ten samples and cannot exceed 15 percent difference (%D) from the average calibration factor of the calibration

curve. The 15% criteria may still be met if the targets are <30% and the average of the %Ds is less than 15% (the client is notified). A CCV must also be analyzed at the end of the analysis sequence. If the CCV fails at any point, GC maintenance may be necessary (see SOP-222), reanalysis may be required (for samples analyzed since the last valid CCV) and a corrective action report must be completed. Alternatively, analytes may be flagged depending on their concentration and the status of the analyte in the CCV standard. No reanalysis is necessary if the analyte is undetected in the samples and recovered high in the CCV. All calibration integrations must be evaluated and any manual integrations are documented by the inclusion of the chromatogram (which includes peak integrations) with the quantitation report. Samples are then quantitated against the initial calibration curve. **For DOD QSM projects, CCVs are analyzed at the beginning and end of the sequence and after every 10 field samples. The CCVs should be within ± 20% difference.**

7.3 RT Windows - Retention time criteria set forth in SW-846 method 8000B section 7.6 are used to set retention time windows. New in-house retention time windows are established after every major change to the system (new column or temperature program). If the established retention time window is less than ± 0.03 minutes, the window defaults to ± 0.03 minutes. Retention times are updated with the first CCV of the day or the mid-level standard of the curve if samples are analyzed directly after a curve.

7.4 LCS - The LCS is analyzed 1/20 samples (1 per extraction batch - up to 20 samples - at varied concentrations). The LCS is spiked using an alternate lot or source from the calibration stock standards. See section 9.2 below for criteria and corrective action.

7.5 Method Blank - Method blanks are extracted at a minimum of 1 per extraction batch - up to 20 samples. See section 9.3 below for criteria and corrective action.

7.6 Samples - Prior to analysis, the samples are prepared for chromatography using SW-846 method 8150B/8151A.

7.7 Data Reduction/Evaluation - Each sample analysis sequence is documented in the run logbook for the instrument. After the sample has been analyzed, the data is processed through the Chemserver 4920 data system. Quantitative measurements are performed as described in SW-846 8000B section 7.10. Rounding is performed using CLP odd/even rounding rules. The following must be checked to determine if the sample will need any reanalysis, cleaning or dilution. Formal data evaluation is detailed in SOP-216 (documented using the USACE Analyst Data Review Checklist for USACE projects).

7.7.1 Analyte concentration after rounding to 2 significant figures must be within the range of the calibration curve. If an analyte exceeds the curve, a dilution must be performed and the next sample must be checked for carryover.

7.7.2 Surrogate standard recovery of DCAA must be checked to make sure it is within limits. See 9.1 below for corrective action. **For DOD QSM projects, DOD limits are used.**

7.8 Identification [See SW-846 method 8000B section 7.9]

7.8.1 Single peak components are identified by retention time on a primary column with confirmation by

retention time on a secondary column. Which column is used for primary/confirmation is determined by the chromatography in the region of the compound. If both columns are equivalent, the highest concentration is reported. **Refer to SOP-224 for guidance with manual integrations. A before and after chromatogram with analyst's initials, date and reason must be included with the data for DOD QSM projects,**

7.8.1.1 Due to co-elution of certain compounds confirmation for all analytes can not be achieved. The percent difference (%D) for compounds, that confirmed from the primary column, on the confirmation column is 40%. The analyst must use experience and judgment to decide if the compound is there. Flag data with a "P" (**"J" for DOD projects**) if the results of the two columns differ by more than 40%. Report the higher of the two results unless overlapping peaks are causing erroneously high results.

7.8.1.2 If a compound is outside of its window on one column but in the window on the other column, the analysts will need to use their judgment or seek guidance from the organic lab manager or another experienced analyst to decide if the analyte is there.

8.0 Calculations:

8.1 Calculate the calibration factor (CF) for each analyte at each concentration as:

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

8.2 The mean CF is calculated as follows:

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

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

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

$$RSD = \frac{SD}{} X\ 100$$

CF

8.4 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.

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

where CCV CF is the calibration factor from the analysis of the verification standard and mean CF is the average calibration factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within ±15% for each analyte before any sample analyses may take place.

8.5 Concentration in water samples is calculated as follows:

[Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to µg/L.]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_x)(V_t)(D)}{(\overline{CF})(V_i)(V_s)}$$

where:

A_x	=	Area (or height) of the peak for the analyte in the sample.
V_t	=	Total volume of the concentrated extract (µL).
D	=	Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.
V_i	=	Volume of the extract injected (µL). The nominal injection volume for samples and calibration standards must be the same.
CF	=	Mean response factor from the initial calibration.
V_s	=	Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

8.6 Concentration in non-aqueous samples is calculated as follows:
[Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to µg/kg.]

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

where:
A_x, V_t, D, and CF are the same as for aqueous samples, and
W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

9.0 QUALITY ASSURANCE/QUALITY CONTROL/CORRECTIVE ACTIONS

9.1 Surrogates –Limits are determined by charting LCSs and method blanks. Surrogate standard recovery must be checked to determine if it is within limits. Corrective action must be taken when the surrogate is out of limits for a sample. Generally, the first action for herbicides is to re-esterify the sample and reanalyze. A corrective action form must be filled out and given to the organic lab manager within 24 hours. The organic lab manager will then make suggestions as to what further action should be taken, for example: sample may need to be reextracted or flagged on the report for a QC problem.

9.2 LCS Sample - The LCS is extracted 1/20 samples (every extraction batch - up to 20 samples - for USACE and GE projects). To prepare the LCS, a blank is spiked with standards prepared from alternate sources or lots than the calibration stock standards, and then extracted. The recoveries are tabulated to generate control charts and limits. See the LCS report form in the appendix for the laboratory-generated limits. If limits have not yet been generated, the limits default to those calculated from 8151A Table 4 (Mean ± 3SDs). If the LCS compound has a recovery above the upper limit, but the same compound is not detected in any of the batch samples, no corrective action is required. For all other situations, the LCS should be reanalyzed for the failed analytes only. If the second analysis fails, all associated samples should be reextracted/reanalyzed for the failed analytes only or the analytes should be flagged on the report. **For DOD QSM projects, DOD limits are used.**

9.3 Method Blanks - The concentration of all method target analytes should be below the MDL **(<RL, common laboratory contaminates; < ½ RL, all other compounds or client/authority specified)** for each method target analyte. If contamination exceeds the MDL, the following corrective actions must be taken. The first step is to assess the effect on the samples. If an analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps should be taken to find/reduce/eliminate the source of this contamination in the method blank. If an analyte is found in the method blank and some, or all, of the other batch samples, corrective action is required. The source of contamination must be investigated and appropriate action taken and documented to find/reduce/eliminate the source of this contamination. The method blank, and

any samples containing the same contaminant, would likely be reextracted/reanalyzed. If a contaminant is found in the method blank and the samples, the compound concentration must be flagged with a 'B' on the final report unless the concentration is greater than 5x that found in the method blank.

9.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD (for full list spikes, the LCS standard is used for this spike). Criteria for the MS/MSD are the same as the LCS limits with an RPD criteria of less than 25%. Samples, which do not meet these criteria due to matrix, must be flagged on the final report for QC problems. Generally, batch control is not based on MS/MSD results unless general method failure is determined to be the problem. In that case, the samples and associated QC would be reanalyzed for the failed analytes only. MS data evaluation must include the consideration of the following factors. **For DOD QSM projects, DOD limits are used.**

9.4.1 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.

9.4.2 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.

9.4.3 MS vs. MSD - If a spiked compound has a similar problem in both the MS and MSD and is not traced to a method problem, no further action may be necessary other than the generation of a corrective action report to document the problem.

9.4.4 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.

9.5 Documentation of Capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

10.0 HEALTH AND SAFETY

10.1 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.

10.2 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.

10.3 MSDS are available for all reagents and standards, which have been purchased. These are located in the office next to the technical director.

10.4 Diazomethane is a carcinogen and may explode under certain conditions. See Method 8151 for details in handling diazomethane.

11.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

Please see Waste Disposal SOP-405 for proper disposal of waste from this sample preparation process. 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.

12.0 REFERENCES

12.1 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method *8151*

12.2 USACE EM 200-1-3, 02-2001; Appendix I; Shell for Analytical Chemistry Requirements

12.2 DOD, Quality Systems Manual for Environmental Laboratories, Ver. 3, Jan. 2006.

13.0 EXAMPLE FORMS

Examples of the water LCS report sheet and the USACE analyst data review checklist are located in the appendix.

APPENDIX

FORM 3
WATER HERBICIDE LAB CONTROL SAMPLE

Lab Name: ELAB, Inc. Contract: QC

Lab Code: NA Batch No.: NA SAS No.: NA SDG No.: QC.LCS

Matrix Spike - Client Sample No.: LCS

SPIKE COMPOUND	SAMPLE ADDED (µg/L)	LCS CONCENTRATION (µg/L)	LCS CONCENTRATION (µg/L)	QC. % REC #	LIMITS REC.
2,4-D	10.00	NA	10.00	100	48-214
2,4-DB	10.00	NA	10.00	100	60-126
2,4,5-TP(Silvex)	1.000	NA	1.000	100	68-166
2,4,5-T	1.000	NA	1.000	100	42-226
Dalapon	25.00	NA	25.00	100	40-110
Dicamba	1.000	NA	1.000	100	69-159
Dichloroprop	10.00	NA	10.00	100	60-110
Dinoseb	5.000	NA	5.000	100	D-110
MCPA	1000	NA	1000	100	24-117
MCPP	1000	NA	1000	100	35-131

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

Spike Recovery: 0 out of 10 outside limits

COMMENTS:_____

ANALYST REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: 8151A

QA/QC Item	Yes	No	NA	2nd Level Review
A. Initial Calibration				
1. Does the curve consist of five Calibration Standards?	___	___	___	_____
2. Is the low standard near, but above the MDL?	___	___	___	_____
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 criteria been met?	___	___	___	_____
C. Continuing Calibration				
1. Are the Continuing Calibration Verification (CCV) standards analyzed every 10 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. Are all samples with concentrations > the highest standard used for initial calibration diluted and reanalyzed?	___	___	___	_____
3. Are all compounds identified on the primary column confirmed on the secondary column?	___	___	___	_____
4. Are Surrogate recoveries within QC limits?	___	___	___	_____

ANALYST REVIEW CHECKLIST

QA/QC Item	Yes	No	NA	2nd Level Review
E. QC Samples				
1. Is the Method Blank extracted at the desired frequency and is its concentration for target analytes less than the MDLs?	____	____		_____
2. Is the LCS extracted at the desired frequency and are the percent recoveries within QC limits?	____	____		_____
3. Is the Matrix Spike/Matrix Spike Duplicate extracted at the desired frequency and are the percent recoveries/RPDs 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 integration checked by a second reviewer to verify why they were performed?	____	____		_____

Comments on any "No" response:

_____
_____
_____
_____
_____
_____
_____

Analyst: _____ Date: _____
Second-Level Review: _____ Date: _____

GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD)

ORGANOCHLORINE PESTICIDES/POLYCHLORINATED BIPHENYLS (PCB) BY

EPA METHOD 608/608.2 AND

SW846 METHOD 8081A/8082

SOP NUMBER: **SOP-211**

REVISION NUMBER: **19**

APPROVED BY:

SECTION MANAGER

QUALITY ASSURANCE OFFICER

EFFECTIVE DATE: **09/10/08**

Date of Last Review: **09/10/08**

GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD) ORGANOCHLORINE PESTICIDES/POLYCHLORINATED BIPHENYLS (PCB) BY EPA METHOD 608/608.2 AND SW-846 METHOD 8081A/8082

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure, SOP, (based primarily on SW-846 Methods 8000B/8081A/8082) is used for the analysis of Pesticide/PCB organic compounds in a variety of matrices (soils, sediments, waters, etc.). Methods SW-846 8082, *Federal Register* Method 608/608.2 and CLP Method for Pesticides have also been used in the development of this SOP. The analyses by these various methods are clearly defined in the respective regulatory manuals. A good understanding of these different methods is essential to the performance of each method. The normal laboratory list of analytes with their LCS limits is found attached in the appendix. Other compounds may be analyzed by this SOP as detailed in section 1.0 of SW-846 Methods 8081A/8082. Any questions left by this SOP should be answered by reading the methods, paying close attention to SW-846 8000B/8081A/8082, EPA 608/608.2 and CLP. If questions still remain unanswered, check with the Organic Lab Manager, Quality Assurance Officer and/or Technical Director.

2.0 METHOD SUMMARY

After sample preparation using the appropriate extraction technique, the sample is introduced into the GC using direct injection. The analytes are separated in the gas chromatograph by a combination of the temperature program and the capillary column. The analytes are then detected by the ECD. Pesticide analytes are identified and confirmed based on the retention time of known standards. PCB and multi-component pesticide analytes are identified based on pattern recognition. Analytes are quantitated relative to known standards using the external standard method.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Section 3.0 and tables 3-1 and 3-2 of the Empirical Laboratories, LLC Quality Assurance Manual includes details concerning sample preservation, containers and handling of samples and extracts. All water and soil samples are stored in the appropriate walk-in cooler in sample storage at a temperature of 1°C – 4.4°C. All extracts are stored in the Hobart in the Extraction laboratory at a temperature of 1°C – 4.4°C. Water samples have a holding time of 7 days from date of sampling to extraction. Soil samples have a holding time of 14 days from date of sampling to extraction (unless otherwise specified for the project). Extracts have a holding time of 40 days from extraction to analysis.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Section 3.0 of SW-846 Methods 8081A/8082 details interferences and potential problems which may be encountered when dealing with pesticide/PCB analyses. Please see sample clean-up SOPs (307, 308, 309, 330, and 334) to evaluate possible clean-up options for any encountered interferences.

5.0. EQUIPMENT AND APPARATUS

5.1 GC's:

5.1.1 Agilent 6890N- complete with temperature programmable gas chromatograph suitable for split/splitless injection.

5.2 Columns:

5.2.1 RTX-CLP (or equivalent): 30 meter x 0.32 mm ID x 0.5 µm film thickness fused silica column.

5.2.2 RTX-CLP II (or equivalent): 30 meters x 0.32 mm ID x 0.5 µm film thickness fused silica column.

5.3 Autosamplers:

5.3.1 Agilent 7683 autosamplers capable of reproducibility from one injection to another, proven by meeting QC and calibration criteria.

5.4 Acquisition Software: HP Chemstation system is interfaced to the GC. The system acquires and stores data throughout the chromatographic program.

5.5 Data Processing Software: Target DB Windows NT data system is interfaced to the HP Chemstation. The system accepts, processes and stores acquired data.

6.0 REAGENTS

6.1 Hexane - pesticide quality or equivalent.

6.2 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label and recorded in the GC standards logbook. The date they are opened is noted on the label and recorded in the GC standards logbook along with their lot number and vendor and given a sequential number. Each standard that is prepared is recorded in the GC standards logbook and given a sequential number. The following are noted in the logbook: standard makeup, solvent used, date received, date opened, date prepared, expiration date and analyst. Each standard label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. All stocks and standards are stored in the refrigerator at a temperature of 1°C-4.4°C from the date they are received/prepared. The refrigerator and freezer temperature is monitored daily with an annually calibrated thermometer and recorded with calibration correction in the GC refrigerator temperature logbook. See the GC standards log book for makeup of intermediate and calibration standards.

6.2.1 The Initial Calibration Verification (ICV) intermediate standard is prepared from the vendor stock standards in the same manner as the Calibration intermediate standards above and is stored in the refrigerator at a temperature of 1°C-4.4°C for up to 6 months. The ICV standard is then prepared at a concentration near the midpoint in the same manner as the Calibration standards above.

7.0 PROCEDURE

The GC/ECD should be primed by injecting a pesticide standard at 100 µg/L and/or PCB standard at 10,000 µg/L, 10 times more concentrated than the mid-level standard. Inject this prior to beginning initial or daily calibration.

7.1 Chromatographic conditions:

7.1.1 ZB MR1/MR2 columns:

GC	ECD3
Purge on	0.50 min.
Injector/Detector temperature	250/350°C
Column flow	@3.4 mL/min
Initial column temperature	100°C for 0.5 minutes
Temperature ramp	15°C/min
Intermediate column temperature	220°C for 5.0 minutes
Second Temperature Ramp	35°C/min
Final Column Temperature	340°C for 2.0 minutes

7.1.2 RTX-CLP/RTX CLPII columns:

GC	ECD4
Purge on	0.50 min.
Injector/Detector temperature	200/330°C
Column flow	@2.4 mL/min
Initial column temperature	110°C for 0.5 minutes
Temperature ramp	15°C/min
Final column temperature	320°C for 2.0 minutes

7.2 Eval Mix – Before pesticide calibration and/or sample analysis, a degradation check standard (evaluation mix) of endrin and 4,4-DDT must be injected. Degradation of either compound must not exceed 15 percent. If 15 percent degradation is exceeded, then corrective action must be taken (GC system maintenance, see SOP-222).

7.3 Calibration - (See SW-846 Method 8000B Section 7.4.2).

7.3.1 Initial Calibration – For single component pesticides and surrogates, a six point calibration is injected and analyzed for each analyte of interest. For Toxaphene and Technical Chlordane a single point standard is analyzed unless they are expected then a five point calibration is injected and analyzed. Injection volume for standards and samples is equal to 2 µL using the same injection technique to introduce both standards and samples (use of auto-injectors makes this a constant). All calibration integrations must be evaluated and any manual integrations are documented by the inclusion of the chromatogram (which includes peak integrations) with the quantitation report. The percent relative standard deviation (RSD) of the calibration factor must be <20% over the working range for each analyte of interest. When the 20% criteria is exceeded for an analyte, a linear calibration may be used if the correlation coefficient (r) is $\geq$0.995, $\geq$0.99 for quadratic with 6 points. Otherwise, a new standard curve should be prepared for each analyte that exceeded the criteria.

Initial calibration for Aroclors may be accomplished by using a six-point curve that contains Aroclors 1016 and 1260. The mixture of these two Aroclors contains many of the peaks represented in the other five Aroclor mixtures(1221, 1232, 1242, 1248 & 1254). The curves for Aroclors 1016 and 1260 are used to show the linearity of the detector and can be used to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. The 1016/1260 curve may also be used to quantitate any 1016 or 1260 hits that may be seen in the samples. The analyst has the choice of running a curve for the

other five Aroclor mixtures or when the calibration factor for the curve for 1016/1260 is <20% a single point calibration at the midpoint level concentration can be used for the other Aroclors. The injection procedure for Aroclors is the same as single component pesticides. The percent relative standard deviation (RSD) of the calibration factor must be <20% but this applies to the average of the quantitation peaks. When the 20% criteria is exceeded for an analyte, a linear calibration may be used if the correlation coefficient factor (r) is ≥0.995, ≥0.99 for quadratic with 6 points. Otherwise, a new standard curve should be prepared for each analyte that exceeded the criteria.

7.3.2 Initial Calibration Verification - A second source standard at the midpoint level is used to check the validity of the curve. The standard recovery for all analytes must be between 85 and 115%(**80-120% DOD QSM Ver.3**). If the second source recovery is above 115%, it is possible that the main standard has deteriorated for that compound. That standard should be remade and reevaluated. If that does not correct the problem, the standard should probably be replaced and a new curve generated. If the second source recovery is below 85%, the second source standard may have deteriorated for that compound. This standard should be remade and reanalyzed. If this does not correct the problem, the standard should be replaced. All calibration integrations must be evaluated and any manual integrations are documented by the inclusion of the chromatogram (which includes peak integrations) with the quantitation report.

7.3.3 Continuing Calibration Verification (CCV) - A mid-level standard must be analyzed every 12 hours (not to exceed 20 samples and cannot exceed 15 percent difference (%D), (**at the beginning and end of sequence and after every 10 field samples, 20%D no average DOD QSM Ver.3**) from the average calibration factor of the calibration curve. A CCV must also be analyzed at the end of the analysis sequence. If a CCV fails, GC maintenance may be necessary (see SOP-222), reanalysis may be required (for samples analyzed since the last valid CCV) and a corrective action report must be completed. If none of the failed target compounds exceed 30% D and the average of all the %Ds are < 15% then the CCV may be used without any further corrective action. Alternatively, analytes may be flagged depending on their concentration and the status of the analyte in the mid-level standard. No reanalysis is necessary if the analyte is undetected in the samples and recovered high in the CCV. All calibration integrations must be evaluated and any manual integrations are documented by the inclusion of the chromatogram (which includes peak integrations) with the quantitation report. Samples are then quantitated against the initial calibration curve.

7.4 RT Windows - Retention time criteria set forth in SW-846 method 8000B section 7.6 are used to set retention time windows. New in-house retention time windows are established after every major change to the system (new column or temperature program) and at initial calibration using the midpoint standard RTs. If the established retention time window is less than +/-0.03 minutes, the window defaults to +/-0.03 minutes. Retention times are updated with the first CCV of the day or the mid-level standard of the curve if samples are analyzed directly after a curve.

7.5 Laboratory Control Sample (LCS) - The LCS is extracted 1 per extraction batch of up to 20 samples. The LCS is spiked with standards prepared from an alternate vendor or lot number than the calibration standards. See section 8.2 below for criteria and corrective action.

7.6 Method Blank - Method blanks are extracted at a minimum of 1 per extraction batch – up to 20 samples. See section 9.3 below for criteria and corrective action.

7.7 Samples - Prior to using Method 608, SW-846 8081A/8082, or CLP (pesticide method) the samples are prepared for chromatography using the appropriate sample preparation and clean up methods (generally SW-846 methods 3510, 3520, 3540, 3640, 3550, 3580, EPA method 608 or CLP).

7.7.1 Example of a sequence run log:

1-Primer A/B Mix-1000 or Primer PCB-10,000
2- EVAL Mix (Pest only)
3- CCV A/B Mix
4- CCV Toxaphene (single point)
5-CCV Chlordane (single point)
6- CCV PCB 1660
7- Method Blank
8-LCS A/B Mix
9-LCS PCB
10-Sample
11-Sample
12-Sample
13-Sample
14-Sample
15-Sample
16-Sample
17-Sample
18-Sample
19-Sample
20-Sample
21-Sample-MS
22-Sample-MSD
23-Sample
24-Sample
25-Sample
26-Sample
27-Sample
28-Sample
29- CCV A/B Mix
30-CCV PCB

7.8 Data Reduction/Evaluation - Each sample analysis sequence is documented in the run logbook for the instrument. After the sample has been analyzed, the data is processed through the Target DB Windows NT data system. Quantitative measurements are performed as described in SW-846 8081A section 7.5.6. Rounding is performed using CLP odd/even rounding rules. The

following must be checked to determine if the sample will need any reanalysis, cleaning or dilution. Formal data evaluation is detailed in SOP-216 (documented using the USACE Analyst Data Review Checklist for USACE projects).

7.8.1 Analyte concentration after rounding to 2 significant figures must be within the range of the calibration curve. If an analyte exceeds the curve, a dilution must be performed and the next sample must be checked for carryover. Any dilution should keep the concentration of the analyte in question within the mid-range to the top half of the curve.

7.8.2 If the sample shows signs of sulfur contamination in the time range where sulfur compounds elute a sulfur cleanup is required [see SOP-307].

7.8.3 If the sample has extraneous peaks eluting in the chromatogram an acid cleanup is required for PCB samples and may be applicable for certain pesticides, (acid clean-up may be required for all PCB samples, check with your supervisor), [see SOP-308].

7.8.4 If the sample has extraneous peaks that are not removed by acid (PCB samples) and is not a sulfur interference, a florisil cleanup is recommended [see SOP-309]. A silica cleanup may also be used [see SOP-331].

7.8.5 Surrogates – Control and warning limits are determined by using all blank spikes in the calculation, (LCSs and method blanks). All limits used are generated in-house or client specified (with the exception that if the in-house limit's highest lower limit would be 90 and the lowest upper limit would be 110 examples: **98**-120 would be **90**-120 or 45-**75** would be 45-**110**). Surrogate standard recovery must be checked to determine if it is within these limits. Two surrogates are added to each sample for pesticides. Only one surrogate, DCB, is added to each PCB sample. Corrective action should be evaluated when any surrogate(s) is outside the action limits for a sample. When only one surrogate for pesticides exceed control limits on the primary and/or confirmation column corrective action may not be required. If both surrogates for pesticides (or DCB for PCB analyses) exceed control limits on the primary and/or confirmation column corrective action is required. A corrective action form should be filled out and given to the organic lab manager when both surrogates for pesticides (or DCB for PCB analyses) are outside the action or warning limits. The organic lab manager will then make suggestions as to what action should be taken, for example: the sample may need to be reanalyzed, reextracted, or flagged on the report for a QC problem. **DOD limits will be used for DOD QSM Ver. 3 projects.**

	WATER	SOIL/SEDIMENT	WIPES
Surrogate	In-House	In-House	In-House
TCMX	25-120	30-120	80-143
DCB	25-130	35-140	73-142

7.9 Identification/Quantitation [See SW-846 method 8081A section 7.6 or method 8082 sections 7.7-7.9].

7.9.1 Single peak components are identified by retention time on a primary column with confirmation by retention time on a secondary or confirmation column. Which column is used for primary/confirmation is determined by the chromatography in the region of the compound. The control limit for percent difference (%D) of compounds that confirmed from the primary column on the confirmation column is 40%. If both columns are equivalent, the highest concentration is reported. If a compound result is >40% difference, it should be flagged with a "P". (**"J" flagged for DOD QSM projects**)

7.9.1.1 Due to coelution of certain compounds confirmation for all analytes may not be achieved. The analyst must use experience and judgment to decide if the compound is there. If a call is made, the data should be flagged appropriately.

7.9.1.2 If a compound is outside of it's window on one column but in the window on the other column, the analyst will need to use their judgment or seek guidance from the organic lab manager or another experienced analyst to decide if the analyte is there.

7.9.2 Multi-peak components (PCB's, Toxaphene and Technical Chlordane) are identified by pattern recognition using an on scale standard chromatogram to compare to an on scale sample chromatogram enabling the analyst to judge whether the sample pattern matches a standard pattern. Confirmation of multi-peak components is required by the method and may be accomplished in several ways. If the sample is from a source known to contain specific Aroclors then this information may be used as a confirmation. Documentation of this approach must meet the requirements outlined in Sec. 7.7.3 of SW-846 Method 8082. Another approach is to use a column of dissimilar stationary phase and compare the pattern to a known Aroclor standard. Finally if the concentration is high enough GC/MS may be used as confirmation.

7.9.2.1 Generally, five unique peaks representing the full range of the multi-peak component are used in the quantitation of the multi-peak components. Note: for USACE projects, five peaks are necessary for the quantitation of multi-peak components.

7.9.2.2 Multi-peak components that still have matrix interference after appropriate sample cleanup steps have been taken may need to be hand calculated using peaks that do not have interference. This should be brought to the organic lab manager's attention.

7.9.2.3 Multi-peak components that exhibit a weathered pattern may need to be hand calculated by the analyst. The analyst will need to use peaks that exhibit the full range of weathering. The number of peaks used to quantitate the multi-peak component will depend on the analyst's judgment of what it will take to achieve the truest concentration of the component. This should be brought to the organic lab manager's attention.

7.9.3 Quantitation – Once a compound has been identified qualitatively, the concentration must then be quantitated. If the RSD of the compound's response factor is 20% or less, then the concentration may be determined using the mean calibration factor, CF, from the initial calibration data. Otherwise, the analyst must use either a calibration curve or a

non-linear calibration model such as polynomial equation for quantitation. Calculations follow in Section 8.0. **Refer to SOP-224 for guidance for manual integrations**

8.0 Calculations:

8.1 Calculate the calibration factor (CF) for each analyte at each concentration as:

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

8.2 The mean CF is calculated as follows:

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

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

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

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

8.4 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.

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

where CCV CF is the calibration factor from the analysis of the verification standard and mean CF is the average calibration factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within ±15% for each analyte before any sample analyses may take place.

8.5 Concentration in water samples is calculated as follows:
[Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to µg/L.]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(\text{A}_x)(V_t)(\text{D})}{(\overline{CF})(\text{V}_i)(V_s)}$$

where:

A_x = Area (or height) of the peak for the analyte in the sample.
V_t = Total volume of the concentrated extract (µL).
D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.
V_i = Volume of the extract injected (µL). The nominal injection volume for samples and calibration standards must be the same.
CF = Mean response factor from the initial calibration.
V_s = Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

8.6 Concentration in non-aqueous samples is calculated as follows:
[Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to µg/kg.]

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(\text{A}_x)(V_t)(\text{D})}{(\overline{CF})(\text{V}_i)(\text{W}_s)}$$

where:
A_x, V_t, D, and CF are the same as for aqueous samples, and

W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

9.0 QUALITY ASSURANCE/QUALITY CONTROL/CORRECTIVE ACTIONS

9.1 Surrogates - All samples and QC are spiked with surrogates. The surrogate recoveries from method blanks and LCS are charted to generate control limits and charts for diagnostic purposes. 8000B See section 7.8.5 above for criteria and corrective action.

9.2 LCS Sample - The LCS is extracted with every extraction batch - up to 20 samples. To prepare the LCS, a blank is spiked with standards prepared from an alternate vendor or lot number than the calibration standards. The recoveries are charted to generate control charts and limits. See the LCS report form in the appendix for the laboratory generated limits. These limits default to method limits if generated limits are wider. If the LCS compound has a recovery above the upper limit, but the same compound is not detected in any of the batch samples, no corrective action is required. For all other situations, the LCS should be reanalyzed for the failed analytes only or results for that analyte should be flagged. If the second analysis fails, all associated samples should be reextracted/reanalyzed for the failed analytes only. **DOD limits will be used for DOD QSM Ver.3 projects.**

9.3 Method Blanks - The concentration of all method target analytes should be below the MDL **(<RL, common laboratory contaminates; < ½ RL, all other compounds or client/authority specified)** for each method target analyte. If contamination exceeds the requirement, the following corrective actions must be taken. The first step is to assess the effect on the samples. If an analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps should be taken to find/reduce/eliminate the source of this contamination in the method blank. If an analyte is found in the method blank and some, or all, of the other batch samples, then corrective action is required. The source of contamination must be investigated and appropriate action taken and documented to find/reduce/eliminate the source of this contamination. The method blank, and any samples containing the same contaminant, would likely be reextracted/reanalyzed. For the common laboratory contaminants, meeting the above requirements is not practical. Random cases of contamination are difficult to control, however, daily contamination is not acceptable and corrective action is essential. If a contaminant is found in the method blank and the samples, the compound concentration must be flagged with a 'B' on the final report unless the concentration is greater than 5x that found in the method blank.

9.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD (for 608 projects, 1 in 10 samples are spiked for MS). For project specified full list MS/MSDs, the LCS standard is used for spiking. Criteria for the MS/MSD are found in Table 5-3 of the Laboratory Quality Assurance Manual. Both the percent recoveries (%R) and relative percent differences (RPDs) are contained in this table (for project specified full list MS/MSDs, the

MS/MSD limits are the same as the LCS limits). Samples that do not meet these criteria due to matrix should be evaluated for placing a flag on the final report due to QC problems. The associated LCS results should be used to verify method performance (section 8.4.3 Method 8081A). MS data evaluation must include the consideration of the following factors.

9.4.1 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.

9.4.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was more than four 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. In this case, the MS/MSD may be evaluated as sample duplicates.

9.4.3 MS vs. MSD - If a spiked compound has a similar problem in both the MS and MSD and is not traced to a method problem, no further action may be necessary other than the generation of a corrective action report to document the problem.

9.4.4 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.

9.4.5 Documentation of capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

10.0 HEALTH, SAFETY, WASTE MANAGEMENT AND POLLUTION PREVENTION

10.1 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.

10.2 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.

10.3 MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves across from the Quality Assurance Officers cube.

10.4 Please see Waste Disposal, SOP-210 and SOP-405 for proper disposal of waste coming from this area within our laboratory. 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.

11.0 EXAMPLE FORMS

Examples of the water and soil LCS report sheet and the USACE analyst data review checklist are located in the appendix.

12.0 REFERENCES

1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III)*; Method 8081A, 8082

2. USEPA *Code of Federal Requlations, 40, CH 1,PT 136;* Method 608, 608.2; APX-B

3. USEPA *Contract Laboratory Program(CLP) for Organics ILM04.2; ILM04.3*

13.0 DEFINITIONS

Refer SOP-431 for a list of definitions.

APPENDIX

FORM 3
WATER PESTICIDE/PCB LAB CONTROL SAMPLE
(In- House)

Lab Name: Empirical Laboratories, LLC Contract:

Lab Code: NA Batch No.: NA SAS No.: NA SDG No.:

Matrix Spike - Client Sample No.: LCS

COMPOUND	SPIKE ADDED (µg/L)	SAMPLE CONCENTRATION (µg/L)	LCS CONCENTRATION (µg/L)	LCS % REC #	QC. LIMITS REC.
Aldrin	0.05000	NA	0.05000	100	25-110
alpha-BHC	0.1000	NA	0.1000	100	45-125
alpha-Chlordane	0.05000	NA	0.05000	100	50-125
beta-BHC	0.05000	NA	0.05000	100	50-130
4,4'-DDD	0.1000	NA	0.1000	100	55-130
4,4'-DDE	0.1000	NA	0.1000	100	45-130
4,4'-DDT	0.1000	NA	0.1000	100	50-140
Dieldrin	0.1000	NA	0.1000	100	55-130
delta-BHC	0.05000	NA	0.05000	100	40-135
Endosulfan I	0.1000	NA	0.1000	100	50-120
Endosulfan II	0.1000	NA	0.1000	100	55-135
Endosulfan sulfate	0.1000	NA	0.1000	100	55-130
Endrin	0.1000	NA	0.1000	100	40-150
Endrin aldehyde	0.1000	NA	0.1000	100	40-130
Endrin ketone	0.1000	NA	0.1000	100	60-130
gamma-BHC (Lindane)	0.1000	NA	0.1000	100	50-130
gamma-Chlordane	0.05000	NA	0.05000	100	50-125
Heptachlor	0.1000	NA	0.1000	100	35-125
Heptachlor epoxide	0.05000	NA	0.05000	100	50-130
Methoxychlor	0.5000	NA	0.5000	100	50-140
PCB-1248	10.00	NA	10.00	100	50-140

Column to be used to flag recovery values with an asterisk

* Values outside of QC limits

Spike Recovery: 0 out of 21 outside limits

COMMENTS: _____

FORM 3
SOIL PESTICIDE/PCB LAB CONTROL SAMPLE
(In-House)

Lab Name: Empirical Laboratories, LLC Contract:

Lab Code: NA Batch No.: NA SAS No.: NA SDG No.:

Matrix Spike - Client Sample No.: LCS

COMPOUND	SPIKE ADDED (µg/kg)	SAMPLE CONCENTRATION (µg/kg)	LCS CONCENTRATION (µg/kg)	LCS % REC #	QC. LIMITS REC.
Aldrin	5.0000	NA	5.0000	100	30-135
4,4'-DDT	10.000	NA	10.000	100	30-160
Dieldrin	10.000	NA	10.000	100	30-150
Endrin	10.000	NA	10.000	100	35-160
gamma-BHC (Lindane)	10.000	NA	10.000	100	30-145
Heptachlor	10.000	NA	10.000	100	40-150
PCB-1248	1000.0	NA	1000.0	100	50-150

Column to be used to flag recovery values with an asterisk

* Values outside of QC limits

Spike Recovery: 0 out of 7 outside limits

COMMENTS: _____

page 1 of 1 FORM III PEST

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):	
Batch Number(s):	
Method:	**8081A/8082**

QA/QC Item	Yes	No	NA	Second Level Review
A. Initial Calibration				
1. Did the evaluation mix pass criteria?				
2. Does the curve consist of at least five Calibration Standards?				
3. Is the low standard near, but above the MDL?				
4. 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 criteria been met?				
C. Continuing Calibration				
1. Are the Continuing Calibration Verification (CCV) standards analyzed every 20 samples or every 12 hours and at the end of the sequence?				
2. Are the % differences within QC limits for all analytes?				
D. Sample Analysis				
1. Did the evaluation mix pass criteria?				
2. Are all sample holding times met?				
3. Are all samples with concentrations > the highest standard used for initial calibration diluted and reanalyzed?				
4. For single peak analytes - are all compounds identified on the primary column confirmed on the secondary column?				
5. For multi-peak analytes - does the pattern of the analyte in the sample match the pattern of the standard?				

6. Are surrogate recoveries within QC limits? (one surrogate both columns) _____ _____ _____ _____

ANALYST DATA REVIEW CHECKLIST

E. QC Samples

1. Is the Method Blank extracted at the desired frequency and is its concentration for target analytes less than the MDLs? _____ _____ _____ _____
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 IDs and units checked for transcription errors? _____ _____ _____ _____
5. Are all manual integrations checked by a second reviewer to verify they were performed correctly? _____ _____ _____ _____

Comments on any "No" response:

_____

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

TOTAL ORGANIC CARBON (TOC)

SM5310C, SW846 METHOD 9060 AND LLOYD KAHN METHOD

SOP NUMBER: **SOP-221**

REVISION NUMBER: **7**

APPROVED BY:

SECTION MANAGER

TECHNICAL DIRECTOR

EFFECTIVE DATE: **10/27/08**

DATE OF LAST REVIEW: **10/27/08**

TOTAL ORGANIC CARBON (TOC)
BY SM5310C, SW846 METHOD 9060 AND Lloyd KAHN
METHOD "*DETERMINATION OF TOC IN SEDIMENT*"

I. SCOPE AND APPLICATION

This SOP describes the measurement of TOC by SM5310C, SW-846 Method 9060 and Lloyd Kahn Method for determination in soil /sediment matrix.

SM5310C is used to determine the concentration of organic carbon in source and drinking water, SW-846 Method 9060 is used to determine concentrations of carbon in saline waters, domestic and industrial wastes and SW846 Method 9060 is modified for soil determination and the L1oyd Kahn Method is used for determination of TOC in soil/sediment and solid matrices. SW846 Method 9060 and the Lloyd Kahn Method require quadruplicate analysis of samples, where as SM5310C requires a minimum of two analyses. These methods should be read over carefully by the analyst and any restrictions should be noted.

II. SUMMARY OF METHOD

The organic carbon is measured using an Shimadzu Total Organic Carbon Analyzer (aqueous samples) and an OI Analytical Solids TOC Analyzer model 1010 (soil/sediment samples). The Shimadzu instrument converts the organic carbon in a sample using wet chemical oxidation. The CO_2 formed is then measured by an infrared detector (replaces ultraviolet detector in SM 5310C). With the model 1010 Solids TOC analyzer, TOC is determined by acidifying a sample and heating it to 250°C to remove the TIC. The sample is then heated to 900°C to combust the remaining TOC. The resulting carbon dioxide from the TOC is detected by a non-disperse infrared (NDIR) detector that has been calibrated to directly display the mass of carbon dioxide detected. This mass is proportional to the mass of TOC in the sample.

The limit of detection for the water method is 0.50 mg carbon/L and the Limit of quantitation is 1.0 mg carbon/L. The limits of detection and quantitation with the soil method depends on the how many grams of sample is used for the analysis. For a 250 mg sample the limit of detection is 460 mg/kg and the limit of quantitation is 1600 mg/kg.

III. SAMPLING HANDLING AND PRESERVATION

3.1 Sampling and storage in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples. NOTE 1: A brief study performed in the EPA Laboratory

indicated that distilled water stored in new, one quart cubitainers did not show any increase in organic carbon after two weeks exposure.

3.2 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the lapse of time between collection of samples and start of analysis should be kept to a minimum. The holding time is 28 days for waters and soils with the exception of the Lloyd Kahn method soils, which requires a 14 day holding time. Also, samples must be kept cool (4°C) and protected from sunlight and atmospheric oxygen.

3.3 When water samples cannot be analyzed immediately, the sample is preserved by acidification to (pH </= 2) with HCl or H_2SO_4. Both water and soil samples are stored at 4°C.

IV. INTERFERENCES

4.1 WATER METHOD

4.1.1 Removal or carbonate and bicarbonate carbon by acidification and purging with purified gas results in the loss of volatile organic substances. The volatiles also can be lost during sample blending, particularly if the temperature is allowed to rise. Another important loss can occur if large carbon-containing particles fail to enter the needle used for injection. Filtration although necessary to eliminate particulate organic matter when only DOC is to be determined, can result in loss or gain of DOC, depending on the physical properties of the carbon-containing compounds and the adsorption of carbonaceous material on the filter, or its desorption from it. Check filters for their contribution to DOC by analyzing a filtered blank. Note that any contact with organic material may contaminate a sample. Avoid contaminated glassware, plastic containers, and rubber tubing. Analyze treatment, system, and reagent blanks.

4.1.2 This procedure is applicable only to homogenous samples which can be injected into the apparatus reproducibly by means of a pipette. The openings of the pipette limit the maximum size of particles which may be included in the sample.

4.2 SOIL METHOD

4.2.1 All materials must be routinely demonstrated to be interference –free under the analysis conditions by running blanks. Use high purity or purified reagents and gases to help minimize interference problems.

4.2.2 The infrared detector is sensitized to CO_2 and accomplishes virtually complete rejection of response from other gases that absorb energy in the infrared region.

V. DEFINITIONS

5.1 ANALYTICAL BATCH-The set of samples extracted /distilled/ or digested at the same time to a maximum of 20 samples.

5.2 CALIBRATION BLANK (CB)- A volume of reagent water in the same matrix as the calibration standards, but without the analyte.

5.3 CALIBRATION STANDARD (CAL)- A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

5.4 FIELD BLANK (FMB)- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, including exposure to a sample bottle holding time, preservatives, and all preanalysis treatments. The purpose is to determine if the field or sample transporting procedures and environments have contaminated the sample.

5.5 FIELD DUPLICATE (FD)- Two samples taken at the same time and place under identical circumstances which are treated identically throughout field and laboratory procedures. Analysis of field duplicates indicates the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures.

5.6 LABORATORY BLANK (LRB)- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, except that it is not taken to the sampling site. The purpose is to determine if the analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

5.7 LABORATORY CONTROL SAMPLE (LCS)- A solution prepared in the laboratory by dissolving a known amount of one or more pure compounds in a known amount of reagent water. Its purpose is to assure that the results produced by the laboratory remain within the acceptable limits for precision and accuracy. (This should not be confused with a calibrating standard, it must be prepared from a source other than the same source as the calibration standards).

5.8 LABORATORY DUPLICATE (LD)- Two aliquots of the same environmental sample treated identically throughout a laboratory analytical procedure. Analysis of laboratory duplicates indicates precision associated with laboratory procedures but not with sample collection, preservation, or storage procedures.

5.9 QUALITY CONTROL CHECK SAMPLE (QCS)- A sample containing analytes of interest at known concentrations (true value) of analytes. The QCS is obtained from a source external to the laboratory or is prepared from standards obtained from a different source than the calibration standards. The purpose is to check laboratory

performance using test materials that have been prepared independently from the normal preparation process.

5.10 METHOD DETECTION LIMIT (MDL)- The lowest level at which an analyte can be detected with 99 percent confidence that the analyte concentration is greater than zero.

VI. REAGENTS/STANDARDS

Store all reagents and standards according to recommendations. All standards should be stored away from light and at 4°C (± 2°C).

6.1 The laboratory reagent blank water used for TOC analysis is obtained from the Modulab Analytical water purification system in the analytical laboratory. **Boiling the water is not necessary as the method states.**

6.2 Potassium hydrogen phthalate, primary stock solution, 1000 mg/L: Dissolve 0.2128g of potassium hydrogen phthalate (primary standard grade) in 100.0 mL water.

6.3. Potassium hydrogen phthalate, standard solutions : A 100 mg/L standard is prepared by transferring 10 mL of the stock solution to a 100 mL volumetric flask and diluting to the mark with water. This solution is prepared on a daily basis.

6.4. The carbonate-bicarbonate solutions are not needed for this instrument.

6.5 Calibration Standards

1. For the water method, calibration standard is Potassium Hydrogen Phthalate. Standards are made from dilutions of the stock 1000 mg/L standard as follows:

   1.0 mg/L = 0.10 mL of 1000 mg/L -> 100 mL
   2.5 mg/L = 0.25 mL of 1000 mg/L -> 100 mL
   5.0 mg/L = 0.50 mL of 1000 mg/L -> 100 mL
   10.0 mg/L = 1.0 mL of 1000 mg/L -> 100 mL
   25.0 mg/L = 5.0 mL of 1000 mg/L -> 200 mL
   50.0 mg/L = 10.0 mL of 1000 mg/L -> 200 mL
   100 mg/L = 10.0 mL of 1000 mg/L -> 100 mL

   A low level standard curve must be run for drinking water samples with the standards made as follows:

   0.25 mg/L = 0.025 mL of 1000 mg/L -> 100 mL

0.50 mg/L = 0.050 mL of 1000 mg/L -> 100 mL
1.0 mg/L = 0.10 mL of 1000 mg/L -> 100 mL
1.5 mg/L = 0.15 mL of 1000 mg/L -> 100 mL
2.5 mg/L = 0.25 mL of 1000 mg/L -> 100 mL
5.0 mg/L = 0.50 mL of 1000 mg/L -> 100 mL
10.0 mg/L = 1.0 mL of 1000 mg/L -> 100 mL

2. The soil method the calibration standard is prepared by using an OI commercially prepared 30% carbon sucrose solution.

6.6 Laboratory Control Sample:

1. For the water method, the Laboratory Control Sample is normally made from a performance evaluation solution of which the true value is known. This solution is given a unique identifier.

2. For the soil method, the Laboratory Control Sample is made from a 30% sucrose solution which is made by weighing up 7.125 grams of EM Reagent Grade Sucrose and diluting to 10 mL with deionized water volumetrically.

6.7. Persulfate oxidation solution: This solution is made by dissolving 60g of sodium persulfate in DI water, adding 15 ml of phosphoric acid and diluting to 500 ml.

6.8 Phosphoric acid solution: Dilute 100 mL of concentrated 85% phosphoric acid in 500 mL of water. This is used for water.

6.9 Phosphoric acid solution 5%: Dilute 59 mL of concentrated 85% phosphoric acid in 1000 mL of water. This is used for soil.

VII. INSTRUMENTATION

7.1 The instrument used for the Water TOC analysis is a Shimadzu Total Carbon Analyzer. An OIC 1010 soil/sediment carbon analyzer is used for soil samples.

7.2 There is a Shimadzu autosampler which will hold 68 samples.

7.3 The corresponding data for each sample is obtained from the Shimadzu software for the water samples. The soil/sediment data are printed out at the organic GC printer.

VIII. AQUEOUS SAMPLE PROCEDURE

8.1 Wearing labcoat, gloves and safety glasses, the standards and check solutions should be taken out of the refrigerator and allowed to warm to room temperature. Also, remove samples from sample storage signing them out appropriately on the internal chain of custody form. Fresh acid and oxidation solutions should be poured into the appropriate containers on the front of the instrument.

8.2 Follow the instructions for operation of the instrument in Chapter 4, section 4.3 of the Shimadzu Model TOC-VWS User Manual. **See Appendix I. for Basic TOC start-up notes for analysis.**

8.3 **Following is a list outlining the order in which the samples should be run.** Each sample VOA vial should be numbered and its identity entered into the TOC schedule. Note: All blanks should be acidified to pH 2 to match the matrix of the samples analyzed.

1. 100 ppm
2. 50 ppm
3. 25 ppm
4. 10 ppm
5. 5.0 ppm
6. 2.5 ppm
7. 1.0 ppm
8. Method blank
9. LCS + 9 samples (including any sample QC
10. 25 ppm
11. 10 samples (including any sample QC)
12. 50 ppm

8.8 Insrument printouts are generated from the software. Normal procedure is followed for preparing reports and the data is second checked before being given to the supervisor.

IX. SOIL/SEDIMENT SAMPLE PROCEDURE

A sample is introduced into the Solid Module via a conditioned sample cup. Once the sample has been introduced the entire analysis sequence is automatic. Please reference Chapter 4 of the OI 1010 Solid Module instrument manual for instrument states and configuration when initially setting the instrument methods up.

TC Mode Instrument Settings:

Analysis Temp:900°C
Analysis Time: 6.5 minutes
Nitrogen Gas Flow: 60-100 psi (external regulator regulator)
Oxygen Gas Flow: 40-60 psi (external regulator)

This is a step by step description of a routine soil TOC analysis.

9.1 The standards and check solutions should be taken out of the refrigerator and allowed to warm to room temperature. The nitrogen and oxygen (internal regulator should be set at 50-60 psi) turned on allowing a nitrogen flow of 350-400mL/minute and an oxygen flow of 180 mL/minute (± 3 mL/minute).

NOTE: DO NOT TURN THE ANALYZER ON BEFORE TURNING THE GAS ON!

9.2 Let the gas flow through the instrument for a few minutes. The instrument should now be turned on and let to stabilize for 30 minutes.

9.3 Condition the cups (with quartz wool in them) using Diagnostics under Instrument Menu commands, (don't condition too many cups at a time since setting in contact with the air can cause contamination).

9.4 Set up the subdirectory (using the current date to ID it) under WinTOC output.

9.5 If doing an initial calibration curve use an appropriate µL syringe to make the following measurements of the sucrose standard in order to achieve the indicated concentrations. Make sure that there are no air bubbles in the syringe. Turn the syringe with the needle pointed up and vibrate the barrel and disperse any air from the syringe. To enter the calibration information on the instrument go to Instrument Cal Menu, type in the calibration standard values and save the file as the cal.. date analyzed.

µL 30% Sucrose STD	Concentration (mg)
0	0
2.0 (1:6 solution)	0.10
3.0	0.90
50	15
100	30

Note: The 1:6 solution of the 30% Sucrose standard is prepared by mixing 100µL of the 30% Sucrose standard with 500µL of water.

9.6 Enter the sequence to be analyzed as listed below:

1. CCV(CCV1+ date analyzed for ID) or Initial calibration – single analyses
2. Method Blank(MB + date analyzed for ID) – single analyses
3. LCS, 15 mg dextrose (LCS + date analyzed for ID) – single analyses
4. NY Cert – 4 replicates
5. Sample – 4 replicates
6. Sample – 4 replicates
7. Sample – 4 replicates
8. Sample – 4 replicates
9. Sample – 4 replicates
10. CCV(CCV1+ date analyzed for ID)2 – single analyses
11. Sample – 4 replicates
12. Sample – 4 replicates
13. Sample – 4 replicates
14. Sample – 4 replicates
15. Sample – 4 replicates
16. CCV (CCV2+ date analyzed for ID) – single analyses
17. Sample – 4 replicates
18. Sample – 4 replicates
19. Sample – 4 replicates
20. Sample – 4 replicates
21. Sample – 4 replicates
22. CCV(CCV3+ date analyzed for ID) – single analyses
23. Sample – 4 replicates
24. Sample – 4 replicates
25. Sample – 4 replicates
26. Sample – 4 replicates
27. Sample – 4 replicates
28. CCV(CCV4+ date analyzed for ID) – single analyses
29. SampleMS – 4 replicates
30. SampleDUP – 4 replicates
31. FCV(CCV4+ date analyzed for ID) – single analyses
32. FCB(FCB4+ date analyzed for ID) – single analyses

9.7 Samples should be stored away from light and at 4°C (± 2°C). Wearing labcoat, gloves and safety glasses remove samples from sample storage signing them out appropriately on the internal chain of custody form.

9.8 Transfer a homogeneous aliquot(~5 g) of the sample into a small pre-labeled aluminum weighing pan. Label each pan with the appropriate sample ID then add enough phosphoric acid (1-2 ml) to remove the Total inorganic carbon (TIC) when the sample is placed in an oven at 250°C. Place the samples in the 250°C oven for 10 minute and begin prepping the sample cups to weigh 0.2g-1.0g of each sample(in quadruplicate). Limit the time that the cups are

exposed to the atmosphere as to reduce potential contamination. **Note: Since the samples are dried in this manner, before the sample aliquot it taken, a % solids determination and calculation is NOT necessary to report the sample concentrations in dry weight.**

9.9 Set the OI 1010 to the TC Mode and start running the sequence beginning with the initial calibration or calibration verification standard as illustrated above. Weigh each sample in quadruplicate making sure to limit the time that samples are exposed to the atmosphere.

9.10 The Excel file for calculations is located in "V:\WCM\TESTS\TOC soil\". The sample identity, its corresponding mgC reading, and the sample weight are entered into the appropriate columns. The Excel worksheet is self explanatory. Normal procedure is followed for preparing reports and the data is second checked before being given to the supervisor.

X. QC REQUIREMENTS

10.1 Analyze a laboratory control sample (LCS) for each batch of samples **(maximum of 10 samples per day).** If the LCS does not fall within the control limits of 80 to 120%, corrective action must be taken to find and correct the problem.

10.2 Run a method blank (PB) for each batch of samples (maximum of 20 samples per day). The PB should be less than 1/2 the reporting limit.

10.3 One matrix spike and matrix spike duplicate must be run per set of 20 samples. For water analysis, a spike and spike duplicate are made by mixing 20 mLs of sample with 0.30 mLs of stock 1000 mg/L standard using an ependorf pipette. The true value is 15 mg/L. The percent recoveries on a MS and a MSD should be within 75 and 125%. Relative percent difference (RPD) on duplicates should be less than 20%. If not, a corrective action (CAR) must be approved by your supervisor.

10.4 Analyze an initial calibration verification (ICV) immediately after the calibration curve. Analyze a calibration check verification (CCV) standard every tenth sample and at the end or after every fifth sample when analyzing samples in quadruplicate. Analyze a CCV after every 5th sample when analyzing soil/sediment samples. The percent recoveries should be in the range of 90 to 110%. The CCV %RSD warning limits are $\leq$15% for aqueous samples and $\leq$20% for soil/sediment samples. If the CCV % RSD exceeds 15%(aqueous) or $\leq$30% (soil/sediment) and the correlation coefficient is less than 0.990 correct the problem and re-analyze the CCV.

10.5 When analyzing water samples, all water blanks before samples and standards must be below the detection limit, otherwise the samples must be rerun.

10.6 Analyze an initial calibration blank (ICB) following the ICV. Analyze a continuing calibration blank (CCB) following each CCV. The ICB and CCB should be less than ± the MDL.

10.7 Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data.

10.8 Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data.

Calculate spikes as follows where everything is in concentration.

$$\% \text{ Recovery} = \frac{\text{Spike} - \text{Sample}}{\text{True Value}} \times 100$$

. Relative percent difference is calculated as follows, with everything in concentration:

$$\text{RPD} = \frac{\text{Higher Concentration} - \text{Lower Concentration}}{\text{Average of Concentrations}} \times 100$$

10.9 SM5310B requires that the analyst repeat injection until consecutive measurements are obtained that are reproducible to within ±10%. A minimum of two injections is required for water samples with three replicates preferred. SW-846 Method 9060 requires quadruplicate analysis of each sample. The Loyd Kahn soil method suggests 1 sample per 20 be run in quadruplicate. Some clients may request that all samples to be done in quadruplicate. Please check with your supervisor if you have any questions about the required numbered of sample replications.

10.10 **For aqueous samples check an acidified 20mg/L inorganic carbon standard quarterly, to assure that purge gas flow is adequate to remove inorganic carbon. The result should be below the reported quantitation limit.**

XI. CORRECTIVE ACTIONS

11.1 INSTRUMENT RELATED

1. ICV not within ± 20% (Soil) or ± 10% (SM 5310C0
a. If the problem is with the solution.

i. Re-prepare, obtain new stock if necessary.
b. If the problem is with the calibration.
i. Recalibrate through analysis of appropriate standards and recheck ICV.
2. CCV not within ± 30% (Soil) or ±15% (SM 5310C)
a. If the problem is with the solution.
i. Re-prepare, obtain new stock if necessary.
b. If the problem is with the calibration.
i. Recalibrate through analysis of appropriate standards and re-prepare /reanalyze the previous ten sample according the following guidelines.
a. If the CCV was biased high, any of the previous ten samples which were below the minimum detection limit do not require reanalysis.
b. If the CCV was biased low, the previous ten samples must be reanalyzed.

*** Incorrectly set gas flow is a common instrument related problem which requires corrective action. Verify that all gas flows are adjusted properly.**

11.2 SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within ±20% aqueous or ±50% soil/sediment
i. The associated sample data must be qualified on the final report.
2. Spike analysis recovery not within ±25% aqueous or ±50% soil/sediment
i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.
ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. A corrective action report must accompany the data and be emailed or given to the supervisor.

XII. HEALTH AND SAFETY

A. 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.

B. 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.

C. MSDS are available for all reagents and standards, which have been purchased. These are located in the administrative section next to the break room.

D. Please see *Waste Disposal; SOP-405* for proper disposal of the waste generated from this area.

XIII. WASTE DISPOSAL and POLLUTION PREVENTION

Please see Waste Disposal SOP-405, for instruction of proper 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.

XIV. METHOD PERFORMANCE

14.1 Precision and Bias for Total Organic Carbon (TOC) by Persulfate-Ultraviolet Oxidation. (Water samples)

Characteristic Of Analysis Concentration determined, mg/L:	Spring Water	Spring Water +0.15 mg/L KHP*	Tap Water	Tap Water +10 mg/L KHP*	Municipal Wastewater Effluent
Replicate 1	0.402	0.559	2.47	11.70	5.88
Replicate 2	0.336	0.491	2.49	11.53	5.31
Replicate 3	0.340	0.505	2.47	11.70	5.21
Replicate 4	0.341	0.523	2.47	11.64	5.17
Replicate 5	0.355	0.542	2.46	11.55	5.10
Replicate 6	0.366	0.546	2.46	11.68	5.33
Replicate 7	0.361	0.548	2.42	11.55	5.35
Mean, mg/L	0.35	0.53	2.46	11.53	5.32
Std. Deviation: mg/L	0.02	0.03	0.02	0.21	0.23
%	6	6	1	2	4
Actual Value, mg/L	-	0.50	-	12.46	-
Recovery, %	-	106	-	93	-
Error, %	-	6	-	7	-

***KHP = potassium acid phthalate.**

14.2 There was no method performance data available for the soil procedure.

XV. REFERENCES

1. Annual Book of ASTM Standards, Part 31, "Water," Standard D 2574-79, p. 469 (1976).

2. Standard Methods for the Examination of Water and Wastewater, 19th ED., Method 5310C (1999).

3. EPA SW-846, Method 9060.

4. Lloyd Kahn Method, *"Determination of Total Organic Carbon in Sediment"*

APPENDIX I.

1. Power up the lamp for warm –up, check reagents inside instrument cavity to make sure all are filled before starting the run.
2. Fill Fresh DI water in 1 gallon jug; DI squirt bottle and 1 L plastic
3. Label and load VOA vials with standards and samples into round tray.
4. Place round tray onto autosampler, get a final sample count for end point and replace lid.
5. Make sure that round tray fits down flush onto the autosampler.
6. On computer screen, select "TOC-Control V" icon.
7. Then select "Sample Table Editor"
8. Enter user name: "analyst initials" select OK.
9. Under "File" select "calibration curve" "OK".
10. Under system select Shimadzu TOC-BWS Enter/next
11. Select Edit Calibration points manually Enter/next
12. Under "Analysis" select "NPOC" then make up your file name (use today's date) Enter/next.
13. Calibration Measurement Parameters are default: Just hit "next"
14. Select "ADD" and enter calibration points starting at (1) 100 mg/L (2) 50 mg/L (3) 25 mg/L (4) 10 mg/L (5) 5.0 mg/L (6) 2.5 mg/L (7) 1.0 mg/L (8) 0.0 mg/L. After 8 points it should show 0.00 mg/L first and 100 mg/L eigth if so "next"
15. Put a check mark in "Correlation Coefficient" check box "next"
16. "next"
17. "finish"
18. Go to file and select "new", "sample run" "ok" "ok" enter file name: user date "save"
19. Now go to insert and select "calibration curve" then scroll till you find your file name/date should have .cal after date "select" the "open"
20. You should now see the sparging /acid addition page which shows a picture of the round sample tray. Under vial manually enter "1"beside 0.00 mg/L.
21. manually enter "2" beside 1.0 mg/L and "3" beside 2.5 mg/L and so on and so forth all the way to "8" this shows what order they ar loaded on the tray. "Enter/OK"
22. Then a screen with your filename/date and all info should be in row 1 only with vial column showing. 1,2,3,4, etc.
23. Select the lightening bolt symbol then enter "use PC settings" this will start initializing wait till screen goes away then you will see the stop light symbol appear with green light showing, select that icon select "keep running" select "standby"
24. Sparging/acid addition page will re-appear just hit "OK"
25. Start ASI tray screen will appear hit "Start"
26. The instrument should start establishing the baseline and move auto tray into position – Lid must be on and samples loaded into corret position will take almost

3 hours to finish. Can view data as its coming off by selecting "view" "sample window". After calibration is done review.

27. Select "File" then "New" then "sample run" "ok"
28. General information screen: No change select "ok"
29. Save as screen: Select today's date for file name example 00month/00day/00year
30. Select "save"
31. Sample Table Screen: Select "insert" then select " auto generate" enter
32. **Page 1** sample group wizard sample source: select "calibration curve" then double click on box with 3 dots …
33. Open latest curve from calibration curves file
34. Highlight latest curve and select "open"
35. Should send you back to page 1 with calibration curve info submitted. Select "next"
36. **Page 2** Sample Parameter: Enter final sample count for "number of samples" select "next"
37. **Page 3** Calibration Curves: No changes Select "Next"
38. **Page 4** Calibration Checks: No changes Select "Next"
39. **Page 5** Controls: No changes select "finish", Select "ok" on "Sparging/ Acid page.
40. Type sequence as they are loaded on tray: ICV, ICB, LCSW, Sample #, client,etc.
41. Once everything is typed in double check that it matches the way samples and QC are loaded..
42. Click or select the lightening bolt symbol then select "use settings on PC". Wait for initializing. When screen goes awy the traffic light symbol should appear next to the lightning bolt symbol. Click on the traffic light symbol.
43. Click or select "shut down Instructions". Then select "standby" Sparging/ Acid addition screen will appear so you can confirm your tray is loaded the wax things are highlighted in blue. Select "OK" if it looks the same.
44. Start ASI measurement: External acid addition should have a check mark click on "start" analysis should begin to start.
45. Click on view and chose "sample window" to watch curves come off and to see beginning values.

GC/MS LOW LEVEL PAH'S

BY SW846 METHOD 8270C SIM

SOP NUMBER: **SOP-231**

REVISION NUMBER: **3**

APPROVED BY:

SECTION MANAGER

QUALITY ASSURANCE OFFICER

EFFECTIVE DATE: **01/16/09**

DATE OF LAST REVIEW: **01/16/09**

GC/MS LOW LEVEL PAH'S BY SW846 METHOD 8270C

1.0 SCOPE AND APPLICATION

This SOP (based primarily on SW-846 Method 8000B/8270C using SIM) is used for the analysis of semi-volatile organic compounds in a variety of matrices (soils, sediments, waters, etc.). The normal laboratory list of PAH analytes with their water and soil LCS limits are found attached in the appendix. Any questions left by this SOP should be answered by reading the method SW-846 8000B/8270C. If questions still remain unanswered, check with the Organic Lab Manager, QA/QC Officer and/or Technical Director. A documentation of capability (DOC) should be performed by each analyst using this method.

2.0 METHOD SUMMARY

After sample preparation using the appropriate extraction technique, the sample is introduced into the GC/MS using direct injection. The analytes are separated in the gas chromatograph by a combination of the temperature program, the pressure program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra from the sample. Analytes are quantitated relative to known standards using the internal standard method.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Section 3.0 and tables 3-1 and 3-2 of the Empirical Laboratories, LLC Quality Assurance Manual include details concerning sample preservation, containers and handling of semi-volatile samples and extracts. All water and soil samples are stored in their respective walk-in coolers at a temperature of 4°C. All extracts are stored in the Hobart in the Extraction lab at a temperature of 4°C. Water samples have a holding time of 7 days from date of sampling. Soil samples have a holding time of 14 days from date of sampling with 40 days from extraction to analysis for waters and soils. (unless otherwise specified for the project).

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

4.1 All raw data (samples & QC) must be evaluated for interferences. If contamination occurs, determine whether the source of interference is in the preparation or clean-up of the samples and take corrective action to eliminate the problem.

4.2 Contamination by carryover can occur when samples of high-concentration and low-concentration are analyzed sequentially. To reduce carryover, the sample syringe must be rinsed with solvent between injections. If an unusually high sample is detected, a solvent blank should be analyzed for cross contamination.

5.0. EQUIPMENT AND APPARATUS

5.1 HP 7890 GC complete with electronic pressure control and temperature programmable gas chromatograph suitable for splitless injection.

5.2 Column: RTX-5MS (or equivalent) 30 m x 0.25 mm I.D. x 0.25 µm film thickness fused silica capillary column or RTX-5 SIL-MS 30 m x 0.28 mm I.D. x 0.5µm film thickness.

5.3 HP 5975 mass spectrometer capable of scanning from 35 to 500 amu every second or less and capable of using Selected Ion Monitoring (SIM), using 70 volts electron energy in electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for decafluorotriphenylphosphine, DFTPP, which meets all the tuning criteria of the EPA methods.

5.4 HP 7683B autosampler capable of reproducibility from one injection to another proven by meeting QC and calibration criteria.

5.5 HP GC/MS interface that gives acceptable calibration points at 10 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.

5.6 Acquisition Software: HP Chemstation system is interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.

5.7 Data Processing Software: Target DB on Windows NT server data system is interfaced to the HP Chemstation. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances in any EICP between specified time or scan-number limits. The most recent NBS mass spectral library is installed.

6.0 REAGENTS

6.1 Methylene chloride **(Please read SOP-336 before handling this solvent in our laboratory.)** – Trace analysis grade.

6.2 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label and recorded in the BNA standards log book. The date they are opened is noted on the label and recorded in the BNA standards log book along with their lot number and vendor. Each standard that is prepared is recorded in the BNA standards log book and given a sequential number. Each standards label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. All stocks and standards are stored in the freezer at a temperature of -15°C ± 5°C from the date they are received/prepared. Standards are brought to room temperature before being used to make standards. Sonication is used if precipitation is observed after bringing to room temperature. The refrigerator and freezer temperature is monitored daily with an annually calibrated thermometer and recorded with calibration correction in the Extraction temperature/calibration logbook. Makeup of common standards is detailed below. See the BNA standards log book for makeup of other standards.

6.2.1 The Decafluorotriphenylphosphine (DFTPP) tuning standard is prepared as follows (includes benzidine, pentachlorophenol and 4,4'-DDT): Using a 10µL syringe, 10µL (GCM-150, Ultra Scientific @ 1000µg/mL, or equivalent) is injected into a 1.0mL volumetric flask containing approximately 0.900mL methylene chloride (Trace Grade) and diluted to volume with same making a

10µg/mL standard. It is stored in the freezer at -15°C ± 5°C for up to 6 months. A direct injection of 1.0µL is used to tune the instrument.

6.2.2 Calibration standards are prepared from a 50µg/mL intermediate standard at a minimum of five concentrations. Calibration standards are prepared semi-annually unless the initial calibration verification standard indicates a problem. To makeup the 50µg/mL intermediate standard inject the indicated amount of the following standards (or equivalent) into a 10mL volumetric containing approximately 1mL methylene chloride (Trace Grade) and dilute to volume with the same. After capping and inverting several times, the solution is transferred into a labeled 10ml, teflon-lined, screw-capped vial and stored in the freezer at -15°C ± 5°C for up to 12 months. See the Appendix for analytes contained in these mixes.

Standard	Conc (µg/mL)	Amount(µL)	Final Conc(µL)
PAH Mix (Vendor,Lot#)	2000	250	50
BN Surrogate (Vendor,Lot#)	5000	100	50

To makeup the calibration standards, inject the indicated amount of a 5.0µg/mL or 0.5 ug/ml intermediate standard, made from a 10x cut of the 50 and 5.0 ug/ml, or the 50 ug/ml into a 2mL vial containing methylene chloride (Trace Grade) and diluted to 1000ul with the same. After capping and inverting several times and adding 5ul internal standard, the standards are transferred into labeled 2ml, teflon-lined, screw-capped vials and stored in the freezer at -15°C ± 5°C for up to 6 months.

Standard	Amount of standard	Amount of 100µg/mL Internal Standard
0.005 µg/mL	10 µL of 0.5 µg/mL	10 µL
0.01 µg/mL	20 µL of 0.5 µg/mL	10 µL
0.05 µg/mL	10 µL of 5 µg/mL	10 µL
0.1 µg/mL	20 µL of 5 µg/mL	10 µL
0.25 µg/mL	50 µL of 5 µg/mL	10 µL
0.5 µg/mL	100 µL of 5 µg/mL	10 µL
1.0 µg/mL	20 µL of 50 µg/mL	10 µL
5.0 µg/mL	100 µL of 50 µg/mL	10 µL
10.0 µg/mL	200 µL of 50 µg/mL	10 µL
20.0 µg/mL	400 µL of 50 µg/mL	10 µL
30.0 µg/mL	600 µL of 50 µg/mL	10 µL
50.0 µg/mL	25 µL of 2000 µg/mL	10 µL

The makeup of the 5.0µg/mL CCV standard is detailed below. Note: MS list spikes and full list spikes for LCS and/or MS/MSD are prepared from an alternate source or lot number than the calibration standards.

5.0µg/mL CCV standard: Using a 100µL syringe, 100µL of a 50µg/mL working standard is injected into a 2 ml vial containing 900µL methylene chloride (Trace Grade) to make a 5.0µg/mL standard. After capping and inverting several times, 10µL of internal standard (100µg/mL) is then added and the standard is stored in the freezer at -15°C ± 5°C for up to 1 week.

6.2.3 The Initial Calibration Verification (ICV) standard is prepared from a vendor stock standard at a concentration of 50µg/mL as detailed below.

5.0 µg/mL ICV standard: Using a 50µL syringe, 50µL (C-507, NSI Solutions @ 100µg/mL, or equivalent) is injected into a 2mL vial containing 950µL methylene chloride (Trace Grade) making a 5.0µg/mL standard. After capping and inverting several times, 10µL of internal standard (100µg/mL) is added and the standard is stored in the freezer at -15°C ± 5°C for up to 6 months.

***For some clients an SPCC curve and CCV are analyzed along with the PAH standards to check for SPCC min. responses. See standard log for preparation of these standards.**

7.0 PROCEDURE

Prior to using SW-846 8270C (semivolatile method) the samples are prepared for chromatography using the appropriate sample preparation and clean up methods (generally SW-846 methods 3510, 3520, 3541, or 3550).

7.1 Chromatographic conditions:

7.1.1 GC

Capillary Column	RTX-5MS 0.25 mm ID
Inlet mode	Pulsed Splitless
Pressure	15.17 psi (on)
Pulse Pressure	20 psi
Pulse time	0.2 minutes
Purge flow	60 mL/min.
Purge time	0.15 minutes
Injector temperature	300°C
Transfer line temperature	280°C
Initial column temperature	50°C for 0.5 minutes
Initial column temperature ramp	20°C/min
Intermed. column temperature	290°C for 0 minutes
Intermed. Temperature ramp	5°C/min
Final column temperature	320°C for 4.9 minutes

7.1.2 MS

Electron energy	70 volts (nominal)
Mass range	35 to 500 amu or SIM
A/D Samples	3
Solvent Delay	4 minutes or as needed

7.2 Tuning - Prior to any calibration or analysis, DFTPP tuning criteria must be met for a 1.0µL injection of the tuning standard [see below]. Tune must be met every 12 hours sample analysis is to be performed.

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

7.3 Calibration

7.3.1 Initial Calibration - An initial calibration curve at no less than five concentration levels must be analyzed and shown to meet the initial calibration criteria before any sample analysis may be performed. Method 8270C requires that the %RSD be less than 15% to use the average response factor for quantitation, the curve is to be used otherwise as long as the correlation factor is ≥0.99. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. Generally, levels for the curve are 0.005 µg/mL, 0.01 µg/mL, 0.05 µg/mL, 0.1 µg/mL, 0.25 µg/mL, 0.5 µg/mL, 1.0 µg/mL, 5.0 µg/mL, 10µg/mL, 20µg/mL, 30µg/mL, and 50µg/mL. 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. All integrations are checked for acceptability. Response factors of less than 0.050 must be supported by the mass spectrum of the lowest standard.

7.3.2 Initial Calibration Verification (ICV) - A second source standard at the 5.0 µg/mL level is used to check the validity of the curve. The standard recovery for all analytes must be between 75 and 125%. If the second source recovery is above 125%, it is possible that the main standard has deteriorated for that compound. That standard should be remade and reevaluated. If that does not correct the problem, the standard should probably be replaced and a new curve generated. If the second source recovery is below 75%, the second source standard may have deteriorated for that compound. The standard should be remade and reanalyzed. If this does not correct the problem, the standard should probably be replaced. If any compound in the ICV exceeds the criteria above, it may be evaluated and initialed by the organic section

manager. If deemed acceptable, the analyst may continue analysis. Any manual integrations are documented by inclusion of the integrated signals with the quantitation report and chromatogram. All integrations are checked for acceptability.

7.3.2 Continuing Calibration Verification (CCV)- Every 12 hours a CCV at 5.0 µg/mL must be analyzed and calculated against the initial calibration curve, then shown to meet the calibration criteria before any sample analysis may be performed. Acceptance criteria for 8270C analytes must be less than or equal to 20% difference or drift (%D, calculations follow in section 7.9). Internal standard areas should be within 50 to 200 percent of the area of the curve midpoint or the previous CCV. Retention times for the internal standards should be within 30 seconds of the retention time of the curve midpoint or the previous CCV. Any manual integrations are documented by inclusion of the integrated signals with the quantitation report and chromatogram. All integrations are checked for acceptability. Samples are then quantitated against the initial calibration curve.

7.4 LCS - The LCS is extracted 1 per extraction batch of up to 20 samples. The LCS is spiked using an alternate source or lot number than the calibration standards. See the LCS report forms in the appendix for the laboratory generated limits. Recoveries for the MS/MSD spike analytes in LCSs are charted annually to generate control limits for samples analyzed by method 8270C. In all cases, the lowest upper limit would be 110% and the lowest lower limit would be 10%. If enough data points are not present to generate limits, the limits default to 10-110% for all other analytes. See Section 8.3 below for corrective action.

7.5 Method Blank - Method blanks are extracted at a minimum of 1 per extraction batch - up to 20 samples. See Section 8.4 below for criteria and corrective action.

7.6 Samples - Prior to analysis, 0.5 mL samples are prepared by verifying volume and spiking with 5uL of the internal standard solution.

7.7 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the chemstation. This run log is signed, dated and paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through Target DB on the Windows NT data system. The following must be checked to determine if the sample will need any reanalysis or dilution. Formal data evaluation is detailed in SOP-200 and documented using the Analyst Data Review Checklist (see Appendix). **See SOP-224 for guidance on manual integrations.**

7.7.1 Internal Standards - Areas should be within 50 to 200 percent of the area of the curve midpoint. Retention time should be within 30 seconds of the retention time of the curve midpoint. If not, the sample and historical data should be evaluated to determine the cause of the problem. If matrix effect is confirmed by reextraction/reanalysis or historical data, complete a corrective action

report and flag the affected compounds on the final report for matrix effect. Note: criteria applies to the continuing calibration, not samples, but is used as an indication of the sample analysis validity.

7.7.2 Surrogates – Control limits are determined annually by charting LCSs and method blanks. In all cases, the lowest upper limit would be 110% and the lowest lower limit would be 10%. All of the three surrogates for each fraction must be within the control limits in order for the extraction batch to be in control. If a surrogate exceeds the limits, the reason for the malfunction must be determined and a corrective action report must be completed. The sample must be reanalyzed, reextracted or flagged for QC problems.

Surrogate	**Water**	**Soil/Sediment**
2-Fluorobiphenyl	34-167	14-129
Terphenyl-d14	34-167	14-129

7.7.3 Analyte concentration must be within the range of the calibration curve after rounding to 2 significant figures. If an analyte exceeds the curve, a dilution must be performed and the next sample must be checked for carryover. Any dilution should keep the concentration of the analyte in question within the top half of the curve.

7.7.4 The qualitative identification of compounds is based on retention time and a comparison of the sample mass spectrum, after background subtraction, with characteristic ions in a reference mass spectrum from the NBS database (NBS75K.I). This database is used as it contains relatively uncontaminated mass spectra of each target compound which cannot be obtained from the daily calibrations during each 12 hour analytical period due to overlapping peaks in the mixes. Characteristic ions from the reference mass spectrum library are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. In addition, the following criteria must be met. The RRT of the sample analyte must be within 0.06 RRT units of the RRT of the standard analyte. The relative intensities of the characteristic ions must agree within 30% of the relative intensities of the same ions in the reference spectrum. Structural isomers that produce very similar mass spectra should be identified as individual isomers so long as their GC retention times differ substantially. A library search may be made for analytes not associated with the calibration for the purpose of tentative identification. NOTE: The GC/MS analyst uses intelligence guided by experience to make the identifications. In uncontaminated spectra where ions are missing due to low concentration, if the major ions are present in the correct ratios at the correct retention time, the identification will be considered positive. In contaminated spectra, special emphasis will be placed upon higher mass ions, and the major ions will usually need to be present as major components of the spectrum (either unsubtracted or subtracted) for the identification to be positive. All assessments of relative intensities of ions will be made by visual estimates from the spectra.

7.8 Quantitation - Once a compound has been identified qualitatively, the concentration must then be quantitated. If the RSD of the compound's response

factor is 15% or less, then the concentration may be determined using the average response factor ($\overline{RF}$) from the initial calibration data. Otherwise, the concentration must be determined from equations based on internal standard calibration using either linear or non-linear calibration. Calculations follow in Section 7.9.

7.9 Calculations:

7.9.1 The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.

A_{is} = Peak area (or height) of the internal standard.

C_s = Concentration of the analyte or surrogate.

C_{is} = Concentration of the internal standard.

7.9.2 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.

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

where CCV RF is the response factor from the analysis of the verification standard and Average RF is the average response factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within ±20% for each analyte before any sample analyses may take place.

7.9.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to µg/L.]

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(V_s)(1000)}$$

where:

A_s = Area (or height) of the peak for the analyte in the sample.

A_{is} = Area (or height) of the peak for the internal standard.

C_{is} = Concentration of the internal standard in the volume extracted in ug/L.

D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.

V_i = Volume of the extract injected (µL). The nominal injection volume for samples and calibration standards must be the same.

$\overline{RF}$ = Mean response factor from the initial calibration.

V_s = Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

7.9.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to µg/kg.]

7.9.5

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(W_s)(1000)}$$

where: A_s, A_{is}, C_{is}, D, and $\overline{RF}$ are the same as for aqueous samples, and

W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

8.0 QUALITY ASSURANCE/QUALITY CONTROL/CORRECTIVE ACTIONS

8.1 Internals - All samples and QC are spiked with internal standards. See section 7.7.1 above for criteria and corrective action.

8.2 Surrogates - All samples and QC are spiked with surrogates. The surrogate recoveries from method blanks and LCS are used to generate control control limits for the surrogates. See section 7.7.2 above for criteria and corrective action. If any surrogate recoveries are below 10%, samples must be re-extracted if sample is available.

8.3 LCS Sample - The LCS is extracted 1 per extraction batch of up to 20 samples. The LCS is spiked using an alternate source or lot number than the calibration standards. If the LCS compound has a recovery above the upper limit, but the same compound is not detected in any of the batch samples, no corrective action is required. For all

other situations, the LCS should be reanalyzed for the failed analytes only. If the second analysis fails, all associated samples should be reextracted/reanalyzed for the failed analytes only or the data must be evaluated for flagging due to QC problems.

8.4 Method Blanks - The concentration of all method target analytes should be below the MDL for each method target analyte(**< ½ RL for DOD QSM**). The first step of corrective action is to assess the effect on the samples. If an analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps should be taken to find/reduce/eliminate the source of this contamination in the method blank. If an analyte is found in the method blank and some, or all, of the other batch samples, then corrective action is required. The source of contamination must be investigated and appropriate action taken and documented to find/reduce/eliminate the source of this contamination. The method blank, and any samples containing the same contaminant, may need to be reextracted/reanalyzed. For the common laboratory contaminants, meeting the above requirements is not practical. Random cases of contamination are difficult to control, however, daily contamination is not acceptable and corrective action is essential. If a contaminant is found in the method blank and the samples, the compound concentration must be flagged with a 'B' on the final report unless the concentration is greater than 10x that found in the method blank.

8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD (for full list spikes, the full list spiking solution is used). Control limits for the MS/MSD recoveries are the same as those for the LCS found in the appendix. RPD limits are found on the LCS report forms in the appendix. Samples which do not meet these criteria due to matrix must be evaluated for flagging on the final report due to QC problems. Generally, batch control is not based on MS/MSD results unless general method failure is determined to be the problem. In that case, the samples and associated QC would be reanalyzed for the failed analytes only. MS data evaluation must include the consideration of the following factors.

8.5.1 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.

8.5.2 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.

8.5.3 MS vs. MSD - If a spiked compound has a similar problem in both the MS and MSD and is not traced to a method problem, no further action may be necessary other than the generation of a corrective action report to document the problem.

8.5.4 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.

8.5.5 Demonstration of Capability (DOC) – Each new analyst must complete a demonstration of capability by analyzing four LCSs with acceptable precision and accuracy. This also must be done when a new instrument is installed or a significant change to the method has been made.

9.0 HEALTH AND SAFETY

9.1 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of gloves and lab coats is highly recommended.

9.2 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.

9.3 MSDS sheets are available for all reagents and standards which have been purchased. These are located on the bookshelf outside the office supply storage room.

REFERENCES

1. *Test Methods for Evaluating Solid Waste*, SW-846,Method 8270C, Third Edition.
2. *DOD Quality Systems Manual for Environmental Laboratories*, 6/2002.
3. *AFCEE QAPP, Version 4.0, July 2004.*

10.0 EXAMPLE FORMS

Definitions and examples of the LCS report sheets (625 water limits, in-house water limits and in-house soil limits), the analyst data review checklist and figure 13 from *Federal Register* method 625 for the tailing factor calculation are located in the appendix.

APPENDIX

TABLE OF DEFINITIONS

1. amu- atomic mass unit
2. PAH-Polyaromatic hydrocarbons
3. BNA-base neutral acid
4. °C- degrees Centigrade
5. CCC- Calibration Check Compound
6. CCV- Continuing Calibration Verification
7. CLP- Contract Laboratory Program
8. %D- percent difference
9. DFTPP- Difluorotriphenyphosphine
10. EICP- extracted ion current profile
11. EPA- Environmental Protection Agency
12. g- gram or grams
13. GC- Gas Chromatograph
14. GC/MS- Gas Chromatograph/Mass Spectrometer
15. ICV- Initial Calibration Verification
16. I.D.- inner diameter
17. ISTD- internal standard
18. LSC- Laboratory Sample Concentrator
19. MDL- method detection limit
20. MS- Matrix Spike
21. MSD- Matrix Spike Duplicate
22. M.S.- Mass Spectrometer
23. µm- micometer
24. µL- microliter
25. mL- milliliter
26. mm- millimeter
27. ng- nanogram
28. NPDES- National Pollutant Discharge Elimination System
29. P&T- purge and trap
30. QC- quality control
31. %R- percent recovery
32. RPD- relative percent difference
33. RRT- relative retention time
34. %RSD- percent relative standard deviation
35. SOP- Standard Operating Procedure
36. Surr.- surrogate
37. SPCC- System Performance Check Compound
38. TCLP- Toxicity Characteristic Leaching Procedure
39. USACE- United States Army Corps Of Engineers

Refer to SOP-431 for additional definitions

FORM 3

WATER PAH LAB CONTROL SAMPLE (8270C)

Lab Name: ELAB Contract:

Lab Code: NA Batch No.: NA SAS No.: NA SDG No.:

Matrix Spike - Client Sample No.:

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	LCS CONCENTRATION (ug/L)	LCS % REC #	QC. LIMITS REC.
Acenaphthene	1.00	0.0000	1.00	100	41-132
Acenaphthylene	1.00	0.0000	1.00	100	43-140
Anthracene	1.00	0.0000	1.00	100	50-139
Benzo(a)anthracene	1.00	0.0000	1.00	100	58-141
Benzo(b)fluoranthene	1.00	0.0000	1.00	100	42-156
Benzo(k)fluoranthene	1.00	0.0000	1.00	100	49-165
Benzo(g,h,i)perylene	1.00	0.0000	1.00	100	12-171
Benzo(a)pyrene	1.00	0.0000	1.00	100	31-142
Chrysene	1.00	0.0000	1.00	100	51-155
Dibenz(a,h)anthracene	1.00	0.0000	1.00	100	28-153
Fluoranthene	1.00	0.0000	1.00	100	47-158
Fluorene	1.00	0.0000	1.00	100	40-140
Indeno(1,2,3-cd)pyrene	1.00	0.0000	1.00	100	20-167
Naphthalene	1.00	0.0000	1.00	100	39-125
Phenanthrene	1.00	0.0000	1.00	100	46-144
Pyrene	1.00	0.0000	1.00	100	39-158
2-Methylnaphthalene	1.00	0.0000	1.00	100	36-121
1-Methylnaphthalene	1.00	0.0000	1.00	100	35-131

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

COMMENTS: _____

page 1 of 1

FORM III SV
FORM 3

SOIL PAH LAB CONTROL SAMPLE (8270C)

Lab Name: ELAB Contract:

Lab Code: NA Batch No.: NA SAS No.: NA SDG No.:

Matrix Spike - Client Sample No.:

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	LCS CONCENTRATION (ug/Kg)	LCS % REC #	QC. LIMITS REC.
Acenaphthene	67.00	0.0000	67.00	100	28-110
Acenaphthylene	67.00	0.0000	67.00	100	23-126
Anthracene	67.00	0.0000	67.00	100	28-136
Benzo(a)anthracene	67.00	0.0000	67.00	100	31-146
Benzo(b)fluoranthene	67.00	0.0000	67.00	100	30-139
Benzo(k)fluoranthene	67.00	0.0000	67.00	100	42-129
Benzo(g,h,i)perylene	67.00	0.0000	67.00	100	21-149
Benzo(a)pyrene	67.00	0.0000	67.00	100	28-128
Chrysene	67.00	0.0000	67.00	100	39-134
Dibenz(a,h)anthracene	67.00	0.0000	67.00	100	30-138
Fluoranthene	67.00	0.0000	67.00	100	30-142
Fluorene	67.00	0.0000	67.00	100	27-116
Indeno(1,2,3-cd)pyrene	67.00	0.0000	67.00	100	17-164
Naphthalene	67.00	0.0000	67.00	100	29-106
Phenanthrene	67.00	0.0000	67.00	100	32-127
Pyrene	67.00	0.0000	67.00	100	28-130
2-Methylnaphthalene	67.00	0.0000	67.00	100	30-111
1-Methylnaphthalene	67.00	0.0000	67.00	100	30-111

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

For 8270-SIM projects, the AFCEE QAPP Ver. 4.0 limits for PAHs in soil and water will be used.

PAH Mix

Acenaphthene
Acenapthylene
Anthracene
Benzo(a)anthracene
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(g,h,i)perylene
Benzo(a)pyrene
Chrysene
Dibenz(a,h)anthracene
Fluoranthene
Fluorene
Indeno(1,2,3-cd)pyrene
2-Methylnaphthalene
1-Methylnaphthalene
Naphthalene
Phenanthrene
Pyrene

BN Surrogates

2-Fluorobiphenyl
Terphenyl-d14

Internal Standards

Perylene-d12
Phenanthrene-d10

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):	
Batch Number(s):	
Method:	**8260/8270 (Circle One)**

QA/QC Item	Yes	No	NA	Second Level Review
1. Is the BFB/DFTPP tune performed every 12 hours and is the tuning criteria met?				
2. Are the RRFs and % RSDs within QC limits for appropriate analytes for the initial calibration? Check the retention times for compounds with the same spectra. Check compounds with different conc.(eg. m/p-xylene, ketones,etc.).				
3. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?				
4. Does the Continuing Calibration Standard (CCV) meet the criteria for the CCCs and SPCCs.				
5. Is the Method Blank run at the desired frequency and is its concentration for target analytes less than the MDLs?				
6. Are the LCS, MS, MSD within control limits and run at the desired frequency?				
7. Are all sample holding times met, analytes within calibration range, IS areas and surrogate recoveries within QC limits?				
8. Was the Method Blank, LCS, MS, MSD and samples loaded to the GCMS_LFSYS Tablespace within the Target DB Database?				

Comments on any "No" response:

_____

_____

_____

_____

Primary-Level Review: _____ Date: _____

Second-Level Review: _____ Date: _____

GC/MS SEMI-VOLATILE BNA-AQUEOUS MATRIX EXTRACTION USING SW-846 METHOD 3510C FOR 8270C/625 ANALYSIS

SOP NUMBER: **SOP-300**

REVISION NUMBER: **17**

APPROVED BY:

SECTION MANAGER

QUALITY ASSURANCE OFFICER

EFFECTIVE DATE: **09/23/08**

DATE OF LAST REVIEW: **09/23/08**

GC/MS BNA - AQUEOUS MATRIX EXTRACTION USING SW846 METHOD 3510C/8270C, 625

I. SCOPE AND APPLICATION/SUMMARY

1. This SOP describes the extraction of BNAs from water by separatory funnel extraction using SW846 Method 3510C and 625. Samples are extracted with methylene chloride and concentrated to an appropriate final volume.

II. INTERFERENCES

1. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
2. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
3. Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

III. APPARATUS AND MATERIALS

- Separatory Funnel - 2-Liter with Teflon stopcock
- Beaker - 400 mL
- Drying /Chromatographic column - 20 mm I.D. x 300 mm or funnel
- Turbo-Vap evaporation tube - 200 mL tube made by Zymark to fit into Turbo-Vap evaporator
- Metal rack - capable of holding six glass evaporation tubes
- Turbo-Vap Evaporator - heated and capable of temperature control ($\pm$5°C); the bath should be vented into a hood.
- Vials - 2 mL glass amber, with Teflon-lined screw cap and 40 mL with Teflon lid.
- pH indicator paper - close range (1.0 - 2.0) and (10.0 - 12.0); wide range (1.0 - 12.0)
- Syringe - 1 mL, 500 mL
- Graduated cylinder - Glass, Class A, 1000 mL, 500 mL, and 100 mL
- Pasteur pipette - length 9" and 5-3/4"
- Pasteur pipette bulb
- Labels - Avery
- Teflon Bottles - 250 mL and 1000 mL
- Ring stand - 3 prong
- Aluminum foil - heavy duty
- 10 mL disposable pipette
- Nitrogen tank - equipped with pressure regulator

IV. REAGENTS

- Reagent Water - Reagent water is gathered in a carboy from source in the instrument lab daily. Remaining water in the carboy is dumped at the end of each day.

- Sodium Hydroxide Solution - (10N), Weigh 400 g NaOH (purchased in a plastic container from Fisher # S318-3 or equivalent) into a 1200 mL fleaker beaker and cover with reagent water. Swirl until all pellets are dissolved. This mixture gets very hot. Let stand until cool. Transfer to a 1-liter volumetric flask with several rinses of reagent water and dilute to 1 liter with reagent water. Transfer to a 1000-mL Teflon container.

- Sodium Sulfate - Granular, anhydrous, trace pure 10 - 60 mesh (purchased in plastic bulk containers from Fisher # S415-10S or equivalent) placed in Pyrex tray and heated at 400°C for a minimum of 4 hrs, removed and cooled in open air in the extraction lab, placed in a 2.5 kg glass amber jug and left at room temperature.

- Glass Wool - Silane Treated (purchased from Supelco #2-0410 or equivalent).

- Sulfuric Acid Solution - (1:1), slowly add 500 mL of H2SO4 (Baker, suitable for trace metal analysis #9673-33 or equivalent) to 500 mL of reagent water in a 1000 mL Teflon container. This mixture will get very warm. Allow to cool before use.

- Extraction Solvent - Methylene Chloride (**Please read SOP-336 before handling this solvent in our laboratory.)** (Dichloromethane - Omnisolv - suitable for spectrophotometry and gas chromatography #DX0831-1 or equivalent).

- The GC/MS operator makes up all surrogates and spikes. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes.

  BNA Surrogate - The base neutral and acid surrogates are normally mixed together in one solution. This solution is purchased from a reputable vendor. Use 0.5 mL of this solution per 1000 mLs of aqueous sample for surrogate amount of 100:200 ug/mL per sample. **(For low level PAHs use 1.0ml of a 1.0µg/mL BN Surrogate spiking solution.)**

  BNA Spiking Solution - The base neutral and acid spiking solutions are normally mixed together in one solution (**This spiking solution contains all the compounds that are normally calibrated by GC/MS**). This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor. Use 0.5 mL of this solution per 1000 mLs of aqueous sample for LCS amount of 100ppm per sample. There are two separate spiking solutions available – one solution has a more complete list of BNA compounds than the other which is called the short or matrix spike list. The long list should be used on all extractions unless your supervisor has approved the short list. The short

list may be used for any 'phenol only' extractions. **(For low level PAHs use 1.0 ml of a 1.0ppm of the LLPAH spiking solution.)**

BNA TCLP Spike – 0.5 mL is added per 100-mL volume. Each matrix type must have its own TCLP spike. TCLP spike should be added after the TCLP has been filtered but prior to refrigeration. From the volume provided by Wet Chemistry, remove a 100-mL aliquot into a suitable container with a teflon lid, and spike as indicated above.

V. PROCEDURE

1. All waters have a seven-day holding time counted from the hour they are sampled. Determine the samples necessary to extract from the following sources (Note: never extract samples of unknown origin without discussion with supervisor):
   - 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.
   - 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.
   - 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.

2. Wearing lab coat, gloves and safety glasses, get samples from refrigerator. 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 amber jar and have a Teflon lid. Find out if any special dilutions need to be made for this client. Routine procedures for difficult matrices are listed below:

   SLUDGE - use only 100 mL and dilute to 1000 mL with reagent water.
   TCLP EXTRACT - use only 100 mL and dilute to 1000 mL with reagent water. A separate matrix spike of 100 mLs (which has already been spiked as explained in the BNA TCLP Spike section above) should be set up at the same time. Dilute to 1000 mL with reagent water.
   BAD MATRIX – for example a liquid that is partially sediment, see your supervisor to find out what dilution, if any should be made. **SPLP extract- use 1 liter**.
   NPDES client - a special list of compounds is required including benzidine. Method 625 requires that there be a spike every ten samples. The sample must be extracted and concentrated in the same day. A GC/MS screen is recommended; therefore this extraction should be coordinated with the GC/MS operator. 1 mL is added to the LCS and the matrix spike.
   ACID EXTRACT WITH BAD MATRIX - a cleanup step is added. Samples are taken to a high pH, extracted with 60 mLs methylene chloride one time as explained below in the BASE NEUTRAL EXTRACTION section. This extract is discarded. The samples

are then taken to a low pH and extracted as an acid extraction. Acid extractions may be concentrated in the TurboVap.

3. **LOW LEVEL POLYAROMATIC HYDROCARBONS (PAHs) –** Samples require a BNA extraction. Use the Surrogate and BN spiking solutions specified. Low level PAHs are normally concentrated on the Turbo-Vap using Round-Bottom TV tubes to a final volume of 0.5 mL

4. Mark the amber glass container of each sample at the water meniscus with "white out" for later determination of sample volume. Check the pH by inverting the sample and touching the wide range pH paper to the portion that remains on the lid. Record this pH in the logbook.

5. Get out enough separatory funnels to extract the number of samples you have plus any additional spikes and a method blank. A method blank and an LCS must be processed with each set of samples. If the sample is a TCLP, blank fluid may be provided along with the extracted TCLP sample(s). Use only 100 mL and dilute to 1000 mL with reagent water. A matrix spike and a duplicate or a matrix spike duplicate must be processed for each analytical batch (up to a maximum of 20 samples). In the event that adequate sample is not provided to do an MS/MSD, an LCS duplicate should be done. Rinse separatory funnels with methylene chloride. Place an Avery label on each separatory funnel containing the following information: Lab #, Client name, Type of Analysis, Initial Volume-Final Volume, and the Lab prepbatch code. The lab batch code is defined as MMDDYYB# where #: 1 = 1st method blank of the day; 2 = 2nd method blank of the day; etc. The Method Blank and LCS label should include all lab #s in this set of samples.

6. Using the 1000-mL glass graduated cylinder marked NANO PURE WATER ONLY, measure 1000 mL of reagent water from the carboy and transfer it to a separatory funnel for the method blank and LCS. Transfer sample to separatory funnel that corresponds to the lab # on the sample bottle.

7. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.

   Generally 0.5 mL of BNA surrogate is added to each sample, spike, and blank with a syringe designated for BNA surrogate. **For low level PAHs use 1.0ml of a 1.0ppm LLPAH Surrogate spiking solution.** Someone must verify that the surrogate has been added by placing a check mark on each label as it is added.

   NOTE: Be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution.

For the sample in each analytical batch selected for spiking, use the 0.5-mL glass syringe designated for BNA spike, to add 0.5 mL of BNA spiking solution. **For low level PAHs use 1.0 mL of the 1.0ppm LLPAHs spiking solution.** Someone must verify that the spike has been added by placing a check mark on each label as it is added. **For DOD QSM projects, all target compounds will be spiked into the LCS and MS/MSD.**

Enter the ID# of the surrogate/spike used and the initials of the person that verified their addition to the sample in the BNA logbook.

8. **ACID EXTRACTION:** Adjust the pH to between 1.0 and 2.0, using 2 mL of 1:1 H2SO4. Add to each sample, spike and method blank. Stopper and shake to insure that pH throughout the sample is changed. Check the drop of liquid hanging from the lid with short-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more H2SO4 solution in small increments, as required to attain the proper pH.

9. Add 40 mL of Methylene Chloride to each empty sample bottle and to the LCS, method blank and MS/MSD funnels. Swirl the 40-mL of methylene chloride that you added to the empty sample bottle and transfer to the corresponding separatory funnel.

10. Seal and shake the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. Alternatively, Teflon funnels may be used and placed in the shaker apparatus with the stopcocks slightly open. When this apparatus is used, the shake should be for 3 minutes.

    NOTE: Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once.

11. Allow the sample to sit for 10 minutes, if necessary, after it has been shaken. It will separate into two layers with the solvent layer on the bottom. If it forms an emulsion (thick, cloudy, viscous, mixture that you cannot see through), drain what you believe to be 40 mL into a 250 mL centrifuge bottle. If the layers are clearly separated, drain the solvent layer into a 400-mL glass beaker.

12. Following Steps 9 and 10, extract two more times with 40 mLs of methylene chloride. Combine the three solvent extracts into the same 400-mL beaker.

13. **BASE NEUTRAL EXTRACTION: Adjust the pH to 11 or slightly greater**, using 10N NaOH. Start by adding 5.0 mLs to each sample, spike, and method blank. Stopper and shake to insure that pH throughout the sample is changed. Check the drop of liquid hanging from the lid with short-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more 10N NaOH in small increments, as required to attain the proper pH. **BNA extraction is necessary when doing low level PAHs.**

NOTE: This step is critical to the extraction procedure. Too much NaOH solution could cause you to lose certain Base Neutral compounds. Be careful on this step.

14. **FOR 8270 extraction**: Extract one more time with 40 mL of methylene chloride following Steps 9 and 10. Combine BN and Acid extracts in a same 400ml beaker, unless the BN extract has large amount of emulsion; then it will be necessary to use a separate 400 mL beaker. Concentrate BN and acid extracts for one final extract.
NOTE: It has been demonstrated that two acid and one BN extraction can be used for normal 8270 samples. This procedure cannot be used for DOD or 625 samples.

For 625 extraction: extract 3 more times with 40 mL methylene chloride following steps 9 and 10. Combine BN extracts in separate 400 mL beaker. Concentrate BN and acid extracts separately for one final extract.

15. In the log book marked BNA extractions, enter the Client name, the Lab #, the date extracted, the initial volume, and 1.0 mL for the final volume and anything unusual that may have occurred with this sample. The final volume for low level PAHs is 0.5 mL.

16. Prepare to dry the sample by either of the following methods:
    16A. Get a ring stand with a double burette clamp attached to it. Cover the burette clamp ends with aluminum foil to prevent the possibility of solvent touching the plastic coated ends and dripping into the extract. Place a drying column into the burette clamp and transfer a small amount of glass wool to the top of it. Tamp it to the bottom with a glass rod so that it adequately covers the hole at the bottom. Add approximately 10 cm of Sodium Sulfate to the column. Rinse with 20 to 30 mL of methylene chloride and discard this rinse into the Chlorinated Waste container in the hood. OR

    16B. Set up a ring stand with funnels. Place a small amount of glass wool in the bottom of it, add ~2" sodium sulfate to the column and rinse with 20-30 mL methylene chloride. Discard this rinse into the Chlorinated Waste container in the hood.

17. If the extract was drained into a centrifuge bottle, at this point you will need to take it to the centrifuge. Push the "ON" button to turn the centrifuge on. Be sure that the large holders are available for the 250-mL centrifuge bottles. The sample must always be balanced. If necessary use a dummy bottle making it similar weight using reagent water. Set the rpm at 2500 and the temperature at 0°C. Close the lid and be sure to press it down until you hear it click. Move the lever at the front of the lid to the "LOCK" position. Turn the time to approximately 15 minutes and bring it back to 10 minutes. As the rotor begins to move, you will be able to see the rpm's in the digital readout. Stay with the centrifuge until it has come up to the rpm's set to insure that it does not become unbalanced. This looks like 8888 on the digital readout. Should this occur, refer to the manual. When the cycle is complete, the digital readout will read 0000. Push the "OPEN" button and the lid will pop up. Move the lever at the front of the lid to the

"UNLOCK" position. Open lid and remove sample. The sample will usually be in two layers with the extract on the bottom.

18. Remove any water layer from the extract in the beaker or centrifuge bottle, by one of two methods. Remove with a Pasteur pipette by carefully pulling up the water layer, on top, and not the solvent. Discard this layer in the sink. Use the smallest amount possible of Na2SO4 by sprinkling the top layer with Na2SO4 until it hardens, separates, and drops to the bottom.

19. TURBO-VAP CONCENTRATION

Low level PAH sample concentration is primarily done by Turbo-Vap using Round-Bottom TV tubes.

- Rinse a Turbo-Vap tube with methylene chloride and arrange it underneath a rinsed, packed drying column or funnel. Pour the extract through the column so that it will collect in the tube. Rinse the 400-mL beaker, which contained the solvent extract twice with 10 to 15 mL of methylene chloride and add each rinse to the column to complete the quantitative transfer. After all the extract has passed through the column, rinse the column with 10 to 15 mL of methylene chloride. Total volume in the glass evaporator tube should not exceed 200 mLs to avoid splattering on the lid of the Turbo-Vap.
- Record the numbers of the Turbo-Vap tube in the BNA logbook and remove the tube to a metal holder. To help prevent cross contamination, place a piece of aluminum foil over the Turbo-Vap tube and punch a small hole in the top so that the nitrogen can be accessed.

- Turbo-Vap Operation: Adjust the pressure of nitrogen gas tank to >30 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 40°C -50°C.

- Place the glass evaporator tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be in the sensor and need removal. (See Turbo-Vap manual).

- When the beep sounds indicating the end of concentration, the extract will be at approximately one half mL (half way up tip of tube). Remove the tube from the bath. Use a 9" Pasteur pipette to draw up the sample and transfer it to the 2-mL vial. THIS IS THE MOST CRITICAL PART OF THE ENTIRE OPERATION!!! A single drop represents about 10 percent of the total sample. Before you move the tip of the pipette from the tube to the vial, be sure that a drop will not form on the end and fall off.

- Draw ~0.25 mL of methylene chloride into a 0.50 mL syringe and add this aliquot to the centrifuge tube. Draw the methylene chloride into a pipette

and rinse the sides of the tube several times. Transfer this rinse to the 2-mL vial. Add methylene chloride from the syringe and repeat the rinsing process until you have ~ 1 mL in the sample extract vial. Compare this volume to a 2-mL dummy vial containing 1 mL of solvent to insure that you have not exceeded 1 mL. For low level PAHs the final volume is 0.5mL. The methylene chloride rinse volume must be adjusted to achieve this final volume. Compare the volume to a 2mL dummy vial containing 0.5 mL of solvent to insure that you have not exceeded 0.5 mL. The GC/MS operator will adjust the sample to the desired final volume and add internal standard just prior to analyses. Cover the extract with a Teflon-sealed screw cap and transfer the label to the vial.

20. 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 BNA logbook and the Avery label to the nearest 10-mL.

21. The extract is now ready to be analyzed. Refrigerate at 4°C or carry directly to the instrument operator. Samples must be signed into the Sample Extract refrigerator. On log provided, enter the sample numbers, the analyst initials, and the date and time the samples were placed into the refrigerator.

VI. DOCUMENTATION OF CAPABILITY (DOC)

Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

VII. WASTE MANAGEMENT AND POLLUTION PREVENTION

Please see Waste Disposal SOP-405 for the proper 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.

VIII. METHOD PERFORMANCE

Refer to SOP-201 for method performance.

IX. HEALTH AND SAFETY

Refer to the MSDS sheets for the chemicals used for health and safety information. Also see SOP-336 for proper use of methylene chloride.

REFERENCES

1. *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition
2. 40 CFR, Method 625.

DEFINITIONS

BNA- base/neutral acid
°C - degrees centigrade
COC - chain of custody
DL - detection limit
g - grams
KD - kuderna danish
LCS - laboratory control sample
µg/L - micrograms per liter
µL - microliter
µg/ml - micrograms per milliliter
ml - milliliter
mm - millimeter
MS - matrix spike
MSD - matrix spike duplicate
PAH- polynuclear aromatic hydrocarbon
RL - reporting limit
SOP - standard operating procedure
v/v - volume to volume

Refer to SOP-431 for further definitions

Fraction: Semi-volatiles Matrix: Water EX0__

Supervisor_____

#	Client	Lab No.	Date Extracted	Setup Initials	pH	Initial Volume(ml)	Final Volume(ml)	Surrogate Added	Surr Initials	Spike Added	Spk Initials	KD	10ml Conc.	15ml Cent.	Conc. Initials	Solvent Lot/Vendor Methylene Chloride	Notes/Comments	#
1										NA	NA							1
2										NA	NA							2
3										NA	NA							3
4										NA	NA							4
5										NA	NA							5
6										NA	NA							6
7										NA	NA							7
8										NA	NA							8
9										NA	NA							9
10										NA	NA							10
11										NA	NA							11
12										NA	NA							12
13										NA	NA							13
14										NA	NA							14
15										NA	NA							15
16										NA	NA							16
17										NA	NA							17
18										NA	NA							18
19										NA	NA							19
20										NA	NA							20
								SAS	SAS							SAS		
								SAS	SAS							SAS		
								SAS	SAS							SAS		
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PESTICIDE/PCBs
AQUEOUS MATRIX EXTRACTION
FOR EPA METHOD 608/608.2 AND
SW846 METHOD 8081A/8082
USING SW846 METHOD 3510C

SOP NUMBER: **SOP-302**

REVISION NUMBER: **16**

APPROVED BY: _____
SECTION MANAGER

_____
QUALITY ASSURANCE OFFICER

EFFECTIVE DATE: **09/23/08**

DATE of LAST REVIEW: **09/23/08**

PESTICIDE/PCB - AQUEOUS MATRIX EXTRACTION BY EPA METHOD 608/608.2 AND SW-846 METHOD 8081A/8082 USING SW846 METHOD 3510C

I. SCOPE AND APPLICATION

1. This SOP describes the extraction of pesticides/PCBs from water by separatory funnel extraction using SW846 Method 3510C and Method 608/608.2.

II. SUMMARY

1. Aqueous samples are extracted three times with methylene choride. The extracts are dried through sodium sulfate and concentrated and exchanged to hexane.

III. INTERFERENCES

1. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
2. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
3. Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

IV. APPARATUS AND MATERIALS

- Separatory Funnel - 2 Liter with Teflon stopcock
- Beaker - 400 mL
- Drying/Chromatographic column - 20 mm I.D. x 300 mm (or funnel)
- Turbo-Vap evaporation tube - 200-mL tube made by Zymark to fit into Turbo-Vap evaporator.
- Metal rack - capable of holding six glass evaporation tubes.
- Turbo-Vap Evaporator - heated and capable of temperature control (+5°C); the bath should be vented into a hood.
- Vials 10-mL glass, with Teflon-lined screw cap
- pH indicator paper - wide range (1.0 -12.0)
- Syringe - 1 mL, 500 µL
- Graduated cylinder - Glass, Class A, 1000 mL, 500 mL, and 100 mL
- Pasteur pipette - length 9"
- Pasteur pipette bulb
- Labels - Avery
- Teflon Bottles - 250 mL and 1000 mL
- Volumetric Flasks - Class A, glass, 1000 mL, 500 mL,100 ml, 50 ml.

- Ring stand - 3 prong
- Burette clamp - double
- Aluminum foil - heavy duty
- 10 mL disposable pipette
- Nitrogen tank - equipped with pressure regulator
- Boiling chips - Teflon

V. REAGENTS

- Reagent water - Reagent water is gathered in a carboy from source in the instrument lab daily. Remaining water in the carboy is dumped at the end of each day.
- Sodium Sulfate (Na2SO4) - Granular, anhydrous, trace pure 10 - 60 mesh (purchased in bulk containers from Fisher # S415-10S or equivalent] placed in a Pyrex tray and heated at 400°C for 4 hrs minimum, removed and cooled in open air in the extraction lab, placed in a 2.5 kg glass amber jug and left at room temperature.
- Glass Wool - Silane Treated (purchased from Supelco #2-0410 or equivalent).
- Sulfuric Acid Solution - (1:1), slowly add 500 mL of H2SO4 (Baker, suitable for trace metal analysis #9673-33 or equivalent) to 500 mL of reagent water in a 1000 mL Teflon container. This mixture will get very warm. Allow to cool before use.
- Sodium Hydroxide Solution - (10N), Weigh 400 g NaOH (purchased in a plastic container from Fisher # S318-3 or equivalent) into a 1200 mL fleaker beaker and cover with reagent water. Swirl until all pellets are dissolved. This mixture gets very hot. Let stand until cool. Transfer to a 1-liter volumetric flask with several rinses of reagent water and dilute to 1 liter with reagent water. Transfer to a 1000-mL Teflon container.
- Extraction Solvent - Methylene Chloride **(Please read S0P-336 before handling this solvent in our laboratory.)** (Dichloromethane - Omnisolv - suitable for spectrophotometry and gas chromatography #DX0831-1 or equivalent)
- Hexane - suitable for gas chromatography (Omnisolv HX0298-1 or equivalent)
- The GC operator makes up all surrogates and spikes. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes.

  TCMX/DCB (2, 4, 5, 6 - Tetrachloro-meta-xylene/Decachlorobiphenyl) - Surrogate solution is prepared in acetone by making a cut on stock purchased from a reputable vendor. The final concentration is 0.5 µg/mL. Generally use 1.0 mL of this solution per 1000ml of aqueous sample.

  PCB Spiking Solution - The PCB of choice (1242, 1248, 1254, or 1016/1260 are the most common) is prepared in acetone at a concentration of 5.0 PPM. PCB stock is usually purchased from RESTEK or equivalent. The PCB to use may be determined by viewing historical data or asking the GC operator. Generally use 0.2- 1.0 mL per 1000ml of aqueous sample.

  Pesticide Spiking Solution - A spiking solution is prepared at the appropriate concentration of 1 ppm. Generally use 1 mL per 1000ml of aqueous sample. For 608 samples, 1 out of every 10 samples must be spiked.

- TCLP's - When necessary to set up a TCLP, in addition to setting up the sample, two matrix spikes must be set up and should include the following: 1.0 mL is added per 100 mL volume. Each matrix type must have its own TCLP spike.

  TCLP Spike 1 - includes Chlordane at 10 µg/mL and Toxaphene at 10 µg/mL. These spikes are made up individually and are also used as the LCS spike. Add 1 mL of each to a 100-mL aliquot.

  TCLP Spike 2 - includes Mix A and Mix B @ 100 to 1000 µg/L ppb (also used as the pesticide spiking solution). These spikes are combined into one solution. Add 1 mL per 100-mL aliquot.

VI. PROCEDURE

1. All waters have a seven-day holding time counted from the hour they are sampled. Determine the samples necessary to extract as follows:

   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.

   Samples requiring RUSH turn around time may be logged in throughout the day that will require your immediate attention. Login personnel will generally communicate this need.

   Check with login 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.

2. 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 amber jar and have a Teflon lid. Find out if any special dilutions need to be made for this client. Routine procedures for difficult matrices are listed below:

   SLUDGE - use only 100 mL and dilute to 1000 mL with reagent water.
   TCLP EXTRACT - use only 100 mL for the sample and dilute to 1000 mL with reagent water. Two matrix spikes of 100 mLs (per client matrix). Dilute to 1000 mL with reagent water.
   BAD MATRIX – for example a liquid that is partially sediment, see your supervisor to find out what dilution, if any, should be made.
   NPDES client - Samples for method 608/608.2 are checked by login to make sure the pH of the sample is in the range of 5.0 and 9.0. If the sample is not in this range, extraction personnel will be notified. At that time, it is the responsibility of the extraction lab to adjust the pH of the sample to the appropriate range (pH of 5-9 using NaOH solution or H2SO4, as necessary) or to extract the sample within 72 hours of sampling. If a pH

adjustment is made, the details of the adjustment must be recorded on the sample chain of custody and the outside of the sample bottle. Set up one full list matrix spike for every ten samples.

3. Mark the amber glass container of each sample at the water meniscus with "white out" for later determination of sample volume. Check the pH by inverting the sample and touching the wide range pH paper to the portion that remains on the lid. Record this pH in the logbook

4. Get out enough separatory funnels to extract the number of samples you have plus any additional spikes and a method blank. A method blank and an LCS are to be processed with each set of samples. If the sample is a TCLP, a blank may be provided along with the extracted TCLP sample(s). Use only 100 mL and dilute to 1000 mL with reagent water. A matrix spike and a duplicate or a matrix spike duplicate must be processed for each analytical batch (up to a maximum of 20 samples) except for TCLPs and 608 samples as noted above. In the event that adequate sample is not provided to do an MS/MSD, an LCS duplicate should be done. Rinse separatory funnels with methylene chloride. Place an Avery label on each separatory funnel containing the following information: Lab #, Client name, Type of Analysis, Initial Volume - Final Volume, and the Lab Prep Batch Code. The lab prep batch code is defined as MMDDYYP# where #: 1 = 1st Method Blank for the day; 2 = 2nd Method Blank for the day; etc. The Method Blank and LCS label should include all lab #s in this set of samples.

5. Using the 500 mL glass graduated cylinder marked NANO PURE WATER ONLY, measure 1 liter of reagent water from the carboy and transfer it to a separatory funnel for the Method Blank and LCS. Transfer sample to separatory funnel that corresponds to the lab # on the sample bottle. Add 50 mL of Methylene Chloride to the empty bottle.

6. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.

   Using the 1.0-mL glass syringe marked TCMX/DCB surrogate, add 1.0 mL of TCMX/DCB surrogate to each sample, blank and spike. A second analyst must verify that the surrogate and spike has been added. Enter the ID# of the standard, amount, and the initials of the analysts in the extraction logbook.

7. Determine if the sample will require a Pesticide Spike or a PCB Spike or both. Proceed as follows:

   Pesticide and PCB - set up two LCS's – one for Pesticide getting a MIX A&B spike and one for PCB, which should be spiked with PCB 1016/1260. In addition to the LCS's, a matrix sample spike and duplicate is spiked for pesticides and PCBs.

Pesticide only – To the sample in each analytical batch selected for spiking, add 1.0mL of Pesticide Spike (Mix A&B) with a glass syringe dedicated for Pesticide Spike.

PCB only - To the sample in each analytical batch selected for spiking, add 1.0 mL of PCB 1016/1260 or 1248 (project specific) unless otherwise specified using a 1.0-mL glass syringe dedicated to that PCB.

NOTE: Due to limited volume received, usually it is necessary to use 1/2 liter to do a spike so that a spike duplicate can be extracted also. If only one liter is provided for spiking purposes, use a 500-mL glass cylinder to measure out half the sample. Transfer to a separatory funnel labeled for the Spike. Measure the remaining sample and transfer to a separatory funnel labeled Spike Duplicate. Add 1/2 the normal amount of spiking solution and 1/2 the normal amount of surrogate.

Enter the ID# of the surrogate and spike used and the initials of the person that verified their addition to the sample in the extraction logbook.

8. If necessary, adjust the pH to between 5.0 and 9.0 using NaOH solution or H2SO4. If a pH adjustment is made, the details of the adjustment must be recorded in the extraction logbook.

9. Swirl the 50-mL of methylene chloride that you added to the empty sample bottle and transfer to the corresponding separatory funnel. In addition, add 50 mLs of methylene chloride to the method blank, LCS, and spikes.

10. Seal and shake the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. Alternatively, teflon separatory funnels may be placed in the shaker apparatus with the stopcock slightly open. When this apparatus is used, the shake should be for 3 minutes.

    NOTE: Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once.

11. Allow the sample to set for 10 minutes, if needed, after it has been shaken. It will separate into two layers with the solvent layer on the bottom. If it forms an emulsion (thick, cloudy, viscous, mixture that you cannot see through), drain what you believe to be 50 mL into a 250 mL centrifuge bottle. If the layers are clearly separated, drain the solvent layer into a 400-mL glass beaker.

12. Following Steps 10 and 11, extract two more times with 40 mLs of methylene chloride. Combine the three solvent extracts into the same 400-mL beaker.

13. In the logbook marked Pest/PCB extractions, enter the Client name, the Lab #, the date extracted, and anything unusual that may have occurred with this sample.

14. Dry the sample by either of the following methods:

    14.1 Get a ring stand with a double burette clamp attached to it. Cover the burette clamp ends with aluminum foil to prevent the possibility of solvent touching the plastic coated ends and dripping into the extract. Place a drying column into the burette clamp and transfer a small amount of glass wool to the top of it. Tamp it to the bottom with a glass rod so that it adequately covers the hole at the bottom. Add approximately 10 cm of Sodium Sulfate to the column. Rinse with of methylene chloride 20-30 mL and discard this rinse.

    14.2 Set up a ring stand with funnels. Place a small amount of glass wool in the bottom of it. Add ~2" sodium sulfate to the column and rinse with 20-30 mL methylene chloride. Discard this rinse.

15. If the extract was drained into a centrifuge bottle, at this point you will need to take it to the centrifuge. Push the "ON" button to turn the centrifuge on. Be sure that the orange holders are available for the 250-mL centrifuge bottles. The sample must always be balanced. If necessary, use a dummy bottle with similar weight using reagent water. Set the rpm at 2500 and the temperature at 25°C. Close the lid and be sure to press it down until you hear it lock. Move the lever at the front of the lid to the "LOCK" position. Turn the time to approximately 15 minutes and bring it back to 10 minutes. As the rotor begins to move, you will be able to see the rpm's in the digital readout. Stay with the centrifuge until it has come up to the rpm's set to insure that it does not become unbalanced. This looks like 8888 on the digital readout. Should this occur, refer to the manual. When the cycle is complete, the digital readout will read 0000. Push the "OPEN" button and the lid will pop up. Move the lever at the front of the lid to the "UNLOCK" position. Open lid and remove sample. The sample will usually be in two layers with the extract on the bottom.

16. Remove any water layer from the extract in the beaker or centrifuge bottle, by one of two methods. Remove with a Pasteur pipette by carefully pulling up the water layer, on top, and not the solvent. Discard this layer. Use the smallest amount possible of Na2SO4 by sprinkling the top layer with Na2SO4 until it hardens, separates, and drops to the bottom.

17. TURBO-VAP CONCENTRATION

    17.1. Rinse a Turbo-Vap tube and arrange it underneath a rinsed, packed drying column/funnel. Pour the extract through the column so that it will collect in the tube. Rinse the 400-mL beaker twice with 10 mL of methylene chloride. Rinse the column with 10 to 15 mL of methylene chloride. For solvent exchange purposes add 50 mL of hexane to each tube. Total volume in the glass evaporator tube should not exceed 200 mLs to avoid splattering on the lid of the Turbo-Vap. While waiting for

the drying column/funnel to quit dripping, record the numbers of the Turbo-Vap tube in the Pesticide/PCB logbook and remove the tube to a metal holder.

17.2 Turbo-Vap Operation: Adjust pressure of nitrogen gas tank to >30 psi. Make sure the tank has 200 psi or more on the main valve. Temperature of the bath should be at 40°C -50°C. To help prevent cross contamination, cover the turbovap tube with aluminum foil and punch a small hole in the top.

17.3 Place the glass evaporator tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be in the sensor and need removal. (See Turbo-Vap manual).

17.4 When the Pesticide and/or PCB samples concentrate to approximately one mL, remove the tube from the Zymark.

NOTE: **For PCBs Only*** Some wastewater samples will form a gel like substance when the hexane is concentrated. Proceed with these samples as follows: Add just enough methylene chloride to make the gel go back into solution. Acid clean the extract and reconcentrate. Exchange with hexane again. If gel forms again, add enough methylene chloride to get gel back into solution. Transfer to a suitable container and record the final volume on the label and the extraction logbook. Note the percentage of methylene chloride in sample also.

17.5 When the beep sounds indicating the end of concentration, the extract will be at approximately one – one half mL (half way up tip of tube). Remove the tube from the bath. Hold the tube and the sample vial in one hand at about a 45° angle. Use a 9" Pasteur pipette to draw up the sample and transfer it to the 4.0-mL vial. Spilling a drop or two during the transfer is critical because one drop represents 5% of the sample.

17.6 Add 1 ml of hexane to the turbovap tube. Draw into the pipette and rinse down the conical portion of the tube several times. Transfer this rinse to the 4.0-mL vial. Rinse again with 1 ml of hexane and transfer to the sample vial. Adjust the volume to 4.0 ml with hexane. Cover the extract with a Teflon-sealed screw cap and transfer the label to the vial.

18. The extract obtained above may now be analyzed. Refrigerate to 4°C. Samples must be signed into the Hobart refrigerator. On log provided, enter the sample numbers, analyst initials, and the date and time the samples were placed into the Hobart.

NOTES:
If the final extract is yellow or dark in color or the matrix is oily and viscous, further cleanup may be desired. Refer to the Florisil Cartridge Cleanup Method, the Copper Cleanup Method, and/or the Acid Cleanup Method for further information. (SOP-309, 307, and/or 308)

Pesticides that have aldrin detected in them may be cleaned using the SW-846 method 3630 (Silica Gel Cleanup). The GC Lab would notify extraction personnel if this cleanup is necessary.

VII. DOCUMENTATION OF CAPABILITY (DOC)

Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

VIII. WASTE MANAGEMENT AND POLLUTION PREVENTION

Please see Waste Disposal SOP-405 for the proper 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.

IX. METHOD PERFORMANCE

Refer to SOP-211 for method performance.

X. HEALTH AND SAFETY

Refer to the MSDS sheets for the chemicals used for health and safety information. Also see SOP-336 for proper use of methylene chloride.

REFERENCES

1. *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition
2. 40 CFR, Method 608.

DEFINITIONS

°C - degrees centigrade
COC - chain of custody
DL - detection limit
g - grams
KD - kuderna danish
LCS - laboratory control sample
µg/L - micrograms per liter
µL - microliter
µg/ml - micrograms per milliliter
ml - milliliter
mm - millimeter
MS - matrix spike
MSD - matrix spike duplicate
PCBs - polychlorinated biphenyls
Pest - pesticides
RL - reporting limit
SOP - standard operating procedure
TCMX - tetrachloro-m-xylene
v/v - volume to volume

Refer to SOP-431 for further definitions

HERBICIDES AQUEOUS

MATRIX by METHOD EPA SW-846 8151A

SOP NUMBER: SOP-304

REVISION NUMBER: 11

APPROVED BY:

SECTION MANAGER

QUALITY ASSURANCE OFFICER

EFFECTIVE DATE: 09/23/08

DATE OF LAST REVIEW: 09/23/08

HERBICIDE - AQUEOUS MATRIX EXTRACTION BY EPA Method SW-846 8151A

I. SCOPE AND APPLICATION

1. This SOP describes the extraction of herbicides from water by separatory funnel extraction using SW846 Method 8151A.

II. SUMMARY

1. Aqueous samples are extracted with diethyl ether, dried in acidified sodium sulfate, concentrated, and esterified with diazomethane.

III. INTERFERENCES

1. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
2. Herbicides can react with alkaline substances and may be lost during the extraction process. Glassware must be acid rinsed and sodium sulfate must be acidified.

IV. APPARATUS AND MATERIALS

- Separatory Funnel - 2 Liter with Teflon stopcock
- Beaker - 400 mL
- Fleaker Beaker 1200 mL
- Erlenmeyer flask - 500 mL
- Drying Column (Chromatographic column) - 20 mm I.D. x 300 mm
- Kuderna Danish (KD) flask - 500 mL
- Concentrator tube - 10 mL part of KD setup
- Snyder column - 3 ball, macro
- Plastic connectors - blue to assemble KD
- Water bath - heated, with concentric ring cover, capable of temperature control. The bath should be used in a hood.
- Boiling chips - Teflon
- Vial - 10 mL with Teflon lined lid.
- pH indicator paper - close range (1.0 and 2.0) and (11.0 and 12.0); wide range (1.0 and 12.0)
- Syringe - 1 mL, 500 µL
- Graduated cylinder - Glass, Class A, 1000 mL, 500 mL, 250 mL, and 100 mL
- Pasteur pipette - length 9" and 5 3/4"

- Pasteur pipette bulb
- Labels - Avery
- Volumetrics - Class A, glass, 1000 mL, 50 mL, 25 mL, a 2 mL graduated pipette, and a 1 mL volumetric pipette.
- Bubbler setup
- Ring stand - 3 prong
- Burette clamp - double
- Rings - large enough to hold KD setup
- Aluminum foil - heavy duty
- 10 mL disposable pipette
- Nitrogen tank - equipped with pressure regulator
- Glass manifold - nine positions
- Balance - capable of weighing to 0.1 grams.
- Mortar and pestle

V. REAGENTS

- Reagent water - Reagent water is gathered in a carboy from source in the instrument lab daily. Remaining water in the carboy is dumped at the end of each day.

- Sodium Hydroxide solution - (10N), Weigh 400 g NaOH (purchased in a plastic container from Fisher # S318-3 or equivalent) into a 1200 mL fleaker beaker and cover with reagent water. Swirl periodically until all pellets are dissolved. This mixture gets very hot. Let stand until cool. Transfer to a 1-Liter volumetric flask with several rinses of reagent water and dilute to 1 Liter with reagent water. Transfer to a 1000-mL Teflon container.

- Acidified Sodium Sulfate - In a Pyrex glass dish, pour enough sodium sulfate to cover the bottom. Add 10 mL of concentrated sulfuric acid. Add enough ether to cover the sodium sulfate and stir with a glass rod. Let this mixture stand in the hood until all of the ether has evaporated, stirring occasionally. Put into beakers and store in the 150°C oven until ready to use. **Prepare only one tray at a time to minimize the amount of ether. Pull hood sash all the way down when evaporating ether. Keep anything hot or anything that might ignite the ether away from the hood during evaporation.**

- Acidified Glass Wool - Loosely fill a 400-mL beaker with glass wool. Rinse with 1:1 Hydrochloric Acid (HCL), reagent water, acetone, and ether. Dump waste

in the appropriate waste container. Allow the ether to evaporate and store in the 150°C oven until ready to use.

- Cold Sulfuric Acid solution - (1:3), Slowly add 250 mL of H_2SO_4 (Baker, suitable for trace metal analysis #9673-33) to 750 mL of reagent water in a 1000 mL Teflon container. This mixture will get very warm. Allow to cool before use. Store in the refrigerator at 4°C.

- Potassium Hydroxide solution (37%) Weigh 37 grams of KOH into a 400-mL beaker and cover with reagent water. Swirl until all the pellets are dissolved. This mixture gets very hot. Let stand until cool. Transfer to a 100-mL volumetric flask with several rinses of reagent water and dilute to 100 mL with reagent water.

- Extraction Solvent - Methylene Chloride (**Please read SOP-336 before handling this solvent in our laboratory.)** (Dichloromethane - Omnisolv - suitable for spectrophotometry and gas chromatography #DX0831-1 or equivalent)

- Methanol - suitable for use in spectrophotometry and gas chromatography (Omnisolv MX0484-1 or equivalent)

- Acetone - suitable for spectrophotometry and gas chromatography (Omnisolv AX0116-1 or equivalent)

- Hexane - suitable for spectrophotometry and gas chromatography Omnisolv HX0298-1 or equivalent)

- Ether - ultra-resi analyzed for organic residual analysis (Baker #9259-02 or equivalent)

- Iso-Octane - for pesticide residual analysis (Fisher #0-297 or equivalent)

- Dichlorophenyl acetic acid (DCAA) - purchased from Ultra Scientific (PPS-165) @ 1.0 ppm concentration. Add 1 ml per 1000 mLs of aqueous sample.

- Herbicide Spiking Solution & TCLP Herbicide Spike - A spiking solution is prepared by the GC operator. The same solution is for dual purposes. Use 1 mL of 1-100 ppm solution per 1 liter of aqueous sample. Spiking solution is purchased as an acid.

- Herbicide TCLP Spike – 1mL is added per 100 mL volume. Each matrix type must have its own TCLP spike. From the volume provided by Wet Chemistry, remove a 100 mL aliquot into a suitable container with a Teflon lid, and spike according to the required extraction.

- Sodium Chloride - ultra pure crystals for liquid chromatography (Baker #4058-07 or equivalent) purchased in a 12 kg plastic bucket.

- N-methyl-N-nitroso-p-toluenesulfonamide (Diazald) – High purity (Aldrich Chemical Co., or equivalent), see SOP-328.

- Silicic acid, H_2SiO_5 – 100-mesh powder, store at 130°C (see SOP-328).

VI. PROCEDURE

1. All waters have a seven-day holding time counted from the day they are sampled. Determine the samples necessary using the following information (DO NOT extract samples for which you have no information!):

   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.

   Samples requiring RUSH turn around time may be logged in throughout the day that will require your immediate attention. Login personnel will generally communicate this need.

   Check with login throughout the day and examine the COC (chain of custody) forms that arrive with each set of samples. If an analysis is on-going, extra QC may be avoided by picking up these extractions on the same day.

2. 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 amber jar and have a Teflon lid. Find out if any special dilutions need to be made for this client. Routine procedures for difficult matrices are listed below:

   SLUDGE - use only 100 mL and dilute to 1000 mL with reagent water.

   TCLP EXTRACT - use only 100 mL and dilute to 1000 mL with reagent water. A separate matrix spike of 100 mLs (TCLP spike) is required per client matrix. Dilute to 1000 mL with reagent water.

   BAD MATRIX – for example a liquid that is partially sediment, see your supervisor to find out what dilution, if any, should be made.

3. Mark the amber glass container of each sample at the water meniscus with "white out" for later determination of sample volume. Check the pH by

inverting the sample and touching the wide range pH paper to the portion that remains on the lid. Record this pH in the logbook.

4. Get out enough separatory funnels to extract the number of samples you have plus any additional spikes and a method blank. A method blank and an LCS must be processed with each set of samples. A matrix spike and a duplicate or a matrix spike duplicate must be processed for each analytical batch (up to a maximum of 20 samples). In the event that adequate sample is not provided to do an MS/MSD, an LCS duplicate should be done. Rinse with methylene chloride. Label the separatory funnels as follows on your Avery labels: Lab #, Client name, Type of Analysis, Initial Volume - Final Volume, and the Extraction date. The Method Blank label should include all lab #s in this set of samples.

5. Remove acidified sodium sulfate from the 150°C oven and allow it to cool.

6. Calibrate balance with ASTM class-1 Troemner weights or equivalent, bracketing desired weight (50g, 20g, 10g, 5g, and 1g) and adjust for any corrections. Record the readings in the Extraction calibration/temperature logbook. Weigh 125 grams of Sodium Chloride and transfer it to each sample, spike, and method blank.

7. Using the 1000 mL glass graduated cylinder marked NANO PURE WATER ONLY, measure 1 liter of reagent water from the carboy and transfer it to a separatory funnel for the method blank and LCS. Transfer sample to separatory funnel that corresponds to the lab # on the sample bottle. Add 60 mL of Methylene Chloride to the empty bottle.

8. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.

   Add 1 ml of (DCAA) Herbicide surrogate to each sample, blank and spike with a glass syringe. Someone must verify that the surrogate has been added by placing a check mark on each label as it is added.

   NOTE: Be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution.

   For the sample in each analytical batch selected for spiking, add 1.0 mL of the Herbicide Spiking solution measured with a glass syringe. Someone must verify that the spike has been added by placing a check mark on each label as it is added.

Enter the ID# of the surrogate and spike used and the initials of the person that verified their addition to the sample in the Herbicide logbook.

9. Shake until all the sodium chloride is dissolved. Add 10 mL of 10N Sodium Hydroxide to each sample, spike and method blank.

10. Using short-range pH paper (11 to 12), check pH of sample by touching pH - sensitive paper to the drop of liquid hanging from the glass stopper. Insure that pH throughout the sample is changed. Compare the color to the chart on the pH paper. If the color is not within range add more 10N NaOH as required to attain the proper pH (>12).

11. Set timer for 1 hour. Allow the sample to set for 1 hour shaking occasionally. This is the hydrolysis step.

12. All glassware from this point forward should be acid rinsed. This includes the fleaker beaker, the 500 mL Erlenmeyer flask, the dry column or funnel, the KD setup and the 15 mL centrifuge tube. Rinse each piece of glassware with Hexane, reagent water, 1:1 HCL, reagent water, acetone and finally with ether.

    NOTE: This step is extremely critical in that if any surface of the glassware that might come into contact with the extract is not rinsed with acid, it could cause the herbicides to be lost. Therefore the glassware should be completely submerged in a 1:1 HCL solution rather than just rinsed.

13. Using the balance, weigh 20 grams of acidified sodium sulfate into enough 500 mL Erlenmeyer flasks to cover the number of samples, spikes and method blanks that you are extracting. The acidified sodium sulfate is usually hardened into chunks when it comes from the oven. Use a mortar and pestle to grind up the chunks until they have the consistency of sand.

14. When the hydrolysis time has expired, swirl the 60 mL of methylene chloride that you added to the empty sample bottle and transfer to the corresponding separatory funnel. In addition add 60 mLs of methylene chloride to the method blank, LCS, and spikes.

15. Seal and shake the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. Alternatively, Teflon separatory funnels may be placed in the shaker apparatus with the stopcock slightly open. When this apparatus is used, the shake should be for 3 minutes.

NOTE: Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once.

16. Allow the sample to set for 10 minutes after it has been shaken. It will separate into two layers with the solvent layer on the bottom. If it forms an emulsion (thick, cloudy, viscous, mixture that you cannot see through), drain what you believe to be 60 mL into a 250 mL centrifuge bottle. If the layers are clearly separated, drain the solvent layer into a waste beaker and discard into the Chlorinated Waste Bottle. **Repeat clean-up steps 15-16 two more times.**

17. Adjust the pH to between 1.0 and 2.0. Using a 50 mL cylinder, add 17 mL of 1:3 COLD H_2SO_4 to all sample, spikes, and blank. Shake to insure that pH throughout the sample is changed. Check pH with short-range pH paper (1.0 to 2.0). Compare the color to the chart on the pH paper. If the color is not within range add more H_2SO_4 solution as required to attain the proper pH.

18. Using a 250mL-graduated cylinder, transfer 120 mL of ether to each sample, spike and method blank. Shake for two minutes manually or three minutes if by shaker method and allow the sample to set for 10 minutes.

19. Drain the bottom layer (aqueous phase) of each sample into a fleaker beaker. Let stand for one to two minutes. Water and ether will separate again. Drain water layer as before.

20. Drain the remaining layer (ether phase) of each sample into the Erlenmeyer flasks containing acidified sodium sulfate and pour the aqueous phase back into the separatory funnel.

21. Repeat steps 18 through 20 twice using 60mL of ether and combining the extracts in the Erlenmeyer flask.

22. Put a Teflon stopper into the 500mL flask. Cover with aluminum foil and allow the extract to remain in contact with the acidified sodium sulfate for approximately two hours or overnight in the hood.

    NOTE: The drying step is very critical to ensuring complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling. The 2-hour drying time is a minimum, however the extract may be held in

contact with the sodium sulfate (sealed, and under a fume hood) overnight.

23. In the logbook marked HERBICIDE extractions, enter the Client name, the Lab #, the date extracted, and anything unusual that may have occurred with this sample.

24. DRYING STEP: Dry the sample by either of the following methods:

    24A. Get a ring stand with a double burette clamp attached to it. Cover the burette clamp ends with aluminum foil to prevent the possibility of solvent touching the plastic coated ends and dripping into the extract. Place an acid rinsed drying column into the burette clamp and transfer a small amount of acidified glass wool to the top of it. Tamp it to the bottom with a 10 mL disposable pipette so that it adequately covers the hole at the bottom. Add approximately 10 cm of Acidified Sodium Sulfate to the column. Rinse with 20 to 30 mL of ether and discard this rinse. OR

    24B. Set up a ring stand with funnels. Place a small amount of acidified glass wool in the bottom of each funnel. Add ~2" acidified sodium sulfate and rinse with 20-30 mL ether. Discard this rinse.

25. Turn on the steam bath and the Turbovap well before they are needed to insure that they are at proper temperature. The setting for the steam bath should be set (between 4 and 5 which yields a temperature around 65 to 85° C) for Herbicide extractions. A higher temperature could cause some of the compounds to be destroyed. Insure that there is enough water in the bath. (Water should cover the coils and be at approximately 1/4 inch below the surface of the overflow device.) The setting for the turbovap is at approximately 35 C.

26. Rinse an acid rinsed Kuderna Danish flask and an acid rinsed concentrator tube with ether and assemble together with a blue clip. Set this in the ring on the ring stand so that it will collect extract as it passes through the drying column/funnel. Pour the entire extract through the column/funnel. Rinse the 500 mL Erlenmeyer flask twice with 10 to 15 mL of ether and add to the drying column/funnel. Finally, rinse the drying column/funnel with 10 to 15 mL of ether. Add a boiling chip.

27. Record the numbers of the Kuderna Danish, and the concentrator tube in the Herbicide logbook.

28. Slip a 3-ball Snyder column into the Kuderna Danish flask and set it on the water bath so that the concentrator tube is immersed in the hot water. The temperature of the bath should be around 85°C. Rinse the 3-ball Snyder

with about 1 to 2 mL ether into the KD. As the extract gets hot, the balls will begin to chatter, but the chambers should not flood with condensed solvent. The extract will evaporate in approximately 15 to 20 minutes. When you can no longer see it boiling above the surface of the bath, you need to keep a close vigil on the sample. Take it from the bath momentarily and check the level of extract in the concentrator tube. Don't allow it to quit boiling before placing it back into the water. When it reaches approximately 1/2 the length of the tube or 5 mL, remove it from the bath, set it in a ring on the ring stand and allow it to cool. Remove the 3-ball Snyder and rinse down the sides of the flask with 2 to 3 mL with ether. Dry the union with a paper towel to remove excess water and prevent contamination. Disassemble the KD apparatus being careful not to lose any extract from the concentrator tube. Transfer the extract to a Turbovap tube using a 9" Pasteur pipette.

29. Place the Turbovap Tube in the turbovap. Allow to concentrate to just below the 1 mL mark on the tube. Add enough ether to bring up to 1mL mark on TV tube.

30. Remove sample from the Turbovap and transfer to 12 mL vial using 9" Pasteur pipette. Add 1 mL of Iso-Octane, and 0.5 mL of methanol to each sample.

31. Esterification should be done as soon after concentration as possible. At this point you will need to coordinate with the GC operator as to when the extracts will be run. It is recommended that herbicides be analyzed as soon as possible after esterification to prevent trans-esterification and other potential reactions that may occur. **See ESTERIFICATION METHOD (SOP -328)**

32. Add 4 mL of Diazomethane made using SOP-328. Be sure that the diazomethane has a deep yellow color prior to use. Cover the extract with a Teflon-lined screw cap and allow standing for 20 minutes. A persistent yellow color indicates that esterification is complete and excess diazomethane is present.

33. After 20 mins, bring to final volume with Hexane using Herb dummy vial. Enter the esterification date, the lot number of the diazomethane used and the initials of the person who esterified the sample extracts in the Herbicide logbook. Fill out the labels for the samples and place on appropriate 12 mL vial. Record the vendor and lot number of the methylene chloride, ethyl ether, isooctane, methanol, and hexane used in the sample extract in the logbook.

34. 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 Herbicide logbook and the Avery label to the nearest 10 mL.

34. The extract is now ready to be analyzed. Refrigerate at 4°C or carry directly to the instrument operator. Samples must be signed into the Sample Extract refrigerator. On log provided, enter the sample numbers, the analyst initials, and the date and time the samples were placed into the refrigerator.

VII. WASTE MANAGEMENT AND POLLUTION PREVENTION

Please see Waste Disposal SOP-405 for proper disposal of waste from this sample preparation process. 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.

VIII. REFERENCES

1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III)*; Method *8151A*

2. *USACE EM 200-1-3, 02-2001; Appendix I; Shell for Analytical Chemistry Requirements*

3. DOD, *Quality Systems Manual for Environment*

IX. HEALTH AND SAFETY

1. 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.

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.

3. MSDS are available for all reagents and standards, which have been purchased. These are located in the office next to the technical director.

4. Use extreme caution when working with concentrated acids and bases. Use safety glasses, or goggles, gloves and lab coat or apron.

4. Diazomethane is a carcinogen and may explode under certain conditions. See Method 8151 for details in handling diazomethane.

X. DOCUMENTATION OF CAPABILITY (DOC)

Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

XI. METHOD PERFORMANCE

Refer to SOP-208 for method performance.

DEFINITIONS

°C - degrees centigrade
COC - chain of custody
DL - detection limit
g - grams
KD - kuderna danish
LCS - laboratory control sample
µg/L - micrograms per liter
µL - microliter
µg/ml - micrograms per milliliter
ml - milliliter
mm - millimeter
MS - matrix spike
MSD - matrix spike duplicate
RL - reporting limit
SOP - standard operating procedure
v/v - volume to volume

Refer to SOP-431 for further definitions

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

SOP NUMBER: SOP-327

REVISION NUMBER: 14

APPROVED BY:

SECTION MANAGER

QUALITY ASSURANCE MANAGER

EFFECTIVE DATE: 09/17/08

DATE OF LAST REVIEW: 09/17/08

METHOD 8330A and 8332

NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

1.0 SCOPE AND APPLICATION

This SOP, based on SW-846 Method 8330A 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. See SOP-431 for common environmental laboratory definitions.

2.0 SUMMARY OF METHOD

Samples are analyzed after appropriate sample preparation using HPLC with identification at 254nm on a C-18 reverse phase column and confirmation at 254nm on a Phenyl-hexyl column. The method can be extended to include PETN and nitroglycerin by analyzing the samples at a wavelength of 210 nm. 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.

3.0 INTERFERENCES AND POTENTIAL PROBLEMS

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.

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.

Degradation products of tetryl appear as a shoulder on the 2,4,6-TNT peak on the synergi column. 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.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

Samples are collected and handled as specified for semivolatile organics, (SW-846 Update IV, Chapter Four). Section 7.0 and table 6-1 and 6-2 of the Empirical Laboratories' Quality Assurance Manual include details concerning sample preservation, containers and handling of semi-volatile samples. Samples and sample extracts are stored in the dark at 4°C. Samples are stored in the sample storage walk-in cooler. Extracts are stored in the Hobart in the extraction laboratory. The holding time for samples is 7 days for waters and 14 days for soils. The holding time for extracts is 40 days.

5.0 INSTRUMENTATION, APPARATUS AND MATERIALS

5.1 Instrumentation

Agilent Series 1100 HPLC System

- Agilent G1311A Quaternary Pump
- Agilent G1379A Degasser
- Agilent 1313A 100 Position Autosampler
- Agilent G1316A Column Compartment
- Agilent G1314A Variable Wavelength Detector
- Agilent HPLC Chemstation

Hewlett Packard Series 1050 HPLC System

- Hewlett Packard Model 79852 1050 Quaternary HPLC Pump
- Hewlett Packard Model 79853 Variable Wavelength UV-VIS Detector
- Hewlett Packard 1050 79855A 21 Position Autosampler
- Hewlett Packard 1050 100 Position Autosampler Upgrade
- Hewlett Packard Model 79856A 1050 Solvent Module
- Hewlett Packard Model G1303A Vacuum Degassing Module
- Dell OptiPlex 933 GX150 Pentium III Computer
- Hewlett Packard PC Communication for HP Chemstation
- Hewlett Packard HPLC Chemstation
- SideWinder Temperature Control Module

5.2 Apparatus and Materials

- Solid-phase extraction system consisting of:
  - Manifold Station, *J.T. Baker spe-12G*, or equivalent
  - Tubing and connectors
  - SFE extraction cartridges, *Porapak®RDX Cartridges* or equivalent
  - Vacuum system capable of maintaining 18 inches of mercury
- Branson ultrasonic bath cooled by a chiller to 15-20°C
- Balance ± 0.01 g.
- Vortex mixer.
- Shaker
- Disposable cartridge filters - 0.45 mm PTFE filter.
- Scintillation Vials - 20 mL, glass.

- Vials - 15 mL, glass, Teflon-lined cap.
- Vials - 40 mL, glass, Teflon-lined cap.
- Disposable syringes - Plastipak, 3 mL and 10 mL or equivalent.
- Volumetric flask with ground glass stopper - 100 mL and 1000 mL.
- Vacuum desiccator - Glass.
- Mortar and pestle - Steel.
- Sieve - 30 mesh.
- Graduated cylinders - 10 mL, 25 mL, 250 mL, 1000 mL.
- Pasteur pipet - length 9 ".
- Manual Sample Mill.
- Wiley Sample Mill.
- Clippers for cutting vegetation

6.0 REAGENTS AND SOLUTIONS

6.1 Reagents

- Acetonitrile, CH_3CN - HPLC grade.
- Methanol, CH_3OH - HPLC grade.
- Calcium chloride, $CaCl_2$ - 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.
- 1% Acetic Acid, CH_3COOH, Solution – Prepare using 5.0mL CH_3COOH to 500mL of Organic free reagent water.
- Sodium Chloride solution - 325 g NaCl per 1000 mL reagent water.

6.2 Calibration/Method Standards

Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label and recorded in the Organic standards logbook. The date they are opened is noted on the label and recorded in the Organic standards logbook along with their lot number and vendor. Each standard that is prepared is recorded in the Organic standards logbook and given a sequential number. Each standards label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. All standards are stored in the refrigerator in the dark at a temperature of 4°C $\pm$ 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 Organic standards logbook.

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

Explosive (Mix#1,#2 and Surrogate) Calibration Stock Solutions: Using a 100µL syringe, 100µL of Mix # 1, Mix # 2, and Surrogate (Restek at 1000 µg/mL) are injected into a 10mL volumetric flask containing approximately 9.5mL 1:1 acetonitril and water and diluted to volume with same to make a 10 µg/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 µg/mL. The lowest calibration standard can be extended down to 0.010µg/ml, if required.

Mix # 3 Calibration Stock Solution: Using a 500µL syringe, 500µL of Mix # 3 (Restek PETN & Nitroglycerin, NG, at 1000 µg/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 µg/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 µg/mL. The working calibration standards must be prepared fresh the day of use.

- 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 µg/mL. Samples are spiked with 1 mL of this solution.

- 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.

  Second Source Stock Solutions: Using a 100µL syringe, 100µL of Combined Stock Solution (Ultra at 1000 µg/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 µg/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 µg/mL.
  Using a 500µL syringe, 500µL of Mix #3(Accustandard at 1000 µg/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 µg/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 µg/mL.

- 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.

  Surrogate Spiking Solution: 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.

7.0 PROCEDURE

All waters have a seven-day holding time and soils have a 14-day holding time. Determine the samples necessary to extract as follows:

- 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.

- 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.

- 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.

- 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.

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.

7.1 High Level Aqueous Extraction Method

- 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. The lab prep batch code is defined as MMDDYYEW# where #: 1 = 1st Method Blank for the day; 2 = 2nd Method Blank for the day; etc. The Method Blank and LCS label should include all lab #s in this set of samples.

- 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.**

7.2 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.

- 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).

- Assemble the manifold for multiple extractions with SPE cartridges.

- 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.**

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

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

- Empty the water trap as needed.

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

- 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. The lab prep batch code is defined as MMDDYYEW# where #: 1 = 1st Method Blank for the day; 2 = 2nd Method Blank for the day; etc. The Method Blank and LCS label should include all lab #s in this set of samples. Bring extract up to 4 ml with acetonitrile and add DI water up to 8 ml. Record this volume in the HPLC extraction logbook. The extract is ready for analysis, proceed to Section 8.0.

- 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 HPLC extraction logbook to the nearest 10-mL. Transfer this information to the Avery label also.

7.3 Soil and Sediment Samples

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 partical size, then the sieve would not be required.

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.

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.

- 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. The lab prep batch code is defined as MMDDYYES# where #: 1 = 1st Method Blank for the day; 2 = 2nd Method Blank for the day; etc. The Method Blank and LCS label should include all lab #s in this set of samples. 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 40 mL vial. To each QC sample, (LCS, MS&MSD), add 8.0 mL of acetonitrile. Then add 1.0 mL of surrogate (2.0 µg/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 with a Teflon-lined cap and place in a shaker for 16-18 hours.
- 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.

8.0 HPLC ANALYSIS

8.1 Chromatographic Conditions

Agilent Series 1100 HPLC System

- Primary Column: C-18 reverse phase HPLC column, 25 cm x 4.6 mm, 5 µm.
- Mobile Phase: 56% methanol/44%water.
- Flow Rate: 1.0 ml/min.
- Injection Volume: 100 µL.
- UV Detector: 254 nm.(210 nm for PETN and nitroglycerin)
- Temperature: 28 degrees Celsius.

Hewlett Packard Series 1050 HPLC System

- Secondary Column: Phenyl-hexyl HPLC column, 25 cm x 4.6 mm, 5 µm.
- Mobile Phase: 37% acetonitrile/63%water.
- Flow Rate: 1.5 ml/min.
- Injection Volume: 100 µL.
- UV Detector: 254 nm.(210 nm for PETN and nitroglycerin)
- Temperature: 28 degrees Celsius.

8.2 Calibration of HPLC

- 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 and then pumped with the appropriate solvent/water mixture for an additional 45 minutes.
- 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. Linearity may be evaluated by calculating the percent relative standard deviation (*%RSD*). Alternatively the average response factor (*RF*) for each analyte may be used if the *%RSD* is less than 20%. Linearity is presumed if r is equal to or greater than 0.995, if r^2 is equal to or greater than 0.99 using six points. QC elements and acceptance limits are summarized in Table 1-8, pg. 22, taken from USACE *Appendix 1, Shell for Analytical Chemistry Requirements;* EM-200-1-3 2/01/01. Also found in Table B-2 of the *DoD Quality Systems Manual*, *Version 3*, January, 2006.
    - 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.

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.

- Check the instrument operating conditions or the initial calibration standards used and make adjustments to achieve a linear calibration curve.
- 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.
- 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.
- 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).

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). The percent recovery for the ICV target analytes determined from the initial calibration curve must agree within ± 15% (**80-**120% **DOD QSM Ver. 3**). If not, the source of the problem must be identified before analysis continues.

- 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(**at the beginning and end of sequence and after every 10 field samples, 20%D no average DOD QSM Ver.3**). The response factor for the CCV must agree within 15% of the response factor of the initial calibration. The same criteria is required for subsequent CCV responses compared to the response factor of the CCV beginning the day. If the CCV criteria are not met and a reanalysis does not meet, a new initial

calibration curve may be needed. The same analyte limitations, as mentioned above for initial calibration, for certain compounds that make the stated method acceptance criteria unattainable are evaluated for percent differences, *%D*, for CCVs. Evaluate the mean of the *%D* or percent drift values for all reported target analytes in the calibration verification standard to verify whether it is less than the method acceptance criteria. To avoid the inclusion of target analytes showing gross method failure, this approach may be utilized as long as the target analytes do not exceed the criteria for poor performers in Table 1-8 pg. 22 (same as above). **In addition the client must be notified of all target analytes exceeding method acceptance criteria, the individual *%D* values for those compounds, and the mean *%D* calculated. DOD QSM Ver. 3 does not allow averaging for the CCV.**

8.3 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 ± 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 it's 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.

8.4 Sample Analysis

- 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). See section 9.0 for detail concerning the QC samples.
- Analyze the samples using the same conditions as the standards. Compounds identified on the C-18 column must be confirmed by injection on the Phenyl-hexyl 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 higher result. Results should be flagged with a ***'P'*** on the report. (**"J" for DOD QSM projects)**

- 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 Phenyl-hexyl column. Column temperature control is employed so retention time shifts should not be a problem.

8.5 Data Reduction/Evaluation

- Each sample analysis sequence is documented in the HPLC run log. After the samples have been analyzed, the data is reduced using Target. All calculations are performed as covered in section 8.6 of this SOP. The following must be checked to determine if the sample will need any reanalysis or dilution:

  1. The initial CCV must be within ±15 % difference of the calibration curve. Each CCV that follows must be within ±15% difference of the calibration curve. Samples analyzed after a CCV which does not meet these criteria must be evaluated per section 8.2 and may require to be reanalyzed with a CCV which meets these criteria. See Table 1-8, pg. 22 (same as above). (**at the beginning and end of sequence and after every 10 field samples, 20%D no average DOD QSM Ver.3**)

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

  3. Surrogate recovery must be within the limits established by the laboratory or default values (USACE-Shell, Appendix 1; EM-200-1-3, 2/01/01.) of 60%-140% for water and 50-150% for solids/project sample matrix(**When analyzing samples for DOD QSM Ver. 3 projects, use DOD limits**). If the surrogate recovery falls outside the limits, the sample may need to be reextracted to verify matrix affect or the sample data must be qualified. See Table 1-8, pg. 22 (same as above).

     - After the data has been reduced and determined to be acceptable, it is placed on a sample report and reviewed. Formal data evaluation is detailed in SOP-216 and documented using the USACE Analyst Data Review Checklist for USACE projects (example on pages 20 and 21). 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 SOP-224 for guidance.**

8.6 Calculations

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

Peak Area (or Height) of the Compound in the Standard

CF = Mass of the Compound Injected (in nanograms)

- The mean CF is calculated as follows:

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

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

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

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

- 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.

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

- 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:

<u>Aqueous Samples</u>**:**

Concentration (µg/L) = $[(A_s)(V_t)(D)]/[(\overline{CF})(V_i)(D)]$

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 µg/L.

<u>Nonaqueous Samples</u>**:**

Concentration (µg/kg) = $[(A_s)(V_t)(D)]/[(\overline{CF})(V_i)(W_s)]$

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, $\overline{CF}$ and V_i have the same definition as for aqueous samples.

9.0 QUALITY ASSURANCE/QUALITY CONTROL/CORRECTIVE ACTIONS

9.1 Surrogate - All samples and QC are spiked with the surrogate. The surrogate recoveries from method blanks, LCS and samples are charted to generate control limits. Default limits (USACE-Shell Appendix 1; EM-200-1-3; 2/01/01) are set at 60-140% for water and 50-150% for

solids/project sample matrix. Surrogate recoveries must be within these limits for all samples and QC or reextraction/reanalysis may be performed to verify matrix affect. If matrix affect is confirmed or it is decided not to reextract/reanalyze, a corrective action report must be completed and forwarded within the same shift in which the problem was discovered. Also, the data must be flagged for QC problems on the final report. See Table 1-8, pg. 22 (same as above).**DOD limits will be used for DOD QSM Ver. 3 projects.**

9.2 LCS Sample - The LCS is analyzed at the frequency required by the regulatory agency or client (every batch or 20 samples, whichever comes first, for USACE projects). To prepare the LCS, a blank is spiked with the calibration standards. The recoveries are charted to generate control charts and limits. The default limits (USACE-Shell Appendix 1; EM-200-1-3; 2/01/01) are 60-120% recovery for waters and soils if the generated limits are wider or if limits have not yet been generated. If the LCS compound has a recovery above the upper limit, but the same compound is not detected in any of the batch samples, no corrective action is required. For all other situations, the LCS should be reanalyzed for the failed analytes only. If the second analysis fails, all associated samples should be reextracted/reanalyzed for the failed analytes only. All recovery problems need to be addressed and steps taken to locate their source. **DOD limits will be used for DOD QSM Ver. 3 projects.**

9.3 Method Blanks - The concentration of all method target analytes must be below ½ of the method reporting limit, MRL (USACE-Shell, Appendix 1; EM-200-1-3, 2/01/01; threshold value below which a result is reported as non-detected), for each method target analyte. The first step of corrective action is to assess the effect on the samples. If an analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps should be taken to find/reduce/eliminate the source of this contamination in the method blank. If an analyte is found in the method blank and some, or all, of the other batch samples, then corrective action is required. The source of contamination must be investigated and appropriate action taken and documented to find/reduce/eliminate the source of this contamination. The method blank, and any samples containing the same contaminant, would likely be reextracted/reanalyzed. If a contaminant is found in the method blank and the samples, the compound concentration must be flagged with a 'B' on the final report unless the concentration is greater than 10x that found in the method blank. In any case, a corrective action report must be completed and forwarded within the same shift in which the contaminant is found.

9.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD. MS/MSD recovery limits are set at 50-140% recovery with a maximum RPD of 50% for normal analytes and 60% for poor performers (See Table 1-8 pg. 22; same as above). Generally, batch control is not based on MS/MSD results unless general

method failure is determined to be the problem. In that case, the samples and associated QC would be reanalyzed for the failed analytes only. MS data evaluation must include the consideration of the following factors. **DOD limits will be used for DOD QSM Ver. 3 projects.**

9.4.1 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.

9.4.2 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.

9.4.3 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.

9.4.4 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.

9.4.5 Documentation of Capability (DOC) - Each analyst must perform a DOC to demonstrate proficiency with this method. See SOP-414 for guidance.

9.4.6 MDLs are performed annually or when there is a change in the method or MDL checks are analyzed quarterly per DOD QSM Ver.3. See SOP-414 for details.

10.0 HEALTH, SAFETY, WASTE MANAGEMENT AND POLLUTION PREVENTION

10.1 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!

10.2 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.

10.3 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.

10.4 MSDS sheets are available for all reagents and standards which have been purchased. These are located in the office next to the technical director.

10.5 Please see Waste Disposal SOP-405, for instruction of proper 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.

REFERENCES

1. *40 CFR, Part 136*; Appendix A
2. *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition
3. *National Environmental Laboratory Accreditation Conference*; Chap. 5, 2003
4. USACE, *EM 200-1-3; Appendix 1; Shell*, 2/2001
5. *DOD Quality Systems Manual for Environmental Laboratories*, Ver. 3, Jan. 2006.

ANALYST DATA REVIEW CHECKLIST

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

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?				

ANALYST DATA REVIEW CHECKLIST
8330 (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 ½ the MRLs?				
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: _____ _____

Table I-8

Summary of measurement quality objectives for Method 8330 Explosives (USACE – Shell 2/01)

QC Element	Target Analyte/Surrogate	Poor Performers/Sporadic Marginal Failures[1]
Initial Calibration (I.9.2.2.8)	Primary Evaluation: r ≥ 0.995, RSD ≤ 20%, r^2 ≥ 0.990 Alternative Evaluation: Mean %RSD for all target analytes ≤ 20% with maximum allowable restriction noted at right for individual analytes.	No allowance Alternative Evaluation: Maximum allowable %RSD for each individual target analyte ≤ 40%
ICV (I.9.3)	%Rec = 85% - 115%	No allowance
CCV (I.9.5/I.9.5.2)	Primary Evaluation: %Drift ± 15%, %D ± 15% Alternative Evaluation: Mean %Drift/%D for all target analytes ≤ 15%, with maximum allowable restriction noted at right for individual analytes.	**No allowance** Alternative Evaluation: Maximum allowable %Drift/%D for each individual target analyte is ± 30%
MB (I.10.2.1/I.11.4.1)	Target Analytes: Analytes < one-half MRL	Not applicable
LCS (I.10.2.2/I.11.4.2)	Water: %Rec = 60% - 120%[2] Solids: %Rec = 60% - 120%[2]	Sporadic Marginal Failures[1]: %Rec = 40% - 150%
MS (I.10.2.4/I.11.4.3/I.11.4.3.2)	%Rec = 50% - 140%[2]	Sporadic Marginal Failures[1]: %Rec = 40% - 150%
MSD/MD (I.10.2.2/I.11.4.4)	RPD ≤ 50%	RPD ≤ 60%
Surrogates (I.10.2.5/I.11.4.5)	Interference-Free Matrix: Water: %Rec = 60% - 140% Solids: %Rec = 50% - 150% Project Sample Matrix: %Rec = 50% - 150%	Not applicable
Target Analyte Confirmation (I.12.3)	RPD ≤ 40%	RPD ≤ 40%

[1] The number of sporadic marginal failure (SMF) allowances depends upon the number of target analytes reported from the analysis. For instance, if between 7 to 15 explosives are reported from the high-performance liquid chromatography analysis, 1 SMF is allowed to the expanded criteria presented for the LCS. If greater than 15 explosives are reported, 2 SMFs are allowed for the LCS. If the MS includes only a subset of compounds, allow only 1 SMF for this QC element.

[2] Due to the tendancy for Tetryl to decompose, an expanded criteria may be applied at 45% -140% for both the water and soil matrices.

SOXHLET EXTRACTION -

BNA AND PEST/PCB

USING SW846 METHOD 3541

SOP NUMBER: **SOP-329**

REVISION NUMBER: **16**

APPROVED BY:

SECTION MANAGER

QUALITY ASSURANCE OFFICER

EFFECTIVE DATE: **09/24/08**

DATE OF LAST REVIEW: **09/24/08**

BNA& Pesticide/PCB NON-AQUEOUS MATRIX (Soxhlet Extraction) USING SW846 METHOD 3541

I. SCOPE AND APPLICATION

1. This SOP describes the extraction of BNAs and pesticides/PCBs from soil, sediment, sludges and waste solids by an automated method (3541).

II. SUMMARY

1. Soil and solid samples are mixed with sodium sulfate and extracted with solvent in a Soxtherm extractor for BNAS or Pesticides/PCBs.. The extracts are then concentrated by either a TurboVap concentrator or by Kuderna- Danish.

III. INTERFERENCES

1. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
2. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
3. Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

IV. APPARATUS AND MATERIALS

- Stainless steel spatula
- Soxtherm automated extractor unit-with 6 position condenser assemblies, internal plumbing, electronic components, stand alone controller unit and a copper collection tank for spent solvent
- Soxtherm extraction beakers-54 x 130 mm, capacity approximately 125-175 mL
- Suitable thimble (33 mm diameter by 80 to 94 mm length cellulose or equivalent
- Drying Column (Chromatographic column) - 20mm I.D. x 300mm
- Boiling chips - Teflon
- Vial – 2-mL amber with Teflon-lined screw cap
- Vial – 12-mL clear with Teflon-lined screw cap
- Syringe - 1 mL, 500 µL
- Graduated cylinder - Glass, Class A, 100 mL
- Pasteur pipette - length 9" and 5-3/4"
- Pasteur pipette bulb
- Labels - Avery
- Ring stand - 3 prong
- Burette clamp - double
- Rings

- Aluminum foil - heavy duty
- Nitrogen tank - equipped with pressure regulator
- TurboVap Concentrator with 200 ml concentrator tubes
- Balance - capable of weighing to 0.1 grams.
- Aluminum pie pans for mixing samples
- Glass wool- Contaminant free.

V. REAGENTS

- Sodium Sulfate (Na2SO4) - Granular, anhydrous, trace pure 10-60 mesh (purchased in bulk containers from Fisher # S415-10S or equivalent) placed in a Pyrex tray and heated at 400 C for a minimum of 4 hrs, removed and then cooled in open air in the extraction lab, placed in a 2.5kg glass amber jug and left at room temperature.

- Glass Wool - Silane Treated (purchased from Supelco #2-0410 or equivalent).

- Methylene Chloride **(Please read SOP-336 before handling this solvent in our laboratory.)** (Dichloromethane) - suitable for spectrophotometry and gas chromatography (Omnisolv #DX0831-1 or equivalent)

- Acetone - suitable for spectrophotometry and gas chromatography (Omnisolv AX0116-1 or equivalent)

- Hexane - suitable for spectrophotometry and gas chromatography (Omnisolv HX0298-1 or equivalent)

- Hexane/Acetone (1:1) (v/v) of the above reagent grades.

- Methylene Chloride/Acetone (1:1) (v/v) of the above reagent grades.

- Surrogate/Spike Solutions - Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes or if the initial concentration of stock is different than that listed below:

  1. **BNA Surrogate (100 ug/ml)** - The base neutral and acid surrogates are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor. Use 0.5 mL of this solution per 15g of non-aqueous sample. **(For low level PAHs use 1.0 ml of 1.0 µg/mL BN Surrogate spiking solution.)**
  2. **BNA Spiking Solution (100 ug/ml)** - The base neutral and acid spiking solutions are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor with same compounds as for calibration. Use 0.5 mL of this solution per 15g of non-aqueous sample.**(For low level PAHs use 1.0 ml of 1.0 µg/mL PAH spiking solution.) The BNA Spiking solutions contains all targets that are**

calibrated for GC/MS. DOD QSM requires all targets to be spiked in the LCS and MS/MSD.

3. **TCMX/DCB (2,4,5,6-Tetrachloro-meta-xylene/Decachlorobiphenyl)Warm and sonicate** - Surrogate solution is prepared in acetone by making a cut on stock purchased from a reputable vendor. 0.5 mL at 0.5 µg/mL of this solution is added per 15g of non-aqueous sample.
4. **PCB Spiking Solution-** Arochlor 1016/1260 or the PCB of choice (1242, 1248, 1254, or 1260 are the most common) is prepared in acetone at a concentration of 5.0 ug/ml. PCB stock is usually purchased from RESTEK or equivalent. The PCB to use may be determined by viewing historical data or asking the GC operator. Use 0.5 mL per 15.0 grams of non-aqueous sample.

5. **Pesticide Spiking Solution** - A spiking solution is prepared at 1.0 ug/ml. Use 0.5 mL per 15 grams of non-aqueous sample.

VI. SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

1. Samples are collected in an appropriate size wide-mouth glass jar (4oz. or 8 oz.) with a teflon-lined cap.
2. Samples are preserved by cooling to 4° C.
3. Holding time is 14 days from collection date to extraction.

VII. PROCEDURE

Extraction Procedure

1. All soils have a 14 day holding time counted from the day they are sampled.Determine the samples necessary to extract using the following information (DO NOT extract samples for which you have no information.):
   - 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.
   - 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.
   - 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.

2. 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 and have a Teflon lid. Find out if any special dilutions need to be made for this client. If the sample has a particularly bad matrix or a strange matrix, see your supervisor to find out if a soxhlet extraction is truly necessary.

3. Get twice the number of aluminum pie pans to prepare the number of samples you have plus any additional spikes or LCSs and a method blank. A method blank and LCS must be processed with each set of samples. A matrix spike, a duplicate or a matrix spike duplicate and a LCS must be processed for each analytical batch (up to a maximum of 20 samples). Make an Avery label for each extract containing the following information: Lab #, Client name, Type of Analysis, Initial Weight - Final Volume, and the Lab prep batch code. The lab prep batch code for BNAs is defined as MMDDYYBs# where #: 1 = 1st method blank of the day; 2 = 2nd method blank of the day; etc. The lab prep batch code for Pest/PCBs is defined as MMDDYYPs# using the same logic. The Method Blank and LCS labels must include all lab #s in this set of samples.

4. Decant and discard any water layer on a sediment sample by carefully pouring this off into a trashcan.

5. Dump the entire sample into an aluminum pie pan and mix sample thoroughly with a spatula until mixture is homogenous. Discard any foreign objects such as sticks, leaves, and rocks.

   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

6. Place an aluminum pie pan on the balance and zero it. Calibrate balance with ASTM class-1 Troemner weights or equivalent, bracketing desired weight (50g, 20g, 10g, 5g, 1g). Record calibration in the Extraction calibration/temperature logbook. Using a spatula, transfer the **appropriate weight, {10-20 grams depending upon client or project specific Detection Limits (DL) and/or Reporting Limits (RL)}**, of a representative sample to the nearest 0.1 gram. Normally 10 or 15 gram sample weights are used. Record this amount on your label. Put your label on the side of the 400-mL beaker. For spiking purposes, weigh 3 aliquots of the appropriate sample. Pick a sample with a good matrix, one that mixes well, non-oily, etc.

7. Add ~ 20 grams of sodium sulfate to the aluminum pie pan. Using a spatula and/or a glass rod, mix the sample thoroughly with the sodium sulfate until it becomes a sandy texture. If necessary, add additional sodium sulfate. When removing the spatula or glass rod from the mixed sample, leave behind all the sample possible. Cover the aluminum pie pan with foil and continue to weigh up the remaining samples. For the method blank and LCS, simply weigh up 15 grams of sodium sulfate. The matrix used for the method blank and LCS must be free of the analytes of interest and processed through the same analytical steps as the samples.

8. Transfer the sample and its label to a soxtherm beaker, which has been prepared using a glass rod to place glass wool in and over the hole at the bottom side.

9. Make up 1:1 Methylene Chloride/Acetone (**for BNAs**) or 1:1 Hexane/Acetone (**for Pest/PCBs**) by mixing 2000ml of methylene chloride or hexane with 2000 ml of acetone in a 4000ml amber jug. Add 120 mL of this mixture to the soxtherm beaker for each sample, method blank, and spike - add boiling chips.

10. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature. Someone must verify that the surrogate/spike has been added by placing a check mark on the label as it is added.

    NOTE: Surrogate and spike should be added just prior to setting on the Soxtherm.

    Using the 1-mL glass syringe designated for BNA surrogate, add 0.5 mL of BNA surrogate to each sample, spike, and blank. **(For low level PAHs use 1.0 ml of the 1.0 µg/mL BN Surrogate spiking solution.)** or using the 1.0-mL glass syringe marked TCMX/DCB surrogate, add 0.5 mL of TCMX/DCB surrogate to each sample, blank and spike.

    For the BNA sample in each analytical batch selected for spiking, use the 1.0-mL glass syringe marked Base Neutral Acid Spiking to add 0.5 mL of the Base Neutral Acid Spiking solution. . **(For low level PAHs use 1.0 ml of the 1.0µg/mL PAH spiking solution.)**
    For Pest/PCB samples, determine if the sample will require a Pesticide Spike and/or a PCB Spike. Proceed as follows:
    Pesticide and PCB - set up two LCS's – one for Pesticide getting a MIX A&B spike and one for PCB which should be spiked with PCB 1660. In addition to the LCSs, a matrix spike/matrix spike duplicate is necessary for the pesticide. Prepare a PCB matrix spike/ matrix spike duplicate if requested by the client.
    Pesticide only – To the sample in each analytical batch selected for spiking, add 0.5 mL of Pesticide Spike (Mix A&B) with a glass syringe dedicated for Pesticide Spike.
    PCB only - To the sample in each analytical batch selected for spiking, add 0.5 mL of PCB 1016/1260 (unless otherwise specified, 1248 for BB&L) using a 1.0 mL glass syringe dedicated to that PCB. Add 20 grams of Na_2SO_4.

11. **Automated Soxhlet:** The *Soxtherm* will extract any number of samples up to six per run. Generally, we set up to complete four sets of six a day or/as sample load permits with required QC. Each extraction cycle takes 1-2 hours. Reference *O.I. Analytical Operator's Manual* for programming and making adjustments to extraction cycle settings.

- The extraction thimbles are placed into the respective extraction thimble holders(the open end of the thimble is nearly flush with the upper edge of the metal ring. Transfer the sample into the extraction thimble. The appropriate spiking solution should be added at this point (surrogates, MS/MSD or LCS etc…). (Placing a small piece of glass wool sufficient to cover the diameter of the thimble will help hold the sample mixture in the thimble.)

- Place 1-2 boiling chips in each extraction beaker and place each extraction thimble into the appropriate labeled beaker then add 120 mLs of the appropriate extraction solvent. (1:1 Acetone/Hexane for Pest/PCBs **or** 1:1 Acetone/Methylene Chloride for BNAs.)

- For loading samples on the *Soxtherm,* the condensers should be in their raised position. If Viton gaskets are to be used, install them in the extraction beakers so that a good seal will form between the extraction beaker and the Teflon extraction cylinder. Install the extraction beakers by depressing the holding clamp and carefully sliding the beaker onto or off the bottom of the Teflon fitting that is situated below the glass condenser. After the extraction beakers containing the samples have been loaded on the *Soxtherm,* push all beakers against the rear limit stop of the hot plate.

- The *Soxtherm* is now ready to run with one of the programs listed below (**remember to check the sight glass on the front of the Extraction Unit to see if the solvent collection tank is near full and should be drained**).

Pest/PCB Extraction Program

Temperature	= 165°C (Maximum temperature 200°C)
Boiling Time	= 30 minutes
Solution Reduction A	= 5x15 ml
Extraction Time	= 30 minutes
Solution Reduction B	= 0 minutes
Solution Cooling	= 0 minutes
Solution Reduction Interval	= 2.5 minutes
Solution Reduction Pulse	= 2 seconds

BNA Extraction Program

Temperature	= 160°C (Maximum temperature 200°C)
Boiling Time	= 30 minutes
Solution Reduction A	= 5x15ml
Extraction Temperature	= 30 minutes
Solution Reduction B	= 0 minutes

Solution Cooling	= 0 minutes
Solution Reduction Interval	= 2.5 minutes
Solution Reduction Pulse	= 2 seconds

- At the end of Solution Cooling time the condensers and extraction beaker assemblies are automatically raised off the heating block and the process completion message appears on the display. Remove the beakers to a hood and continue with the process as listed below.

13. Enter the following information in the BNA, Pesticide and/or PCB extractions logbook: Client name, Lab #, date extracted, initial weight, final volume, soxtherm position, and anything unusual that may have occurred with this sample. Enter the ID# of the surrogate and spike used and the initials of the person that verified their addition to the sample in the appropriate logbook. Also record the vendor and lot number of the methylene chloride or hexane and acetone used.

14. Dry the sample by either of the following methods:

14A. Get a ring stand with a double burette clamp attached to it. Cover the burette clamp ends with aluminum foil to prevent the possibility of solvent touching the plastic coated ends and dripping into the extract. Place a drying column into the burette clamp and transfer a small amount of glass wool to the top of it. Tamp it to the bottom with a glass rod so that it adequately covers the hole at the bottom. Add approximately 10 cm of Sodium Sulfate to the column. Rinse with 20 to 30 mL of methylene chloride or hexane and discard this rinse into the appropriate waste container (make certain that methylene chloride waste goes in the chlorinated solvent waste container in the hood).

14B. Set up a ring stand with funnels. Place a small amount of glass wool in the bottom of it. Add ~2" sodium sulfate to the column and rinse with 20-30 mL methylene chloride or hexane for Pest/PCBs. Discard this rinse.

15. TURBO-VAP CONCENTRATION

A. Turn on the TurboVap and set the temperature at 40°C -50°C. This temperature is necessary to evaporate the acetone. Adjust pressure of nitrogen gas tank to >30 psi. Make sure the tank has 200 psi or more on the main valve.

B. Rinse a Turbo-Vap tube and arrange it underneath a rinsed, packed drying column/funnel. Pour the extract through the column/funnel so that it will collect in the tube. Total volume in the glass evaporator tube should not exceed 200 mLs to avoid splattering on the lid of the Turbo-Vap. Therefore the process cannot be completed in one step. Pour enough of the extract from the 400 mL beaker to reach the 200 mL level mark on the Turbo-Vap tube. To help prevent cross contamination, cover the turbovap tube with aluminum foil and punch a small hole in the top of it.

A portion of the extract will remain in the beaker. This should be set aside with the funnel used to dry the first portion of the sample placed in the mouth of the beaker.

C. Record the Turbo-Vap tube number, the vendor and lot number of hexane used in the appropriate extraction logbook.

D. Place the glass evaporator tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be in the sensor and need removal. (See Turbo-Vap manual).

E. When the extract has been concentrated to ~1.0 mL, remove it from the Turbo-Vap and find the remaining extract. Reuse the original funnel and pour the remaining extract through it so that it drains into the turbovap tube. Rinse the 400 mL beaker twice with 10 -20 mLs of methylene chloride or hexane for Pest/PCBs transferring the rinse to the funnel each time. Finally rinse the funnel with ~ 10 mLs of solvent. Place a piece of aluminum foil over the tube and with a sharpie mark an "X" on the aluminum foil. Remove the tube to a metal holder. Concentrate as before.

F. When the beep sounds indicating the end of concentration, the extract will be at approximately one half mL, remove the tube from the bath. The extracts are ready to be transferred into their appropriate vials.

- **For Pest/PCB Completion:** Hold the tube and the sample vial in one hand at about a 45° angle. Use a 9" pasteur pipette to draw up the sample and transfer it to the 10 mL vial. Be careful not to spill any sample during the transfer because one drop represents 5% of the sample and is a critical loss.
- Fill a 10 mL graduated cylinder with 9.5 mL of hexane. Add 1/2 of the hexane to the tube. Draw into the pipette and rinse down the conical portion of the tube several times. Transfer this rinse to the 10 mL vial. Add the remaining hexane and repeat this rinsing process. Cover the extract with a Teflon-sealed screw cap.

- **BNA Completion:** When the volume has been reduced to 1-0.5(~0.4)mL, remove the tube from the Turbo-Vap and place in a metal holder.
- Use a 9" Pasteur pipette to transfer the extract to a 2 mL amber vial. THIS IS THE MOST CRITICAL PART OF THE ENTIRE OPERATION!!! A single drop represents about 10 percent of the total sample. Before you move the tip of the pipette from the tube to the vial, be sure that a drop will not form on the end and fall off.
- Draw ~0.20 mL of methylene chloride into a 0.50 mL syringe and add ~ half of this aliquot to the tube. Draw the methylene chloride into a pipette and rinse the sides of the tube several times. Transfer this rinse to the 2 mL vial. Add methylene chloride from the syringe and repeat the rinsing process until you have ~ 1 mL in the sample extract vial.

- Compare this volume to a 2 mL dummy vial containing 1 mL of solvent to insure that you have not exceeded 1 mL. For low level PAHs the final volume is 0.5mL. The methylene chloride rinse volume must be adjusted to achieve this final volume.

Compare the volume to a 2mL dummy vial containing 0.5 mL of solvent to insure that you have not exceeded 0.5 mL. The GC/MS operator will adjust the sample to the desired final volume and add internal standard just prior to analyses. Cover the extract with a Teflon-sealed screw cap and transfer the label to the vial.

NOTE: If the final extract is yellow or dark in color or the matrix is oily and viscous, further cleanup may be desired. Refer to SOPs 307 to 309 and 330, 331, 333 for further cleanup information. Discuss cleanup possibilities with the analyst and/or your supervisor.

The extract obtained above may now be analyzed. Refrigerate at 4°C or carry directly to the analyst. Samples must be signed into the Sample Extract refrigerator. On log provided, enter the sample numbers, your initials, and the date and time the samples were placed into the refrigerator.

Determination of % Dry Weight – Weigh 5-10 grams of the sample from the bulk jar used for dry weight analysis in a tared crucible or aluminum pan. Dry overnight at 105°C. Allow to cool in a dessicator before weighing. Calculate % dry weight as follows:

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

VIII. DOCUMENTATION OF CAPABILITY (DOC)

Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

IX. WASTE MANAGEMENT AND POLLUTION PREVENTION

Please see Waste Disposal SOP-405 for the proper 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.

X. METHOD PERFORMANCE

Refer to SOP-201 and SOP-211 for method performance.

XI. REFERENCES

1. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 3541.

DEFINITIONS

BNA - Base/ Neutral/Acid
°C - degrees centigrade
COC - chain of custody
DL - detection limit
g - grams
KD - kuderna danish
LCS - laboratory control sample
µg/L - micrograms per liter
µL - microliter
µg/ml - micrograms per milliliter
ml - milliliter
mm - millimeter
MS - matrix spike
MSD - matrix spike duplicate
PAH - polynuclear aromatic hydrocarbons
PCBs - polychlorinated biphenyls
Pest - pesticides
RL - reporting limit
SOP - standard operating procedure
TCMX - tetrachloro-m-xylene
v/v - volume to volume

Refer to SOP-431 for further definitions

LABORATORY SAMPLE RECEIVING, LOG IN AND STORAGE STANDARD OPERATING PROCEDURES

SOP NUMBER: **SOP-404**

REVISION NUMBER: **12**

APPROVED BY:

SECTION MANAGER

TECHNICAL DIRECTOR

EFFECTIVE DATE: **01/05/09**

DATE OF LAST REVIEW : **01/05/09**

LABORATORY SAMPLE RECEIVING, LOG IN AND STORAGE

This SOP lists in as much detail as possible our daily procedures for sample receiving, log in and storage of laboratory samples. Keep in mind that there may be project specific requirements that are more strict or different than our routine procedures. In these instances, the project specific requirements must be met and followed. Although a few project specific requirements are detailed in this SOP, i.e. USACE certification issues, not every situation can be addressed. If there is ever any uncertainty on what procedures must be followed, please see the Testing Coordinator immediately. If ever in doubt, always go with the more stringent requirements. This document will constantly be reviewed and revised as necessary.

SAMPLE ACCEPTANCE CRITERIA

A sample may be rejected for compliance purposes if it does not meet the following criteria. Analyses may only proceed after notification and approval to proceed from the client or from the laboratory manager.

1. Sample must be properly preserved and in the proper container for the requested analysis.
2. Sample integrity must be maintained. The container shall be intact without cracks, leaks, or broken seals.
3. Adequate sample volume must be received for the requested analysis, including volume for any requested QA/QC (MS/MSD).
4. The sample ID on the bottle label must match the sample ID listed on the chain of custody.
5. The sample container label and the chain of custody must be completed with indelible ink. The sample label must be intact and list all necessary information; to include: sample date, sample time, sampler, and sample ID/location. The chain of custody shall also indicate sample date and time, requested analyses, and all necessary client information.
6. Sample temperature must be less than 6°C or received on ice.
7. Sample must be within holding time for the requested analysis.

These issues are discussed in more detail below under the "Sample Receiving" section of this document.

I. Sample Receiving

A. Samples are received at the Empirical Laboratories on French Landing Drive in Nashville, TN.

1. The majority of samples are shipped in coolers by couriers such as Federal Express and UPS. All couriers are generally received in the Empirical Laboratories Sample Receiving (SR) area on the fifth floor. The laboratory is located close to the Federal Express (FedEx) distribution station, therefore we often pick up our coolers at the FedEx

location and transport them back directly to the laboratory. Some coolers and/or samples are delivered directly to the SR area by the sampler and/or client.

2. Some coolers and/or samples may be received directly by Empirical Laboratories Sample Receiving personnel. If samples are hand delivered by the client make sure that necessary paperwork is included and that you sign and date the chain of custody. If the *Empirical Laboratories* ***Chain of Custody [Attachment II***] is used the white and yellow copy of the chain of custody is retained and the pink copy must be given to the client.

B. When going through the required steps for Sample Receiving and Sample Log In, keep in mind that a ***Corrective Action Report (CAR) for Sample Receiving [Attachment III]*** must be completed to document any problems, discrepancies, project changes, etc. encountered during the process. This includes but is not limited to incorrect sample containers, improper preservatives [chemical and temperature], chain of custody discrepancies, sample descriptions, etc. A CAR may be completed just to keep a record of a situation, which is not actually "out of compliance."

1. Make sure that all information on the CAR is stated clearly and very detailed. Many times it is necessary to refer to these documents a year or more after they were completed. Document all correspondence including name, date, company and response.

2. The CAR forms must be numbered starting with No. 001 at the beginning of the year (e.g. 01-001). No two forms should have the same number. All CARs must be forwarded to the Project Manager and/or receiving manager for approval and distribution. THIS MUST BE DONE ASAP OF WHEN THE PROBLEM/SITUATION IS DISCOVERED.

C. Visually inspect all coolers for tampering, custody seals present and intact (if applicable) leakage, etc. If a cooler has been damaged beyond repair, unpack the samples and discard the cooler as to not reuse it. If you suspect a cooler may be damaged or is extremely dirty this cooler must not be reused. If coolers were sent by Federal Express, examine the Federal Express airbills for the number of packages in the shipment and make sure that all the packages (coolers, boxes etc.) in a group have been received. If there are any problems the Project Manager must be contacted immediately. If anything looks unusual, take the time to check it out and document the situation and findings.

D. Open each cooler in order to quickly inspect the contents and to locate the chain of custody. Sample Receiving personnel should wear the following personal protection equipment: gloves, safety glasses and a laboratory coat. All coolers received from projects with the **US Army Corps of Engineering (USACE) and AFCEE** projects should be opened under the hood in the sample storage room. Sign, and list the date and time received on the chain of custody. The time received <u>must</u> reflect the actual time the samples were received even though they may be logged into the system at a later time. Samples received on Saturday may be processed on the following Monday morning, or samples received late in the day during the week may be processed the next morning. All cooler(s) <u>must</u> be opened, examined for leakage, breakage etc., the temperature measured and the chain of custody signed and dated to

reflect the actual date and time which they were received. The samples must be delivered to the appropriate analytical department or put in cold storage as soon as possible.

1. Attach any shipping receipts, work orders, etc. to the chain of custody.

2. If a chain of custody or other paperwork is not sent, the client must be contacted and the samples temporarily placed on hold in cold storage. In some instances the log-in person may complete a chain of custody. The required information may be found on the sample containers or it may be necessary to call the client to get the missing information (i.e. sample ID, collection date and time, etc.). Note on the chain of custody that it was completed by laboratory personnel and record the name of the person with whom you spoke. All attempts to encourage our customers to complete a chain of custody or submit written information for samples must be made.

3. Project specific paperwork may be required. For all projects, a ***Cooler Receipt Form [Attachment IV]*** must be completed for each cooler received. Sample receiving personnel must begin completing this form as soon as a cooler is received and complete this form as samples go through the log in process.

E. The temperature of each cooler or set of samples must be measured as quickly as possible using a thermometer with 0.1°C increments. This thermometer must be calibrated against a NIST certified thermometer once a year and this information recorded in a bound notebook. The Certificate of Calibration for the NIST thermometer is kept on file at the QAO's desk. The thermometer must be tagged with the unique identification number of SR#1 and serial #; (Sample Receiving #1), the date calibrated and the correction factor. This information must also be recorded in a bound notebook. Only this thermometer can be used for recording the temperature of sample coolers upon receipt.

1. To measure the temperature, open the temperature control blank if supplied, point the IR thermometer at the liquid surface, wait 30 seconds for temperature to stabilize. Read the temperature to the nearest 0.1 °C. The corrected value temperature must also be recorded on the chain of custody. (This value will also be recorded into the LIMS at a later point.). All regulatory compliance samples received from North Carolina that do not meet the temperature requirement will be segregated and the client will be notified of the non-compliance. The samples will not be analyzed until we receive client notification to proceed with analyses.

2. If the temperature exceeds 6°C for any sample, the Project Manager or Sample Receiving personnel must contact the client immediately. There may be tighter temperature control limits for specific project requirements. The customer must make the decision to either continue with the analyses or resample. Make sure the client is aware that if the samples are analyzed, the following qualifier is normally included on the final report: "The shipping cooler temperature exceeded 6°C upon receipt to Empirical Laboratories. This may have an impact on the analytical results. The concentration may be considered as

estimated." Not all samples for the project will be flagged, just those samples received above 6°C.

Many times we are not able to get in touch with the client quickly and the best judgment on how to handle the samples must be made after discussion with the Testing Coordinator and/or Laboratory Director or Technical Director. The samples may still need to go through the log in process although it may be eventually determined that the samples will not be analyzed or the samples may temporarily be placed on hold and not logged in. Above all do not allow the samples to set out at room temperature for an extended period of time while waiting for a decision. **A CAR outlining the problem and all correspondence must be completed.**

The only exceptions to the 6°C rule are:

a. Water samples for all Metals, (except Chrome 6+ and mercury) that have been preserved with HNO_3 to a pH of ≤ 2. *Keep in mind that non-aqueous sample for Metals must be cooled.*

b. Samples for Fluoride, Chloride and Bromide.

c. Waste/Product samples for all parameters.

d. Samples generated in the Aquatic Toxicology laboratories and brought directly to Sample Receiving after they are collected. Sample receiving personnel should place these in cold storage as soon as possible.

e. Samples collected locally by Empirical Laboratories personnel or local customers that hand deliver their samples. In some instances these samples may not have had time to cool down, however, these samples should have been placed on ice in an attempt to cool them to the proper temperature. It should be noted if samples are "Received On Ice" (ROI).

f. Samples that are received on ice and it is evident that the client made a good faith attempt to properly cool the samples.

F. If several coolers are received at once, they must be inspected to determine the order in which the samples should be unpacked and logged in. The following priorities should be given:

1. Any analyses, which have a 24-72 hour holding, time. It is the log-in person's responsibility to notify the department manager or section group leader of such samples via e-mail and verbally. In addition to notifying the analyst of samples with short holding times, log-in personnel must log this information into a separate record book daily. It is the analysts' responsibility to review this information and initial each page at the end of each day.

2. Any sample which has almost exceeded its' holding time. (Especially watch for this with waters organic extractions, Solids and Sulfides, all of which have only 7 days). A list of parameters and holding times is posted in the log-in room.

   a. If a sample is received already out of holding time, the project manager must be contacted. The sample can be analyzed at the client's request, but it will be qualified on the final report as being analyzed out of holding time. The project manager must inform you of the client's need.

   b. If a sample is received with limited holding time remaining for any parameter it may be necessary to contact the project manager so that he/she can contact the client. If the sample has to be analyzed on a rush basis to meet the holding time a rush charge may apply. Also it may not be possible to analyze the sample within the holding time due to sample load, etc. A CAR must be completed.

3. Samples requiring rush turnaround.

   a. If sample(s) require 24-hour turnaround they will take first priority. Other rush requests also have high priority.

   b. The Project Manager and/or Section Manager must be contacted for approval concerning any unscheduled rush requests.

G. Unpack all samples from the cooler. If there are any known or suspected hazards this must be done under a hood. All coolers from USACE projects should be unpacked under a hood. It may be necessary to rinse off the outside of the containers in the sink and/or wipe them off with a paper towel.

   1. Visually inspect them for tampering and custody seals (if applicable). Sort and inventory the samples against the chain of custody by arranging them in the same order as they are listed on the chain of custody. Normally samples are assigned log numbers in the same order as they are listed on the chain of custody but for certain projects or situations it is acceptable to arrange them in a manner which will make them easiest to log in.

   2. Check for leakage as this could compromise the sample integrity. If any spillage occurred in the cooler make sure this is noted. Also list all the other samples in the cooler as cross contamination could occur. A CAR must be completed and the Project Manager and/or the customer may need to be notified in these situations. It may be necessary to resample.

H. Check the chain of custody information against the information recorded on the containers. If these do not agree, contact appropriate person (s) - Project Manager, sampler, client, etc. All problems must be documented with a CAR.

1. If major changes are made on the chain of custody received from an engineering job, then the PE should submit written confirmation of these changes or make the corrections and initial them directly on the chain of custody.

2. Any error found on the chain of custody must be marked through with one line, initialed and dated and the correction written in.

I. Note any unusual requests, methodology, hazards (known or suspected) to the Project Manager and/or Laboratory Section Manager or analysts before the samples are actually logged in. Keep notes of any problems (improper containers, preservatives, temperature, or descriptions, etc.) A CAR must be completed and the analyst or manager should be notified immediately. If ever in doubt, fill one out!

II. Sample Log In

A. After samples have been unpacked, sorted and reviewed, they are then ready to be assigned log numbers and continue through the log in process. Make sure that the parameters for the samples are clearly marked on the chain of custody. If we prepared the sample kits there should be a sample kit work order form. Contact the Project Manager if there are any questions, problems, etc.

B. Assign a work-order and sample number to each individual sample and record it on each sample container and the chain of custody.

1. All containers with the same description must have the same sample number even if they have different preservatives and require different tests. However, each different fraction (bottle type and/or preservative) should be designated with a letter (A, B, C, etc.)

2. Grab and composite samples from the same sample location must be considered as separate samples. It may be necessary to use "grab" or "composite" as part of the sample description to distinguish between the samples. Only assign different log numbers to them if the parameters are clearly marked as grab and as composite. Do not assume that VOC must be analyzed from grab samples so therefore the client must have taken a grab sample.

3. Sample numbers must begin with 001 at the beginning of each year (e.g. 0101001).

C. Check the following items and record this information on the cooler receipt form to further ensure sample integrity. A CAR must be completed if any of the following requirements are not met and it may be necessary to contact the client. We can perform the analyses in most cases and will do so with the client's approval, however the results may be qualified in some manner on the final report.

Preserving sample integrity throughout the log in procedure must be one of our section's top priorities. This includes not only ensuring that the proper chemical preservatives have been added but also that the samples are received and maintained at the proper temperature. ***When samples are unpacked they must be placed in cold storage as soon as possible even if they have not been through the entire log in procedure***. All samples for NPDES compliance monitoring from North Carolina will be stored at a temperature range of 1.0 to 4.4°C. All other NPDES samples will be stored at 4.0 ± 2.0°C. On the days we receive a large volume of samples, or are short handed, etc., we may not be able to completely log in all samples until late in the day or even the next day. Samples should not set out at room temperature if there is a delay. The samples must temporarily be placed in cold storage until you are able to complete the log in procedure. This should also be done when we take lunch breaks.

[Make sure the VOC containers are not temporarily stored in a non designated VOC only storage area.]

1. Determine if the samples were received at the proper temperature. (See section IC)

2. The sample descriptions on the bottle should match those on the chain of custody. (See section 1H)

3. Check to determine if the proper chemical preservatives were added to adjust the sample to the correct pH. All regulatory compliance samples received from North Carolina that do not meet the preservation requirement will be segregated and the client will be notified of non-compliance. The samples will not be analyzed until notification to proceed with analyses is received from the client. A list of parameters and the required chemical preservatives is posted in the log-in room. The verification of this preservation will be recorded on the Cooler Receipt Form for all projects. If Empirical Laboratories prepared and shipped out the sample containers they will have been pre-preserved unless instructed otherwise by the client. Complete traceability of the preservatives used to pre-preserve the sample containers and to preserve samples in the log-in area is required. A bound notebook must be used to trace this information and must include the following: Lot #, Type of preservative, Date Prepped, Amount and Analyst Name. This information must also be labeled on each container, re-pipetter, etc. that the preservative is stored in. Each lot of HNO3 used for Metals preservation must be tested prior to using them for preservation. These analyses are kept on file.

    a. The pH of each container (except VOA vials) which requires pH preservation must be checked. Do not open and check the pH of VOA vials in sample receiving/log-in.

    b. The pH of preserved samples is checked and confirmed using pH narrow range indicator paper. When the client request pH analysis on samples and they must be reported and measured for pH using the narrow range paper, rather then a pH meter, the accuracy of each batch of indicator paper must be calibrated to the nearest tenth versus certified pH buffer and recorded into a bound logbook in accordance with SW846 method 9041A pH Paper method.

c. When taking the pH reading, DO NOT PUT THE pH PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Pour up a small aliquot and dispose of this volume after the pH is taken. For some samples (wastes) the indicator paper may not be accurate due to interferences. The observation of the appropriate color change is a strong indication that no interferences have occurred. If it appears as if there is interference, the pH must be measured using the pH meter. [See SOP ATSD-187 pH , Electrometric.]

4. The following guidelines must be followed to check pH preservation:

a. Water samples for Cyanide analyses must be preserved to a pH of >12 with NaOH upon collection. If the pH of these samples is between 11.0-12.0 upon receipt, and the samples are at the proper temperature and not over 48 hours old it will not be necessary to complete a CAR, however the sample should be adjusted to $\geq$12.0 unless project/client specific requirements are to contact the client first.

b. Water samples for Metals analyses must be preserved to a pH of $\leq$2.0 with HNO_3 upon collection. If the pH of these samples is between 2.0-.3.0 upon receipt, and the samples are not over 48 hours old it will not be necessary to complete a CAR, however the sample should be adjusted to $\leq$2.0. unless project/client specific requirements are to contact the client first.

c. Samples requiring analyses which are preserved with H_2SO_4 (i.e., Nitrogen compounds, Total Phenolics, Oil and Grease, Total Phosphorus, etc.) can be accepted up to a pH of 2.5 without a CAR, however the sample should be adjusted $\leq$2.0 unless project/client specific requirements are to contact the client first. Samples for sulfide analysis must have a pH >9.

d. If a sample is not properly preserved, log-in personnel must either do the following:

- To meet project specific requirements, including all USACE projects, the client must be notified before preserving or adding additional preservative to the sample unless otherwise instructed. If the client instructs us to add chemical preservatives to a sample, complete traceability of the preservatives used is required (See section IIC, #3). A CAR must be completed.

- For other projects it may be acceptable to preserve the sample accordingly before the sample is placed in storage. Complete traceability of the preservatives used is required (See section IIC, #3). A CAR outlining the project and the steps taken must be completed.

- All metals samples preserved upon receipt must be held 24 hours before proceeding with analysis. These samples must be entered into the Unpreserved Metals Log and a CAR generated and the client notified to see if the lab is to proceed with analysis.

e. In some instances it may not be possible to adjust the sample to the proper pH due to matrix problems which cause excessive foaming or require an unusually large amount of acid. Do not continue to add acid if a few mL's of acid does not lower the pH. Notify the Project Manager, Metals Manager and/or analyst. They will make the decision if the sample will be diluted, not analyzed, etc. A CAR must be completed in these situations. Make sure you note on the container and in the LIMS notes that the sample is not at the proper pH as well as any useful information (i.e., foaming, strong odor, etc.).

f. A CAR may not be required for samples generated in the Aquatic Toxicology Laboratories and brought directly to Sample Receiving after they are collected but before they are preserved. Log-in personnel must preserve the samples accordingly before they are placed in storage. Complete traceability of the preservatives used is required (See section IIC, #3). A CAR outlining the project and the steps taken must be completed.

5. Check to make sure samples are in proper containers and that there is adequate volume for all the parameters requested and no leakage.

6. If VOA vials are present, each vial must be inverted and checked for head space. "Pea-sized" bubbles (i.e. bubbles not exceeding 1/4 inch or 6 mm in diameter) are acceptable and should be noted, however, a CAR is not required. Large bubbles or head space is not acceptable and a CAR must be completed. If this occurs, the client must be contacted. The samples can be analyzed with their approval, however the report will be qualified and the data may be questionable. All VOA vials will be preserved with $Na_2S_2O_3$ (0.2g) when chlorine is known to be, or suspected to be present.

7. All pesticide samples to be analyzed by method 608 will be checked by the sample receiving personnel for the correct pH range of 5.0 to 9.0. The pH of the sample(s) will be communicated via E-mail to the Section Manager and appropriate analyst.

8. All chlorinated effluent samples received for Cyanide must be checked for residual chlorine. The one liter sample container should initially contain 1 to 2g/L of Ascorbic Acid. <u>Potassium Iodide starch indicator paper will be used for detecting the presence of residual chlorine. DO NOT PUT THE TEST PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Pour up a small aliquot, neutralize, test and dispose of this volume after the sample is checked.</u> If the test paper turns blue, the sample must be treated for residual chlorine. Add Ascorbic Acid, approximately 0.6g at a time and recheck the sample until there is no residual chlorine present. If the sample required this treatment this information must be included in the LIMS notes. This must be done by log-in personnel before leaving the receiving area. It may be necessary to notify the Inorganic Manager and/or analyst.

9. Be aware of holding time requirements. (See section 1D)

D. Once sample containers have been numbered, they must be checked by another laboratory individual to ensure that the log number on the container matches the log number and sample ID on the Chain of Custody. A ***Sample Receiving Custody and Disposal Form [Attachment VII]*** must be completed each day. Samples should not leave the log-in area until this has been completed. *[see IIC; it may be necessary to temporarily store samples in cold storage until the samples can be second checked, the amount of time that the samples are at room temperature must be minimized as much as possible.]* The original is to remain in Sample Receiving until the samples are disposed of. Once the document is complete, the original will be kept on file. The following information must be logged onto this form:

1. Client and Log #s
2. Date/Time Unpacked
3. Logged In/Numbered By (Initials)
4. 2nd Checked By (Initials)
5. Date/Time Placed in Cold Storage
6. Storage Area (Walk In, VOC Cooler, Quarantined Soils, Quarantined-VOC, Other)
7. Disposed of By/Date
8. Method of Disposal

E. Notify the proper analyst if samples have been logged in for analyses which have a 24-48 hour holding time or if a 1-2 day turnaround has been requested. The log number and description on sample (s) must be second checked before it is released to the analyst. (The analyst can second check the sample, but must initial the custody form.)

III. Sample Storage

A. After samples have been correctly logged in they are then transferred to one of the following cold storage areas and arranged in numerical order by the assigned log in/LIMS sample number. ***Note that aqueous VOC samples must be segregated from all other samples.***

1. The Blue Air refrigerator in Sample Storage Room: All aqueous VOC's must be stored in this refrigerator. Storage blanks consisting of organic free water from the laboratory may be required for specific projects. These will be analyzed for VOCs only. ***Storage blanks are required for all DOD projects.***

2. Walk In Refrigerator in Sample Storage Room: All aqueous samples for all analyses must be stored in this refrigerator.

3. Soil Walk-In Refrigerator in back BC Laboratory: All quarantined and non-quarantined soil samples for all analyses must be stored in this refrigerator.

B. Quarantined soils are those quarantined by the US Department of Agriculture. These soil samples must be segregated from other soil samples during storage. A separate disposal log must be maintained for these soils including the location, date and quantity of the soil received and processed. Soil residues from quarantined samples must be treated according to regulations after testing (see Sample Disposal SOP). Quarantined soils are defined as:

1. Soil taken from much of the southeastern US and parts of New York and Maryland at a depth of three feet or less. *Soils from three feet or more are not regulated provided they are stored separately.* A map of the regulated areas in the United States entitled ***Soil Movement Regulations [Attachment VIII]*** is posted in the log-in room.

2. All soils taken from foreign sources, US. territories and Hawaii .

NOTE: All soils are treated as quarantined soils and are disposed of in accordance with USDA regulations. Above for information purposes only.

C. All samples must be stored in one of the three refrigerators detailed above with the following exceptions:

1. Matrices that may be adversely affected by the cold temperature. (e.g. surfactant samples, multi-phase samples)

2. Highly contaminated waste or product type samples that could jeopardize the integrity of other samples in the walk in cooler. Often these can be stored at room temperature. If these require refrigeration see the Project Manager for other options.

D. The temperature of each sample refrigerator must be monitored and recorded each day by Wet Chem personnel by the following method. A Mercury thermometer or digital min/max thermometer with 1° increments must be used. Each thermometer must be calibrated against a NIST certified thermometer once a year**(digital thermometers quarterly)** and this information recorded in a bound notebook. The Certificate of Calibration for the NIST thermometer is kept on file at the QAO's desk. The thermometers must be tagged with a unique identification, the date calibrated and the correction factor.

The tolerance range for all refrigerators is 1 to 6°C. This range and the range using the corrected reading must be posted on the outside of each cooler. If the temperature exceeds this range, corrective action measures must be put in place immediately. A CAR must be completed specifically noting the date and time the problem was discovered. The Project Manager, Laboratory Director and Technical Director will be notified in order to assess the situation. It may be necessary to put a service call in to the refrigeration repair service.

E. All personnel removing samples from any refrigerator must sign them in and out. This is done by completing the ***Sample Custody Form [Attachment IX]*** which is attached to the door of each refrigerator. These completed forms are kept on file [see section III, #4F]

F. The water walk in refrigerator in the sample room is the largest refrigerator and stores a large majority of the samples. A back up compressor is hooked into the system and scheduled to automatically come on if the main compressor fails. There is a digital min/max thermometer, which monitors the temperature 7 days a week. This thermometer will be calibrated quarterly against the NIST thermometer.

G. As stated above the temperatures for all refrigerators that samples are stored are checked each day Monday-Friday and monitored seven days a week with min/max thermometers. Pay close attention to these readings and watch for signs of possible problems.

H. A temperature maintenance record book is kept for each refrigerator.

I. Samples must be held for a minimum of 30 days after the final report unless specified otherwise. For USACE projects, samples must be held for a minimum of 60 days after the final report unless otherwise specified. See SOP ATSD 405 entitled Analytical Laboratory Waste Disposal SOP for guidance on disposal of samples.

IV. Laboratory Information Management System (LIMS)

A. Log the sample information into the LIMS for each sample. Every attempt should be made to get every sample logged into the LIMS by the end of the day. All information entered should be clearly stated and recorded on the COC provided. After opening the main menu of the LIMS, select the 'Work Orders' tab from the 'Sample Control' drop down menu. Now click on the 'Add' button to create a new Work Order. You will see the following:

1. ***Client*:**

   Select the client I.D. by clicking on the pull-down and choosing from the client list. This list is in alphabetical order. If the desired client is not on the list, a new client must be created by the project manager or I.T. director.

2. ***Projects*:**

   Click on 'Projects' and choose the project I.D. The projects will be client specific. After the project is chosen the "project information" areas should fill in. The 'Project Name,' 'Project Number,' 'TAT,' 'Client Project Manager,' 'Lab Project Manager,' and 'Comments' information should also appear. If there are no applicable project choices, a project must be created by the project manager or I.T. director. There are two types of projects:

a. Internal -- Empirical Laboratories projects;

b. External -- direct laboratory clients.

3. ***Comments:***

This area is to be used to note any information from the project manager for all work orders of this project. It can also be used to list any work order specific notes; this includes but is not limited to information concerning rush turnaround, deliverables or other QC requirements, analyte concentrations, safety issues, quarantined soils, CAR #s, preservation or matrix problems, etc.

4. ***Received By:***

Enter the name of the person who received the samples.

5. ***Logged In By****:*

Enter the name of the person who logged in the samples.

6. ***Received:***

Enter the date and time received separated by a space and using military time.
Example: *08/02/2008 08:30*

7. ***Project/Package Date Due:***

After the date and time received have been entered, the date due for both of these fields will be calculated. If this information is not correct or needs to be amended later, check with the project manager before doing so.

8. ***Shipping Containers:***

Click on the 'Coolers' button and enter the temperature and condition upon receipt. If more than one cooler was received, each cooler must be assigned a different name. For example, if these came in by dedicated courier, enter the last four numbers of the Tracking Number as the name. After all of a cooler's information has been entered, click the 'Save' button. If more than one cooler was received, click the 'Add' button and repeat the process above, then click 'Done' after all the coolers' info has been saved.

9. ***COC Number:***

If an identifiable COC number is listed, record that ID here.

10. ***Shipped By**:*

    Enter the courier used to deliver the samples. If the samples were picked up by a lab employee or dropped of by the client/representative, enter 'Hand-Delivered.'

After these items have been completed, click 'Save,' then the 'Samples' button to continue. To begin entering information for a sample, click the 'Add' button on the bottom of the Samples screen.

11. ***Sample Name:***

    a. Only abbreviate if description is too long for the spaces allotted in the LIMS. This information should come directly from the chain of custody. The sample ID entered into the LIMS will be the sample ID on the final report.

    b. If no sample ID is provided, or is indistinguishable from other samples listed, contact the project manager to ascertain distinction in the samples. Include date as part of the description if this is the only way to differentiate the samples.

    c. When logging in trip blanks that do not have an ID assigned by the client, list them as "Trip Blank # _____". This information should be on the containers. A log book must be kept in the sample kit room which lists all trip blanks and the date they were filled. This will ensure consistency with the descriptions for trip blanks. Make sure you record the trip blank on the chain of custody if it is not listed.

12. ***Collection Date:***

    Enter the date and time the sample was collected. You must use military time and separate by a space. Often the time collected is not given. Although this is a sampling requirement, this information may not be crucial unless a parameter with a short holding time or a data deliverables package is required. All attempts should be made to get all our clients to supply this information.

13. ***Lab/Report Matrix:***

    Click on pull down and select matrix. Many times it is difficult to discern the matrix if it is not specified on the COC, and log-in personnel must use their best judgment with regard to analytes/methods requested. Keep in mind that the detection limits and units on the LIMS reports are linked to the matrix. In some cases it may be necessary to ask the Section Managers about the matrix selection.

14. ***Sample Type:***

This is used to differentiate between special types of samples (i.e. Field Duplicates, Equipment Blanks, Trip Blanks, etc.). If there is no definite way to determine that a sample should be classified as something else, then "SAMP-Client Sample" will be selected as the sample type. Do not list a sample as anything other than a Client Sample unless noted on the COC of are instructed by the client to do so.

15. ***Container:***

Click on the drop down list and select the appropriate bottle type. If multiple bottles are received for the same sample, then move down to the next line and select all other containers as required. Repeat this process until all containers for the sample are listed. As each container is entered, an individual number is assigned to it by the LIMS system. This number is also listed on the container labels that are printed from the LIMS, and is placed on the corresponding bottle for container tracking purposes.

16. ***Fraction:***

When more than one container is sent with the same preservation/analysis (i.e. Volatiles & Extractables), the sample containers can be grouped together using a fraction code. This is simply an alphabetical notation added to each container to allow analysts to verify the preservative and proper sample volume to use when performing analysis. **This should be in no way used to record the sample volume used during analysis or reporting**, as these codes are entered by log-in personnel on an as needed basis, and do not provide an individual container designation by which to track any given container.

17. ***pH*** (Container Preservative)***:***

Use this to document the pH check information taken during sample unpacking. If no preservative was used, then nothing is required in this field.

18. ***Comments:***

Enter any information that is applicable at the sample level.

19. ***Field Analysis:***

Click on field analysis tab and enter field information when provided.

20. ***Work Analyses:***

Select all parameters requested for the sample from this list.

a. If the required test code is not listed, and the sample matrix is not a contributing factor, click the Work Analyses tab to open the All Analyses list. When selecting from this expanded list, be careful to select the proper method as all methods available for the current matrix will be selectable.

b. If any analyses are selected from the All Analyses list, the Project Manager in charge should be notified so that the correctness of methods and pricing can be checked and updated as needed.

c. All preparation codes for analytes are entered and stored by the system independently of the test codes selected, except in the cases of Dry Weight analysis, and TCLP/SPLP preparation (tumbling). In the case of the TCLP/SPLP prep codes, these are entered alongside the other required analyses automatically by the LIMS when a TCLP/SPLP analyte is selected. As for Dry Weight, it is required for all solids testing except in the cases of TCLP/SPLP analysis, Explosives only analysis, and/or any pure product/non-soil based sample when specified by the client.

21. ***Analyses Comments:***

These comments should be used for any notes that only apply to that particular test code.

22. ***RTAT:***

If the Rush Turn-Around Time for this sample is known at the time of log-in, this information should be updated here.

23. ***Save:***

Once all applicable information is entered for a sample, click the save button. At this time the LIMS applies the Laboratory Sample ID to the sample. This is a four part ID code composed of the following:

a. A 2-digit numeral of the year. Example (**08**11248-06)

b. A 2-digit numeral of the month. Example (08**11**248-06)

c. A 3-digit numeral of the work order number. This number reset to 001 at the beginning of each month. Example (0811**248**-06)

d. A 2-digit numeral of the sample number separated by a dash. Example (0811248**-06**). This number is different for each sample in a work order, and a single work order cannot contain more than 99 samples. If more sample numbers are needed, a new work order number will have to be assigned to the applicable set of sample.

24. ***Add/Edit/Copy:***

    Use these selections to add more samples to the work order, or to change existing information prior to label printing.

Once all the tests have been selected and all samples have been added in the work order, a work order summary and all container labels are printed. Labels are checked for accuracy against the containers while being labeled. At this point log-in of this group of samples is complete.

B. After log-in of a work order is complete, the COC can then be scanned into the system, attached to the work order on the Work Order screen, and the work order can be updated to Available status so as to be seen by the analysts.

V. Daily Follow Up for Sample Receiving/Log In

A. Wipe out the inside of coolers and return all Empirical Laboratories coolers to the sample kit room. Discard any coolers that are cracked, broken or filthy.

B. Complete the short holding times log book as required. This must be done as early in the day as possible.

C. If any samples were received for RUSH turnaround, then a ***RUSH SHEET [Attachment XII]*** must be completed and distributed to all laboratory personnel via e-mail. If ever in doubt of which analysts should be notified, pass them out to everyone. Always give copies to the Laboratory Director, Administrative Assistant and Section Managers. It may be necessary to send out two RUSH sheets per day (one around mid-day and the other at the end of the day).

D. Complete any required CARs.

E. At the end of the day organize all paperwork received and generated for the day. The following should be given to the Project Manager (section supervisor):

    1. The original chains of custody and yellow original or copy of each. The CRF will accompany the CoC for the project.

    2. Any information (letters, regulatory limits, etc.) from a client which was received with any samples.

    3. All CARs.

4. LIMS sample receiving logs.

5. Copies of any RUSH sheets which have been distributed

6. Sample Receiving Custody and Disposal Form.

7. Cooler receipt form.

G. All the above information from the day will be reviewed as soon as possible.

1. All LIMS logs must be 2nd checked by a different person than the person entering the information into the LIMS. Each set of logs must be initialed dated by the person 2nd checking. These will be kept on file at the Project Manager desk.

2. If any corrections or changes are required, all laboratory personnel will be notified by distributing a ***Sample Log Change Form [Attachment XIII]*** through email distribution. A ***Sample Log Change Form*** by the project manager will also be sent out if a client adds or deletes any parameters, changes sample IDs, etc.

H. The Testing Coordinator will distribute the following after they have been through the 2nd QA check:

1. Copies of the LIMS receiving reports to necessary laboratory personnel.

2. Original (white copy) chains of custody are given to the Project Manager. These will be sent with the final report to the client.

3. Finalized/approved CARs must be sent to the:

   a. Organic Manager

   b. Inorganic Manager

   c. Laboratory Manager

   d. Laboratory Director {optional}

   e. Quality Assurance Officer

   f. Administrative Assistant

   g. Client {optional}

4. Copies of any project/sample specific information to the Section Manager and analysts.

I. Information will be filed as follows:

1. Chains of custody:

   a. Original (white copy) is returned to the customer with the final report along with the CRF.

   b. Yellow copy originals or photo copies will be filed by client and kept in the Sample Receiving Room.

   c. Photo copies will be kept in a notebook in numerical order in the Sample Receiving Room.

   d. Pink copies should be retained by the sampler.

2. CARs

   a. CARs can be found at V:\LAB\log-in\login(year)\logcar(year).

3. Sample Change Forms and RUSH Sheets

   a. Sample Change Forms are distributed by email.

   b. RUSH Sheets are found at V:\LAB\login\Rushsheets

4. At the end of each year, files for that year are boxed and archived. Make sure files are labeled properly and place them in banker's boxes. Complete a storage box file form with as much detailed information as possible. The Laboratory Administrative Assistant will label and number the boxes and incorporate the storage boxes into the laboratory file archive system. Boxes containing files from Sample Receiving are kept on site for 1-2 years and then may be moved to off site storage upon release from the Project Manager.

VI. Miscellaneous

A. All projects which require deliverables or other QC requirements should be listed in the notes section of the LIMS.

B. If samples are received from a new client or a new job number that is not in the LIMS, a new client code must be set up. This information should be on the chain of custody or it may be necessary to contact the customer if the information is incomplete.

C. Samples from the Aquatic Toxicity Laboratory (ATL) are logged into the LIMS for billing and long-term tracking purposes. The receiving information and proper assignment of test's are reviewed by the ATL Manager. The samples are then logged in by ATL personnel.

D. A flow chart outlining sample receiving and the flow of data, reporting and invoicing is attached as ***Attachment XIV.***

E. A ***Telephone Conversation Log [Attachment XV]*** may be required to document information and may be attached to or used as a CAR.

F. All log books used in the Sample Receiving and Sample Storage Areas are numbered. The following log books are presently maintained. All log books must be "Z"ed out. The Testing Coordinator will review the log books each week to check for completeness.

Log Book ID	**Log Book Description**
SRLB #3	pH Paper Calibration
SRLB #4	Tracking of VOC Trip Blanks, Organic Free Water and Chemical Preservation

Attachments to SOP 404

II	Chain of Custody Record
III	Corrective Action Report for Sample Receiving/Log In
IV	Cooler Receipt Form
V	List of Short Holding Time (Immediate-72 hrs.) Parameters
VII	Sample Receiving Custody and Disposal Form
VIII	Map of Quarantined Soil Areas in the US.
IX	Laboratory Sample Custody Form for Walk In Refrigerator
X	Container Codes for the LIMS
XI	Routine NPDES Clients
XII	RUSH Sheet
XIII	Sample Log Change Form (Green Sheet)
XIV	Flow Chart, Laboratory Sample Tracking System

[Attachments I and VI were removed during the editing process and not added to the SOP.]

ANALYTICAL LABORATORY WASTE DISPOSAL

SOP NUMBER: **SOP-405**

REVISION NUMBER: **4**

APPROVED BY:

TECHNICAL DIRECTOR

QUALITY ASSURANCE OFFICER

EFFECTIVE DATE: **09/26/03**

DATE OF LAST REVIEW: **04/23/08**

Analytical Laboratory Waste Disposal
Standard Operating Procedure

I. SCOPE AND APPLICATION:

Empirical Laboratories, LLC laboratory waste includes excess client sample waste and waste that are generated while performing an array of analytical services, some of which are hazardous. These wastes must be disposed of in a manner that is safe, cost efficient and in accordance with hazardous waste regulations.

A. Wastes can be broken down into the following categories:

1. Unused portions of actual samples received from outside clients.

   a. Unused aliquots of completed water samples.

   b. Unused aliquots of completed non-aqueous samples.

2. Soils from quarantined areas

3. All other soils, sediments, building debris, wipes etc.

4. Hazardous waste generated within the laboratory as part of numerous analytical procedures.

II. SUMMARY OF PROCEDURES:

A. There are four options for disposing of unused sample portions:

1. Return completed samples and any generated waste from these samples to the client.

2. Throw the sample away after confirming that it is non-hazardous.

3. Disposal through a waste vendor in either a sealed drum or lab pack. This is normally done twice a year.

4. Treat the sample to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

B. There are two options for disposing of laboratory generated waste:

1. Disposal through a waste vendor in either a sealed drum or lab pack. This is normally done twice a year. The waste must be stored properly until the waste is transported off site.

For example: Solvent waste must be stored in the vented flammable cabinet.

2. Treat the waste to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

III. EQUIPMENT/APPARATUS:

A. Proper safety equipment in good working condition. This includes gloves, lab coat and safety glasses/goggles (voluntary use of cartridge respirator allowed see area manager or QAO).

B. USDOT approved drums for storing and shipping hazardous waste.

C. Fume hood vented outside the building.

D. Flammable storage cabinet which is vented to the outside

IV. PROCEDURE

Waste disposal is done under the management and coordination of the Sample Receiving Manager, Section Managers and the Health and Safety Officer.

A. Disposal of completed aqueous samples:

Completed samples are kept in cold storage for approximately three weeks after the final report has been mailed. Engineering support projects involving CLP work, litigation cases etc. may be saved for longer than three weeks at the request of the project manager.

No samples should be disposed of without approval from the responsible area manager or analyst. **At this point the area manager and/or analyst will communicate information about samples deemed as hazardous**.

1. The majority of the water samples (ground, surface and drinking) are non-hazardous and are disposed of by pouring them down the sink.

a. This must be done under the hooded area located near the walk in cooler next to the extraction lab. Make sure that the sash is closed far enough to produce sufficient ventilation. The tap water should be turned on to supply copious wash for sample disposal.

b. Proper safety equipment **must** be used including safety glasses (face shield if necessary), lab coat and gloves.

c. **Be alert to potential problems: for example, separate Cyanide waste from acid waste. Neutralize acid waste that will be poured down the acid drain and don't mix waste/samples thought to contain Cyanide with samples that are acidified. Also, look for things such as phase separation, odd color, odor etc. Check with the area manager or Health and Safety Officer before disposing of any questionable samples.**

d. Tap water must be running during the time samples are poured out and for approximately 10 minutes after so sufficient flushing and dilution takes place.

e. All containers must be rinsed out, all identifying markings defaced or removed, and thrown into the trash.

f. All samples disposed of in this manner must be documented in the bound disposal log.

2. If water samples are hazardous (known or suspected), one of the following steps must be taken.

a. Samples may be returned to the client. If you plan to ship the unused portion back to the client check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations. **If the samples are not returned to the client they must be stored properly until picked up by a waste vender.**

b. Treat the sample to make it non-hazardous. One example of this is if the sample is highly corrosive, the pH may be adjusted.

c. Store the sample properly until either a sealed drum or lab pack is sent out.

d. All samples disposed of in this manner must be documented in the bound disposal log.

B. Disposal of completed non-aqueous samples:

The majority of non-aqueous samples are soils or sediments, although there may also be building debris, wipes, oils**,** and occasionally product type samples.

1. If samples are non-hazardous they must have all identifying markings defaced or removed, and thrown into the trash. On specific projects we may also opt to return the unused portions to the client even if they are non-hazardous.

2. If non-aqueous samples are hazardous (known or suspected), one of the following steps must be taken.

a. Samples may be returned to the client. If you plan to ship the unused portion back to the client check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations**. If the samples are not returned to the client they must be stored properly until picked up by a waste vender.**

b. Store the sample properly until a lab pack is sent out.

3. Soil samples taken at a depth of three feet or less from areas, which have been quarantined by the US Department of Agriculture (USDA), must first be treated at the laboratory to prevent the spread of any plant pests. The USDA has detailed proper treatment procedures of which we use the following:

a. The sample is heated to 180°C(356°F)in a vented oven for one hour.
b. After the heating the samples are placed close to a hood to cool and are marked as being ready for disposal.

4. Once the samples have undergone treatment they can then be disposed of by one of the procedures for non-aqueous samples**. All samples disposed of in this**

manner must be documented in the bound disposal logbook with the following information:

a. Client
b. Sample #s
c. Date(s) treated
d. Treatment method used

C. Disposal of laboratory generated waste:

Generated waste is stored in satellite areas until a waste pick up occurs. These satellite areas must be maintained properly.

1. Waste handling and disposal within each laboratory section:

Each laboratory analyst and section manager is responsible to assure that **handling** operations within their area are being followed according to the laboratory requirement.

a. General Chemistry/Inorganic

Each analyst performining specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the inorganic manager or the safety officer.

- Concentrated acid waste, **(>2% by volume) and** dilute mercury waste (mercury, chemical oxygen demand, total kjeldahl nitrogen and chloride analyses waste) are poured into the Acid Satellite Waste drum in the general chemistry laboratory. **Document the type and amount of waste in the acid waste logbook, then initial and date the entry**.
- Dilute acid waste **(<2% by volume or less)** are neutralized and poured down an acid drain with copious amounts of tap water.
- **All other non-hazardous sample waste, reagents and standards are poured down the drain with copious amounts of tap water.**

b. Metals

Each analyst performining specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the inorganic manager or the safety officer.

- Concentrated acid waste, aqueous sample waste digestates and old unused calibration standards **(>2% by volume)** are poured into the Acid Satellite Waste drum in the general chemistry laboratory.
- Non-aqueous sample digestate wastes are decanted off the soil/solid samples into the Acid Satellite Waste drum in the General Chemistry Laboratory. **Rinse the soil/solid with tap water several times and discard the first rinsate into the Acid Satellite Waste drum and the sequential rinsates decant down an acid drain with copious amounts of tap-water.**
- **Throw the soil/solids in the trash once the acid has been rinsed free**.
- **Cr6 digestates as with all concentrated metal/acid waste are poured into the Acid Satellite Waste drum.**

c. Organic Extraction Laboratory Area

Each analyst performining specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the organic manager or the safety officer.

- Concentrated acid waste is discarded into the Acid Satellite Waste drum in the General Chemistry Laboratory.
- Non-chlorinated solvent waste (Acetone, Ether, Hexane, Methanol ….etc…) pour into the Non-Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory.
- Chlorinated solvent waste (Methylene Chloride, Chloroform, chlorinated standard and spike waste) pour into the Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory.

****Note: Laboratory generated solvent waste is transferred to the appropriate Satellite Solvent Waste Drum (chlorinated or non-chlorinated) weekly or as deemed necessary. Disposal of solvent waste is done under the direction of the organic laboratory manager. These drums are located in the chemical reagent storage room and only authorized laboratory staff are allowed to add waste solvent to these drums. The date of addition to the**

drum , type and quantity of solvent is entered into the '*Organic Solvent Waste Logbook*' located on the shelf next to the drums.

- **Aqueous sample waste from extracted samples (once the extraction solvent has been removed) is poured down the drain and flush with copious amount of tap water.**
- Non-aqueous sample waste and sodium sulfate waste is dumped into a waste container under an extraction laboratory hood and left overnight or until the solvent is evaporated and then the waste is discarded into the trash.

d. Gas Chromatography (GC)/High Performance Liquid Chromatography (HPLC) Laboratory

- Autosampler vials are discarded into the appropriately labeled box located under the table in the GC/HPLC Laboratory.

PCB Box – all samples/standards
Pesticide Box – all samples/standards
Herbicide Box – all samples/standards
8330 Box – all samples/standards
Methylene Chloride Box- all samples/standards that contain methylene chloride (Diesel Range Organics, DRO)

- Sample and spike extract vials are separated according to the contents in the vial.

Acid cleaned extracts are combined into a separatory funnel and the acid layer separated from the solvent. The acid portion is discarded into the Acid Satellite Waste drum in the general chemistry laboratory. The solvent waste is discarded into the appropriate solvent waste bottle (chlorinated/non-chlorinated waste) located in the hood in the organic extraction laboratory.

After the mercury has been recovered from the mercury cleaned samples, the extracts are discarded into the proper waste solvent bottle (chlorinated/non-chlorinated waste) in the hood in the organic extraction laboratory. The mercury waste is consolidated and discarded into the mercury waste container located in the vented hood in the chemical storage room. **Mercury used for removing sulfur from samples has been discontinued in our laboratory**.

Unused stock and working standards are discarded into the chlorinated solvent waste bottle located in the organic extraction laboratory. The empty vials are rinsed

several (3) times with solvent and the solvent rinsate poured into the solvent waste and the vials with labels removed are discarded into the glassware waste container.

e. Gas Chromatography/Mass Spectrometry

- Volatile sample, standard and reagent waste

Waste from the instrument - Aqueous sample waste is collected in waste bottles via waste lines from the instrument. The bottles are emptied into buckets and poured down the drain(pH is < 2% by volume). A small amount of methanol used to clean glassware is also dumped into the bucket and poured down the drain. While disposing of sample waste always run the cold tap water 10-15 minutes. Non-aqueous waste from sample analyses is retained and dispose of in the samey as the unused sample. Unused sample is held for sample disposal by the sample receiving area, see A and B listed above.

Standards - **Unused stock and working standards** are discarded into the chlorinated solvent waste bottle located in the organic extraction laboratory. The empty vials are rinsed several (3) times with solvent and the solvent rinsate poured into the solvent waste and the vials with labels removed are discarded into the glassware waste container.

In conjunction with section managers, the sample receiving area disposes of solid sample waste and unused aqueous and solid samples, see procedures A and B listed above.

- Semivolatile sample and standard waste disposal

Methylene chloride waste solvent and standard waste in vials are poured into the chlorinated waste bottle in the hood in the organic extraction laboratory. The empty vials are rinsed with solvent and the solvent poured into the waste solvent bottle. The vials with labels removed are discarded into the glassware waste disposal container.

Auto sampler vials are collected in buckets and stored under the hood in the organic extraction laboratory. **Periodically the vials are consolidated in lab packs for disposal by a licensed waste disposal company**.

f. Bioassay Laboratory

- Aqueous sample waste and a small amount of methanol are poured down the drain with copious amounts of tap water. Larger amounts of methanol used for glassware cleaning are collected in beakers and evaporated at room temperature.

- Hazardous or product samples are returned to the client.

D. Consolidation of satellite waste for contractor disposal:

In conjunction with the Safety Officer, the sample revieving supervisor is responsible to coordinate waste disposal operations with outside waste disposal contractors.

1. Solvent waste from the areas discussed above is periodically consolidated into two drums located in the Chemical Storage room (c. *Organic Extraction Laboratory Area*, *** Note**). A drum designated either chlorinated or non-chlorinated solvent waste is available to receive the appropriate solvent waste. When the drums become full (fluid surface six inches below the top of the drum), an authorized hazardous waste contractor will be scheduled to remove them to proper waste disposal.

2. The Acid Satellite Waste drum is also disposed through the authorized hazardous waste contractor once the drum is full to the level of six inches below the top of the drum.

3. Consolidated autosampler and standard vials are periodically Lab-Packed in drums and disposed through the authorized hazardous waste contractor.

4. The Laboratory Health and Safety Officer will administer the Waste Disposal Program and maintain current information to track quantities of waste generated and stored on-site.

It is the continuous objective of our laboratory to find ways to decrease the amount of waste generated.

STANDARD OPERATING PROCEDURE (SOP) FOR LABORATORY SAMPLE STORAGE, SECURE AREAS AND SAMPLE CUSTODY

SOP NUMBER: **SOP-410**

REVISION NUMBER: **6**

APPROVED BY:

SECTION MANAGER

TECHNICAL DIRECTOR

EFFECTIVE DATE: **09/08/08**

DATE OF LAST REVIEW: **09/08/08**

STANDARD OPERATING PROCEDURE (SOP) FOR LABORATORY SAMPLE STORAGE, SECURE AREAS AND SAMPLE CUSTODY

Empirical Laboratories, LLC is located on the fifth floor of a building which is locked and monitored by a guard after normal business hours. No unauthorized personnel are permitted within the facility without a proper escort and a visitor's badge. During non business hours, all doors to the building are locked and the elevators are security coded (i.e. a code must be entered in order to get the elevator to open on the fifth floor.) All stairway doors are locked and only Empirical Laboratories, LLC personnel have a key to the fifth floor stairway door. The doors to the lab in the hallway have a key code. There is a buzzer at the door to Login to allow entry for sample and supply deliveries.

The majority of samples are shipped in coolers by couriers such as Federal Express and UPS. All couriers are generally received in the Shipping/Sample Receiving (SR) area on the fifth floor. The laboratory is located close to Federal Express (FedEx) distribution station, therefore we pick up our coolers at the FedEx location on Saturdays and transport them directly to the laboratory. Some coolers and/or samples are delivered directly to the SR area by the sampler and/or client. The SR personnel must not leave any packages/cooler without authorized receipt from laboratory personnel. Samples must be accompanied by some type of chain of custody record. Sample receiving personnel sign, and list the date and time received on the chain of custody. The time received must reflect the actual time or validation date and time of receipt for the samples although they may be placed in cold storage and logged into the system at a later time. The method of delivery is listed on the cooler receipt form(CRF). The tracking # (if available) is attached to the chain of custody.

Once sample containers have been assigned a laboratory ID number, they must be checked by another laboratory individual to ensure that the log number on the container matches the log number and sample ID on the Chain of Custody. A Sample Receiving Custody and Disposal Form (attached) must be completed each day. Samples should not leave the log-in area until this has been completed. A copy of this form must be given to the Testing Coordinator at the end of the day. The original is to remain in Sample Receiving until the samples are disposed. Once the document is complete, the original will be kept on file. The following information must be logged onto this form:

- Client and Log #s
- Date/Time Unpacked
- Logged In/Numbered By (Initials)
- 2nd Checked By (Initials)
- Date/Time Placed in Cold Storage
- Storage Area (Walk In, Blue Air-VOCs, Quarantined Soils, Quarantined-VOC, Other)
- Disposed of By/Date
- Method of Disposal

Original samples are stored in following areas of the laboratory.

1. Blue Air Refrigerator in Sample Storage Room: All water VOCs must be stored in the refrigerator.

2. Walk In Refrigerator in Sample Storage Room: All waters for all analyses except VOCs must be stored in this refrigerator.

4. Soil Walkin Refrigerator for all soils.

All soils are treated as quarantined.

All samples must be stored in one of the three refrigerators detailed above with the following exceptions:

1. Matrices that may be adversely affected by the cold temperature. (e.g. surfactant samples, multi-phase samples)

2. Highly contaminated waste or product type samples which could jeopardize the integrity of other samples in the walk in cooler. Often these can be stored at room temperature. If these require refrigeration see the Testing Coordinator for other options.

Any person removing samples from the storage areas listed above, must sign them out on a laboratory custody sheet (attached). The individual performing the processing becomes responsible for the samples at this point. The samples are maintained in the

secure possession of the individual processing the samples. When the processing is completed, the samples are returned and signed back into the appropriate storage area. It must be noted if the entire sample volume was used and that the container was discarded.

Sample extracts and digestates are stored in the following areas:

1. All metals digestates are stored in the metals instrument laboratory. The transfer from the digestion analysts to the ICAP analysts is documented in the metals digestion log book.

2. Non - ZHE TCLP extracts are returned to the refrigerator in which the original samples are stored. For ZHE samples, the extract is returned to the refrigerator in which the original VOC sample containers are stored.

3. Extracts from medium level VOC analyses are also stored in the Soill Walk – in or VOC sample freezer in the VOC Lab.

4. All Organic extracts are stored in a Beverage Air side by side refrigerator in the organic extraction laboratory.

The generation of all sample extracts/digests and their movement through the laboratory will also be tracked on a laboratory custody sheet or in a log book. The individual performing the processing becomes responsible for the samples at this point. The samples are maintained in the secure possession of the individual processing the samples. When the processing is completed, the extracts are returned and signed back into the appropriate storage area. The metals digestates are not removed from the metals instrument laboratory.

After the analytical results have been reported, the original samples, sample extracts, and digestates will remain in secure storage until they are disposed of in accordance with the Waste Disposal Standard Operating Procedure. Samples will be held for a minimum of 30 days after the final report unless specified otherwise. Sample extracts and digestates are held for a minimum of 60 days after the final report unless project specific requirements state otherwise. See SOP No. 405 entitled Laboratory Waste Disposal SOP for guidance on disposal of samples.

The following personnel as of September 08, 2008 have access to all sample storage areas:

Chandra Arthur	Herbie Johnson
Ashley Bester	Dahae Kim
Roger Burr	Dustin Lynch
Tanisha Custer	Marcia McGinnity
Rick Davis	Badeen Mekael
Barbara Dawson	AntonioMontiero
Betty DeVille	Ashley Morris
Amanda Fei	Gino Moore
Kendra Gentry	E. J. Overby
Jason Goodman	Brenton Powers
Sonya Gordon	Brian Richard
Gwen Hallquist	Franklin Rivers
Andrew Holder	William Schwab
Jade Holliman	Christy Thompson
John Hughes	Renee Vogel
Karu Huka	Randy Ward

In the event that an employee is terminated, the supervisor is responsible for collecting the employee's keys.

For additional information see SOP No. 404 entitled Laboratory Sample Receiving, Log-In and Storage.

QC Limits

Explosives

Compound	Water Limits	Soil Limits
1,3,5-Trinitrobenzene	55-125	70-120
1,3-Dinitrobenzene	65-145	85-130
2,4,6-Trinitrotoluene	60-130	45-140
2,4-Dinitrotoluene	60-140	75-130
2,6-Dinitrotoluene	60-130	65-130
2-Amino-4,6-dinitrotoluene	60-135	60-125
2-Nitrotoluene	35-135	65-130
3-Nitrotoluene	45-130	65-130
4-Amino-2,6-dinitrotoluene	55-140	30-175
4-Nitrotoluene	55-130	65-130
HMX	55-140	75-140
Nitrobenzene	35-130	20-145
RDX	60-145	80-120
Tetryl	10-170	55-150
Nitroglycerin	60-120	60-120

PAHs SIM (AFCEE 4.0)

Compound	Water Limits	Soil Limits
Naphthalene	39-125	29-106
2-Methylnaphthalene	36-121	30-111
1-Methylnaphthalene	35-131	30-111
Acenaphthene	41-132	28-110
Acenaphthylene	43-140	23-126
Fluorene	40-140	27-116
Phenanthrene	46-144	32-127
Anthracene	50-139	28-136
Fluoranthene	47-158	30-142
Pyrene	39-158	28-130
Benzo(a)anthracene	58-141	31-146
Chrysene	51-155	39-134
Benzo(b)fluoranthene	42-156	30-139
Benzo(k)fluoranthene	49-165	42-129
Benzo(a)pyrene	31-142	28-128
Indeno[1,2,3-cd]pyrene	20-167	17-164
Dibenzo(a,h)anthracene	28-153	30-138
Benzo(g,h,i)perylene	12-171	21-149

Metals

Compound	Water Limits	Soil Limits
Aluminium	80-120	80-120
Arsenic	80-120	80-120
Boron	80-120	80-120
Barium	80-120	80-120
Beryllium	80-120	80-120
Calcium	80-120	80-120
Cadmium	80-120	80-120
Cobalt	80-120	80-120
Chromium	80-120	80-120
Copper	80-120	80-120
Iron	80-120	80-120
Potassium	80-120	80-120
Magnesium	80-120	80-120
Manganese	80-120	80-120
Molybdenum	80-120	80-120
Silver	80-120	80-120
Sodium	80-120	80-120
Nickel	80-120	80-120
Lead	80-120	80-120
Selenium	80-120	80-120
Antimony	80-120	80-120
Tin	80-120	80-120
Titanium	80-120	80-120
Thallium	80-120	80-120
Vanadium	80-120	80-120
Zinc	80-120	80-120
Mercury	80-120	80-120

Nitrate/Nitrite 300

Compound	Water Limits
Nitrate	90-110
Nitrite	90-110

TCLP Organics

Compound	Water Limits
Benzene	75-125
Carbon tetrachloride	75-135
Chlorobenzene	75-120
Chloroform	75-125
1,2-Dichloroethane	70-135
1,1-Dichloroethene	70-125
2-Butanone	65-145
Tetrachloroethene	70-125
Trichloroethene	80-125
Vinyl chloride	65-140

o-Cresol	40-110
m-Cresol	40-110
p-Cresol	40-110
1,4-Dichlorobenzene	25-110
2,4-Dinitrotoluene	65-110
Hexachlorobenzene	65-110
Hexachloro-1,3-butadiene	15-110
Hexachloroethane	15-110
Nitrobenzene	40-110
Pentachlorophenol	50-145
Pyridine	10-110
2,4,5-Trichlorophenol	60-105
2,4,6-Trichlorophenol	55-105
Chlordane	45-119
Endrin	45-140
Heptachlor	30-115
Heptachlor epoxide	45-140
Lindane	30-150
Methoxychlor	40-140
Toxaphene	41-126
2,4-D	25-120
2,4,5-TP (silvex)	20-110

ATTACHMENT 1-E

MRP SITE VISIT SUPPORT DOCUMENTATION

Navy/Tetra Tech Site Visit of May 2007

BRUNSWICK NAVAL AIR STATION, MAINE
AGENDA FOR KICKOFF MEETING

WEDNESDAY MAY 30, 2007

TIME	SUBJECT	PRESENTER
8:30 to 8:45	Meet and Greet	Linda Klink, TtNUS
8:45 to 10:00	Site Overviews • Kickoff Meeting Objectives - Gather Site-Specific Information - Develop MEC Data Quality Objectives (DQOs)	Ralph Basinski, TtNUS
10:00 to 10:20	Health and Safety Briefing	Ralph Brooks, TtNUS
10:20 to 10:30	Break	
10:30 to 11:30	Travel to Quarry for Site Walk • Observe Historical Operations Areas • Observe Site Features and Transport Pathways • Photographs of Significant Features/Sampling Locations • Geophysical Technology Demonstration Location • Access Issues (clearing & grubbing, drill rig, etc.)	Ralph Brooks, TtNUS Dale Mosher, BNAS
11:30 to 12:15	Travel to Former Munitions Bunker West Area • Observe Historical Operations Areas • Observe Site Features and Transport Pathways • Photographs of Significant Features/Sampling Locations • Access Issues (clearing & grubbing, drill rig, etc.)	Ralph Brooks, TtNUS Dale Mosher, BNAS
12:15 to 1:15	Lunch	
1:15 to 2:45	Travel to Site 12 for Site Walk • Observe Historical Operations Areas • Observe Site Features and Transport Pathways • Photographs of Significant Features/Sampling Locations • Geophysical Technology Demonstration Location • Access Issues (clearing & grubbing, drill rig, etc.)	Ralph Brooks, TtNUS Dale Mosher, BNAS
2:45 to 3:45	Travel to Machine Gun Boresight Range • Observe Historical Operations Areas • Observe Site Features and Transport Pathways • Photographs of Significant Features/Sampling Locations • Access Issues (clearing & grubbing, drill rig, etc.)	Ralph Brooks, TtNUS Dale Mosher, BNAS
3:45 to 4:30	Travel to Skeet Range for Site Walk • Observe Historical Operations Areas • Observe Site Features and Transport Pathways • Photographs of Significant Features/Sampling Locations • Access Issues (clearing & grubbing, drill rig, etc.)	Ralph Brooks, TtNUS Dale Mosher, BNAS
4:30 to 4:40	Break	
4:40 to 5:00	MEC Work Plan Scoping • Overview of DQO Process • TRIAD approach - Systematic Planning - Dynamic Work Plans - Real Time Analysis • Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP) - What is it? - What does it require? - How should it be used for Brunswick MRP sites?	Ralph Basinski, TtNUS

THURSDAY MAY 31, 2007

TIME	SUBJECT	PRESENTER
8:30 to 9:15	Travel to Topsham Skeet Range for Site Walk • Observe Historical Operations Areas • Observe Site Features and Transport Pathways • Photographs of Significant Features/Sampling Locations • Access Issues (clearing & grubbing, drill rig, etc.)	Ralph Brooks, TtNUS Dale Mosher, BNAS
9:15 to 9:25	Break	
9:25 to 10:00	Machine Gun Boresight Range & Skeet Range & Topsham Skeet Range DQOs • MEC DQOs (avoidance) • Review of Baseline Information from PA • Problem Definition	Ralph Basinski, TtNUS Ralph Brooks, TtNUS
10:00 to 11:00	Former Munitions Bunker West DQOs • MEC DQOs • Review of Baseline Information from PA • Problem Definition • Investigation Goals • Definition of Inputs to Achieve Goals • Define Study Boundaries • Review Sampling and Analysis • Develop Acceptance Criteria	Ralph Basinski, TtNUS Ralph Brooks, TtNUS
11:00 to 11:10	Break	
11:10 to 12:15	Quarry DQOs • MEC DQOs • Review of Baseline Information from PA • Problem Definition • Investigation Goals • Definition of Inputs to Achieve Goals • Define Study Boundaries • Review Sampling and Analysis • Develop Acceptance Criteria	Ralph Basinski, TtNUS Ralph Brooks, TtNUS
12:15 to 1:15	Lunch	
1:15 to 2:45	Site 12 DQOs • MEC DQOs • Review of Baseline Information from PA • Problem Definition • Investigation Goals • Definition of Inputs to Achieve Goals • Define Study Boundaries • Review Sampling and Analysis • Develop Acceptance Criteria	Ralph Basinski, TtNUS Ralph Brooks, TtNUS
2:45 to 3:00	Break	
3:00 to 3:30	Site 12 Explosives Safety Submittal	Ralph Brooks, TtNUS
3:30 to 4:30	Site 12 and Quarry Geophysics Technology Demonstration • Test Items • Locations • Acceptance Criteria	Ralph Brooks, TtNUS Joe Parish, TtEMI
4:30 to 4:50	Review of Action Items and Schedules	Linda Klink, TtNUS

Worksheet.

Repeat for October 23-24 Stakeholders mtg.

SITE-SPECIFIC TRAINING DOCUMENTATION

My signature below indicates that I am aware of the potential hazardous nature of p activities at NAS Brunswick located in Brunswick, Maine, and that I have received a brief hazards and selected control measures presented below:

- Names of designated personnel and alternates responsible for site safety and he
- Safety, health, and other hazards present on site (UXO/MEC/Natural Hazards)
- Use of personal protective equipment and control measures to minimize potential hazards
- Contents of the Abbreviated Health and Safety Plan
- Emergency action procedures

I further state that I have been given the opportunity to ask questions, that my questions have been answered to my satisfaction, and that I agree to abide by the procedures and policies addressed in this plan.

Name (Printed)	Signature	Date
Linda Klink - Tetra Tech NASB Coordinator	Linda Klink	5/30/07
Dale C. Mosher IRCOOP		5/30/07
Ralph Basinski TENUS		5/30/07
LT. James Sisk NASB ESO		5/30/07
Joe Parish Tetra Tech EM		5/30/07
David Vellar NASB		5.31.07
Mike Dale EPA		10/23/07
Carolyn A. Lepage BACSE Pollution	Carolyn A. Lepage	10/23/07
Dawn C. Kincaid BRAC PMO NE	Dawn C. Kincaid	10/23/07
Amy VANDERCOOK NAVFAC		10/23/07
G. Chris Evans (MEDEP)	G. Chris Evans	10/23/07
Claudia Sait (MEDEP)	Claudia Sait	10/23/07
Carol Stein G-F for EPA	Carol Stein	10/23/07
David F. McTigue G-F for EPA		10/23/07
Orlando J. Monaco Navy RPM	Orlando J. Monaco	10/23/07
Carol G. Warren Brunswick LRA		10/23/07
James Coffman TENUS	James D Coffman	10/23/07
Charles Race TENUS		10/23/07
Richard Roedner NASB ENV.	Richard J. Roedner	10/24/07

Instructor(s): Ralph Basinski TENUS

LRA - Local Redevelopment Authority

Stakeholder Site Visit of October 2007

MRP SITES MEETING
BRUNSWICK NAVAL AIR STATION, MAINE
10/23/07

NAME	AFFILIATION	email
Charles Race	Tetra Tech	Charles.Race@tetratech.com
Jim Coffman	Tetra Tech	Jim.Coffman@tetratech.com
LONNIE MONACO	NAVY	orlando.monaco@navy.mil
Dave McTique	Gannett Fleming (for EPA)	dmctique@gfnet.com
Carol Stein	Gannett Fleming (for EPA)	cstein@gfnet.com
Claudia Sait	MEDEP	claudia.b.sait@maine.gov
Chris Evans	MEDEP	gordon.c.evans@maine.gov
Amy VanDercook	NAVFAC	amy.vandercook@navy.mil
Dawn Kincaid	BRAC PMO NE	dawn.kincaid@navy.mil
Mike Daly	EPA	daly.mike@epa.gov
Dale Mosher	NASB	dale.mosher@navy.mil
Ralph Brooks	TetraTech	ralph.brooks@tetratech.com
Carolyn Lepage	BACSE Tech Advisor	calepage@adelphia.com
Carol Warren	Brunswick LRA	carol@wacubu.com
Rich Roedner	Topsham LRA	rroedner@topshammaine.com
Lisa Joy	NASB Env	lisa.joy@navy.mil
Linda Klink	Tetra Tech	linda.klink@tetratech.com

AGENDA FOR MRP SITES MEETING AND SITE WALKS
BRUNSWICK NAVAL AIR STATION, MAINE
OCTOBER 2007

LOCATION: PARKWOOD INN CONFERENCE ROOM, BRUNSWICK, MAINE

TUESDAY OCTOBER 23, 2007

TIME	SUBJECT	PRESENTER
8:15 to 8:30	Meet and Greet/Review Agenda	Linda Klink, TtNUS
8:30 to 8:40	Kickoff Meeting Objectives - Meet UFP-QAPP requirements - SI versus RI requirements - Gather Site-Specific Information - Discuss MEC Data Quality Objectives (DQOs) and revisit MC DQOs	Ralph Basinski, TtNUS
8:40 to 8:55	Geophysical Surveying and Test Plots	Jim Coffman, TtNUS
8:55 to 9:15	Health and Safety Briefing	Ralph Brooks, TtNUS
9:15 to 9:30	Machine Gun Boresight Range Discussion (MC only)	Linda Klink/Ralph Brooks, TtNUS
9:30 to 9:45	NASB Skeet Range Discussion (MC only)	Linda Klink/Ralph Brooks, TtNUS
9:45 to 10:00	Break and Prepare for Site Walks	
10:00 to 11:00	Travel to Machine Gun Boresight Range for Site Walk (MC only)	Ralph Brooks, TtNUS Dale Mosher, NASB
11:00 to 12:00	Travel to NASB Skeet Range for Site Walk (MC only)	Ralph Brooks, TtNUS Dale Mosher, NASB
12:00 to 1:00	Lunch	
1:00 to 1:20	Site 12 Discussion (MC and MEC)	Linda Klink/Ralph Brooks/Jim Coffman, TtNUS
1:20 to 3:00	Travel to Site 12 for Site Walk (MC and MEC)	Ralph Brooks, TtNUS Dale Mosher, NASB NASB EOD
3:00 to 3:15	Break	
3:15 to 4:30	Team Input/Discussions: • Machine Gun Boresight Range • NASB Skeet Range • Site 12	All Team
4:30 to 4:40	Review Action Items/Decisions	Ralph Basinski, TtNUS
4:40 to 4:45	Review Agenda for following day	Linda Klink, TtNUS

WEDNESDAY OCTOBER 24, 2007

TIME	SUBJECT	PRESENTER
8:10 to 8:20	Meet and Greet/Review Agenda	Linda Klink, TtNUS
8:20 to 8:40	Former Munitions Bunker West Discussion (MC and MEC)	Linda Klink/Ralph Brooks/Jim Coffman, TtNUS
8:40 to 9:00	Quarry Discussion (MC and MEC)	Linda Klink/Ralph Brooks/Jim Coffman, TtNUS
9:00 to 9:15	Topsham Skeet Range Discussion (MC only)	Linda Klink/Ralph Brooks TtNUS
9:15 to 9:20	Health and Safety Reminders	Ralph Brooks, TtNUS
9:20 to 9:30	Break and Prepare for Site Walks	
9:30 to 10:45	Travel to Former Munitions Bunker West for Site Walk (MC and MEC)	Ralph Brooks, TtNUS Dale Mosher, NASB
10:45 to 12:00	Travel to Quarry for Site Walk (MC and MEC)	Ralph Brooks, TtNUS Dale Mosher, NASB
12:00 to 1:00	Lunch	
1:00 to 2:15	Travel to Topsham Skeet Range for Site Walk (MC only)	Ralph Brooks, TtNUS Dale Mosher, NASB
2:15 to 3:00	Break	
3:00 to 4:15	Team Input/Discussions: • Former Munitions Bunker West • Quarry • Topsham Skeet Range	All Team
[illegible] to 4:25	Review Action Items/Decisions	Ralph Basinski, TtNUS
4:25 to 4:45	Wrap-up/Feedback	Ralph Basinski, TtNUS

Worksheet.

Repeat for October 23-24 Stakeholders mtg.

SITE-SPECIFIC TRAINING DOCUMENTATION

My signature below indicates that I am aware of the potential hazardous nature of p activities at NAS Brunswick located in Brunswick, Maine, and that I have received a briefi hazards and selected control measures presented below:

- Names of designated personnel and alternates responsible for site safety and he
- Safety, health, and other hazards present on site (UXO/MEC/Natural Hazards)
- Use of personal protective equipment and control measures to minimize potential hazards
- Contents of the Abbreviated Health and Safety Plan
- Emergency action procedures

I further state that I have been given the opportunity to ask questions, that my questions have been answered to my satisfaction, and that I agree to abide by the procedures and policies addressed in this plan.

Name (Printed)	Signature	Date
Linda Klink - Tetra Tech NASB Coordinator	Linda Klink	5/30/07
Dale C. Mosher IRCOOP		5/30/07
Ralph Basinski TENUS	Ralph Basinski	5/30/07
LT James Scott NASB ESO		5/30/07
Joe Parish TetraTech EM		5/30/07
Dawn Vallar NASB		5.31.07
Mike Daly EPA		10/23/07
Carolyn A. Lepage BACSE Redevelopment	Carolyn A. Lepage	10/23/07
Dawn C. Kincaid BRAC PMO NE	Dawn C. Kincaid	10/23/07
Amy VANDERCOOK NAVFAC		10/23/07
G. Chris Evans (MEDEP)	G. Chris Evans	10/23/07
Claudia Sait (MEDEP)	Claudia Sait	10/23/07
Carol Stein G-F for EPA	Carol Stein	10/23/07
David F. McTigue G-F for EPA		10/23/07
ORLANDO J. MONACO Navy RPM	Orlando J. Monaco	10/23/07
Carol G. Warren Brunswick LRA		10/23/07
James Coffman TENUS	James D. Coffman	10/23/07
Charles Race TENUS		10/23/07
Richard Roedner NASB ENV.	Richard J. Roedner	10/24/07

Instructor(s): Ralph Brooks TENUS

LRA - Local Redevelopment Authority

APPENDIX B

MACHINE GUN BORESIGHT RANGE XRF RESULTS

Klink, Linda

From: Daly.Mike@epamail.epa.gov
Sent: Tuesday, July 01, 2008 1:21 PM
To: paul.burgio@navy.mil; todd.bober@navy.mil; Klink, Linda; claudia.b.sait@maine.gov; gordon.c.evans@maine.gov
Cc: Keefe.Jerry@epamail.epa.gov
Subject: NAS Brunswick: Revised EPA data from EPA XRF Sampling @ Boresight Range

Attachments: Machine Gun Boresight Range XRF Pb Results.1 May 08.xls; 08050003metmsrev.pdf; Brunswick_Soil_Sampling_05012008.pdf

Machine Gun Boresight Range XR.. | 08050003metmsrev.pdf (442 KB) | Brunswick_Soil_Sampling_050120...

Hi Folks,

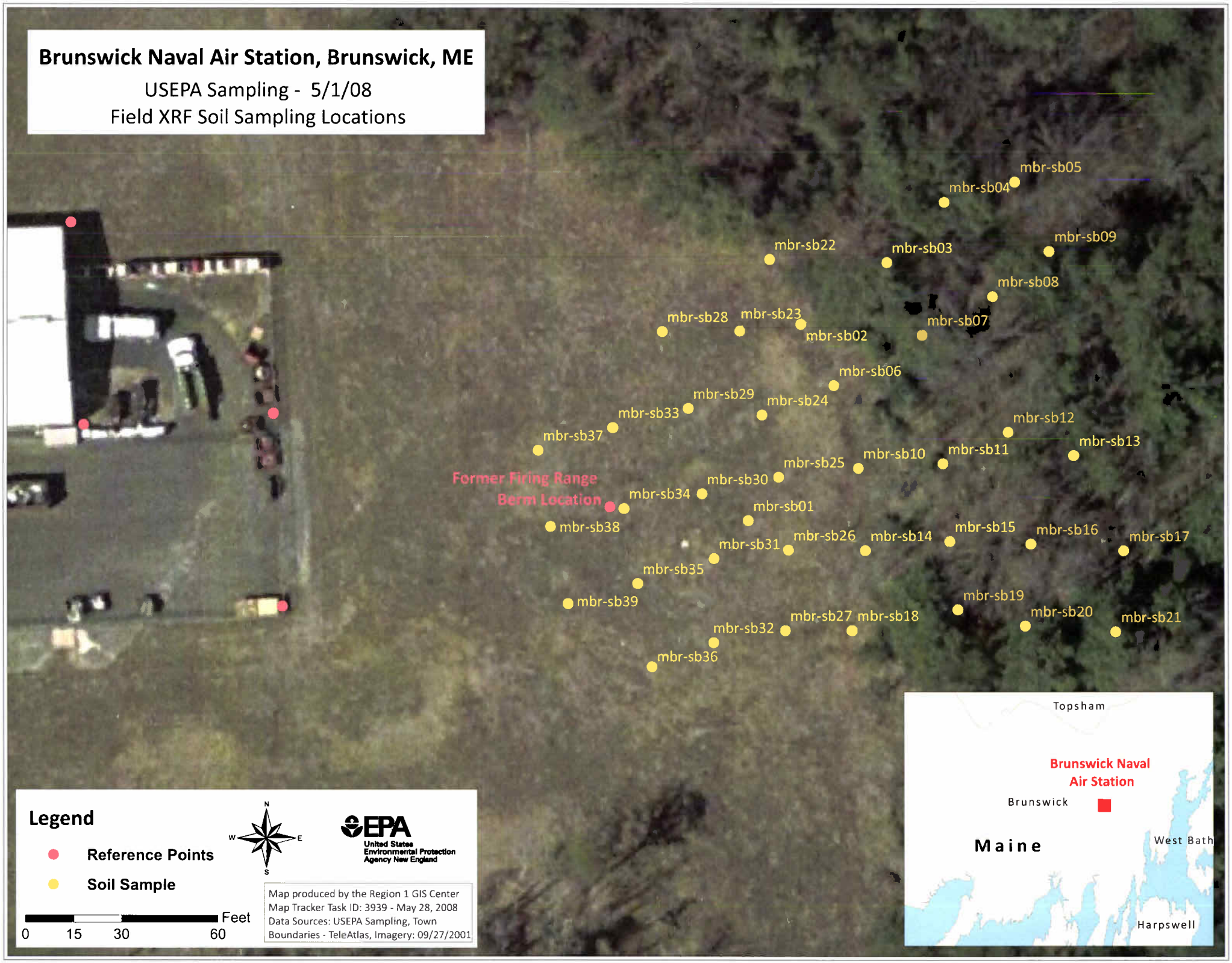
I'm forwarding to you a more complete set of data from EPA's XRF sampling effort at the Boresight Range SI site on 1 May. I asked our field and fixed lab folks to go back and provide me data for other metals that were analyzed besides lead. This data give a more complete picture of metals concentrations for the area that was sampled. I assume we will all delve back into MMRP SI MC (gotta love these acronyms) issues in the near future as a BCT.

Have a Happy 4th!

Mike

(See attached file: Machine Gun Boresight Range XRF Pb Results.1 May 08.xls)(See attached file: 08050003metmsrev.pdf)(See attached file: Brunswick_Soil_Sampling_05012008.pdf)

USEPA X-Ray Fluorescence Pb Soil Sampling Results - Former Machine Gun Boresight Range NAS Brunswick - May 1, 2008						
Soil Sample	Pb Concentration (ppm)	Zn Concentration (ppm)	Cu Concentration (ppm)	Ni Concentration (ppm)	Longitude	Latitude
MBR-SB-01	1640	245	204	430	-69.927505	43.890079
MBR-SB-01 (6-12")	226	226	62	<53		
MBR-SB-01 (12-16")	610	125	<74	<130		
MBR-SB-02	17	<26	<43	<51	-69.927430	43.890245
MBR-SB-02 DUP	21	<27	<43	<52		
MBR-SB-03	86	24	45	<39	-69.927326	43.890294
MBR-SB-04	76	<22	58	<40	-69.927254	43.890343
MBR-SB-04 DUP	82	<23	<37	<42		
MBR-SB-05	77	<21	40	<36	-69.927169	43.890357
MBR-SB-06	17	<24	<39	<46	-69.927395	43.890191
MBR-SB-07	61	<24	<39	<45	-69.927289	43.890230
MBR-SB-08	83	<18	<31	<37	-69.927203	43.890260
MBR-SB-09	43	<23	53	<45	-69.927133	43.890296
MBR-SB-10	25	<27	<43	<51	-69.927372	43.890119
MBR-SB-10 (6-12")	19	<27	<44	<52		
MBR-SB-10 (12-18")	<14	<29	<46	<54		
MBR-SB-10 (18-24")	<16	<30	<48	<60		
MBR-SB-11	26	30	<43	<50	-69.927272	43.890119
MBR-SB-12	49	<21	<33	<39	-69.927193	43.890143
MBR-SB-13	48	27	33	<34	-69.927117	43.890120
MBR-SB-14	98	28	<43	<51	-69.927369	43.890048
MBR-SB-15	42	<30	<50	<58	-69.927269	43.890052
MBR-SB-16	79	<23	43	<44	-69.927173	43.890046
MBR-SB-17	34	<25	<40	<46	-69.927065	43.890036
MBR-SB-18	28	<30	<49	<61	-69.927390	43.889980
MBR-SB-18 DUP	37	<27	<43	<56		
MBR-SB-19	25	<22	<35	<42	-69.927264	43.889993
MBR-SB-20	33	<20	<32	<39	-69.927185	43.889976
MBR-SB-20 DUP	28	<22	<35	<41		
MBR-SB-21	76	<17	33	<33	-69.927080	43.889967
MBR-SB-22	401	109	<100	<210	-69.927463	43.890302
MBR-SB-22 (6-12")	112	<35	<57	<85		
MBR-SB-22 (12-18")	44	<28	<44	<55		
MBR-SB-22 (18-24")	88	<26	<42	<52		
MBR-SB-23	26	<27	<43	<53	-69.927503	43.890242
MBR-SB-24	23	38	<39	<48	-69.927482	43.890169
MBR-SB-25	38	<27	<45	<54	-69.927466	43.890115
MBR-SB-26	94	26	<41	<50	-69.927459	43.890052
MBR-SB-27	30	<27	<44	<50	-69.927468	43.889983
MBR-SB-28	24	24	<40	<47	-69.927595	43.890245
MBR-SB-29	<13	<26	<42	<50	-69.927569	43.890178
MBR-SB-30	226	56	<58	<74	-69.927558	43.890104
MBR-SB-30 DUP	226	94	<58	<72		
MBR-SB-30 (6-12")	52	<26	<42	<51		
MBR-SB-30 (12-18")	46	<27	<44	<50		
MBR-SB-30 (18-24")	36	<22	<38	<44		
MBR-SB-31	30	26	<39	<46	-69.927548	43.890048
MBR-SB-32	71	<29	<48	<55	-69.927554	43.889976
MBR-SB-33	57	51	<54	<61	-69.927659	43.890165
MBR-SB-34	76	<28	<45	<54	-69.927651	43.890095
MBR-SB-35	160	<33	84	<64	-69.927640	43.890030
MBR-SB-36	68	34	<53	<60	-69.927629	43.889958
MBR-SB-37	208	<29	<49	<59	-69.927750	43.890149
MBR-SB-37 DUP	199	<37	67	<68		
MBR-SB-38	42	31	54	<53	-69.927740	43.890083
MBR-SB-39	96	<30	<51	<63	-69.927724	43.890016


Brunswick Naval Air Station, Brunswick, ME
USEPA Sampling - 5/1/08
Field XRF Soil Sampling Locations
mbr-sb05
mbr-sb04
mbr-sb09
mbr-sb22
mbr-sb03
mbr-sb08
mbr-sb28
mbr-sb23
mbr-sb07
mbr-sb02
mbr-sb06
mbr-sb29
mbr-sb24
mbr-sb33
mbr-sb12
mbr-sb37
mbr-sb13
mbr-sb10
mbr-sb11
mbr-sb25
Former Firing Range
Berm Location
mbr-sb30
mbr-sb34
mbr-sb01
mbr-sb38
mbr-sb15
mbr-sb16
mbr-sb17
mbr-sb26
mbr-sb14
mbr-sb31
mbr-sb35
mbr-sb39
mbr-sb19
mbr-sb20
mbr-sb21
mbr-sb27
mbr-sb18
mbr-sb32
mbr-sb36
Legend
Reference Points
Soil Sample
N
W
E
S
EPA
United States Environmental Protection Agency New England
Feet
0
15
30
60
Map produced by the Region 1 GIS Center
Map Tracker Task ID: 3939 - May 28, 2008
Data Sources: USEPA Sampling, Town Boundaries - TeleAtlas, Imagery: 09/27/2001
Topsham
Brunswick Naval Air Station
Brunswick
Maine
West Bath
Harpswell

EPA
Region 1, New England

United States Environmental Protection Agency
Office of Environmental Measurement & Evaluation
11 Technology Drive
North Chelmsford, MA 01863-2431

Laboratory Report

June 26, 2008

Michael Daly - HBT
US EPA New England, Region 1
One Congress Street
Boston, MA 02114 - 2023

Project Number: 08050003
Project: Brunswick Naval Air Station - Brunswick, ME
Analysis: Metals in Soil Medium Level by ICP
EPA Chemist: Michael Dowling
MD 6/26/08

Analytical Procedure:

All samples were received and logged in by the laboratory according to the USEPA New England Laboratory SOP for Sample Log-in.

Samples were analyzed following the EPA Region I SOP, EIASOP-INGICP7.

Sample preparation was done following the EPA Region I SOP, INGMETALSPREP6.SOP.

Samples were analyzed by inductively coupled plasma - atomic emission spectrometry using pneumatic nebulization. Preparation and analysis SOP's are based on "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Revision 2, Final Update III, Methods 3050B and 6010B," respectively.

Date Samples Received by the Laboratory: 5/2/08

Results relate only to the items tested or to the samples as received by the Laboratory. This analytical report shall not be reproduced except in full, without written approval of the laboratory.

Report may contain multiple sections and each section will be numbered independently.

If you have any questions please call me at 617-918-8340

Sincerely,

Daniel N. Boudreau 6/30/08
Daniel N. Boudreau
Chemistry Team Leader

Oualifiers:

RL	Reporting limit
ND	Not Detected above reporting limit
NA	Not Applicable
NC	Not calculated since analyte concentration is ND
J1	Estimated value due to MS recovery outside accceptance criteria
J2	Estimated value due to LFB result outside acceptance criteria
J3	Estimated value due to RPD result outside acceptance criteria
J4	Estimated value due to LCS result outside acceptance criteria
B	Analyte is associated with the lab blank or trip blank contamination. Values are qualified when the observed concentration of the contamination in the sample extract is less than 10 times the concentration in the blank.
R	No recovery was calculated since the analyte concentration is greater than four times the spike level.

Comments:

The samples were prepared and analyzed by ESAT contractors.

Sample results are in mg/Kg, dry weight.

Only lead results were originally reported. The results for additional metals are included on this report as per Mike Daly's request.

US ENVIRONMENTAL PROTECTION AGENCY
NEW ENGLAND LABORATORY

Brunswick Naval Air Station - Brunswick, ME

Metals in Soil Medium Level by ICP

Client Sample ID:	MBR-SB01	Lab Sample ID:	AA82438
Date of Collection:	5/1/2008	Matrix	Soil
Date of Digestion:	5/13/2008	Final Volume:	50 mL
Date of Analysis:	05/16/2008	Digestate Dilution:	1
Volume Digested:	N/A	pH:	N/A

CAS Number	Parameter	Concentration mg/Kg	RL mg/Kg	Qualifier
7429-90-5	Aluminum	**5800**	19	
7440-36-0	Antimony	ND	9.6	
7440-38-2	Arsenic	ND	19	
7440-39-3	Barium	**26**	2.9	
7440-41-7	Beryllium	ND	0.96	
7440-43-9	Cadmium	**4.9**	2.9	
7440-70-2	Calcium	**2600**	19	
7440-47-3	Chromium	**320**	2.9	
7440-48-4	Cobalt	**3.3**	2.9	
7440-50-8	Copper	**14**	2.9	
7439-89-6	Iron	**17000**	9.6	
7439-92-1	Lead	**1000**	9.6	
7439-95-4	Magnesium	**1800**	19	
7439-96-5	Manganese	**170**	1.9	
7440-02-0	Nickel	ND	5.8	
7782-49-2	Selenium	ND	19	
7440-22-4	Silver	ND	2.9	J2
7440-28-0	Thallium	ND	19	
7440-62-2	Vanadium	**14**	2.9	
7440-66-6	Zinc	**52**	2.9	

Comments:

US ENVIRONMENTAL PROTECTION AGENCY
NEW ENGLAND LABORATORY

Brunswick Naval Air Station - Brunswick, ME

Metals in Soil Medium Level by ICP

Client Sample ID:	MBR-SB12	Lab Sample ID:	AA82439
Date of Collection:	5/1/2008	Matrix	Soil
Date of Digestion:	5/13/2008	Final Volume:	50 mL
Date of Analysis:	05/16/2008	Digestate Dilution:	1
Volume Digested:	N/A	pH:	N/A

CAS Number	Parameter	Concentration mg/Kg	RL mg/Kg	Qualifier
7429-90-5	Aluminum	**5700**	19	
7440-36-0	Antimony	ND	9.7	
7440-38-2	Arsenic	ND	19	
7440-39-3	Barium	**30**	2.9	
7440-41-7	Beryllium	ND	0.97	
7440-43-9	Cadmium	ND	2.9	
7440-70-2	Calcium	**340**	19	
7440-47-3	Chromium	**8.3**	2.9	
7440-48-4	Cobalt	ND	2.9	
7440-50-8	Copper	**9.3**	2.9	
7439-89-6	Iron	**8300**	9.7	
7439-92-1	Lead	**57**	9.7	
7439-95-4	Magnesium	**740**	19	
7439-96-5	Manganese	**64**	1.9	
7440-02-0	Nickel	ND	5.8	
7782-49-2	Selenium	ND	19	
7440-22-4	Silver	ND	2.9	J2
7440-28-0	Thallium	ND	19	
7440-62-2	Vanadium	**22**	2.9	
7440-66-6	Zinc	**20**	2.9	

Comments:

US ENVIRONMENTAL PROTECTION AGENCY
NEW ENGLAND LABORATORY

Brunswick Naval Air Station - Brunswick, ME

Metals in Soil Medium Level by ICP

Client Sample ID:	MBR-SB22	Lab Sample ID:	AA82440
Date of Collection:	5/1/2008	Matrix	Soil
Date of Digestion:	5/13/2008	Final Volume:	50 mL
Date of Analysis:	05/16/2008	Digestate Dilution:	1
Volume Digested:	N/A	pH:	N/A

CAS Number	Parameter	Concentration mg/Kg	RL mg/Kg	Qualifier
7429-90-5	Aluminum	**6400**	19	
7440-36-0	Antimony	ND	9.5	J1
7440-38-2	Arsenic	ND	19	
7440-39-3	Barium	**55**	2.8	
7440-41-7	Beryllium	ND	0.95	
7440-43-9	Cadmium	**22**	2.8	
7440-70-2	Calcium	**1900**	19	
7440-47-3	Chromium	**170**	2.8	
7440-48-4	Cobalt	**3.5**	2.8	
7440-50-8	Copper	**11**	2.8	
7439-89-6	Iron	**14000**	9.5	
7439-92-1	Lead	**490**	9.5	
7439-95-4	Magnesium	**1700**	19	
7439-96-5	Manganese	**160**	1.9	
7440-02-0	Nickel	ND	5.7	
7782-49-2	Selenium	ND	19	
7440-22-4	Silver	ND	2.8	J2
7440-28-0	Thallium	ND	19	
7440-62-2	Vanadium	**14**	2.8	
7440-66-6	Zinc	**46**	2.8	

Comments:

US ENVIRONMENTAL PROTECTION AGENCY
NEW ENGLAND LABORATORY

Brunswick Naval Air Station - Brunswick, ME

Metals in Soil Medium Level by ICP

Client Sample ID:	MBR-SB35	Lab Sample ID:	AA82441
Date of Collection:	5/1/2008	Matrix	Soil
Date of Digestion:	5/13/2008	Final Volume:	50 mL
Date of Analysis:	05/16/2008	Digestate Dilution:	1
Volume Digested:	N/A	pH:	N/A

CAS Number	Parameter	Concentration mg/Kg	RL mg/Kg	Qualifier
7429-90-5	Aluminum	**8200**	19	
7440-36-0	Antimony	ND	9.6	
7440-38-2	Arsenic	ND	19	
7440-39-3	Barium	**33**	2.9	
7440-41-7	Beryllium	ND	0.96	
7440-43-9	Cadmium	ND	2.9	
7440-70-2	Calcium	**1200**	19	
7440-47-3	Chromium	**17**	2.9	
7440-48-4	Cobalt	**3.7**	2.9	
7440-50-8	Copper	**53**	2.9	
7439-89-6	Iron	**8600**	9.6	
7439-92-1	Lead	**160**	9.6	
7439-95-4	Magnesium	**1300**	19	
7439-96-5	Manganese	**110**	1.9	
7440-02-0	Nickel	**7.1**	5.8	
7782-49-2	Selenium	ND	19	
7440-22-4	Silver	ND	2.9	J2
7440-28-0	Thallium	ND	19	
7440-62-2	Vanadium	**14**	2.9	
7440-66-6	Zinc	**26**	2.9	

Comments:

US ENVIRONMENTAL PROTECTION AGENCY
NEW ENGLAND LABORATORY

Brunswick Naval Air Station - Brunswick, ME

Metals in Soil Medium Level by ICP

Client Sample ID:	MBR-SB37	Lab Sample ID:	AA82442
Date of Collection:	5/1/2008	Matrix	Soil
Date of Digestion:	5/13/2008	Final Volume:	50 mL
Date of Analysis:	05/16/2008	Digestate Dilution:	1
Volume Digested:	N/A	pH:	N/A

CAS Number	Parameter	Concentration mg/Kg	RL mg/Kg	Qualifier
7429-90-5	Aluminum	**8100**	20	
7440-36-0	Antimony	ND	9.8	
7440-38-2	Arsenic	ND	20	
7440-39-3	Barium	**28**	2.9	
7440-41-7	Beryllium	ND	0.98	
7440-43-9	Cadmium	ND	2.9	
7440-70-2	Calcium	**1100**	20	
7440-47-3	Chromium	**12**	2.9	
7440-48-4	Cobalt	**4.3**	2.9	
7440-50-8	Copper	**30**	2.9	
7439-89-6	Iron	**9300**	9.8	
7439-92-1	Lead	**230**	9.8	
7439-95-4	Magnesium	**1700**	20	
7439-96-5	Manganese	**160**	2.0	
7440-02-0	Nickel	**8.3**	5.9	
7782-49-2	Selenium	ND	20	
7440-22-4	Silver	ND	2.9	J2
7440-28-0	Thallium	ND	20	
7440-62-2	Vanadium	**16**	2.9	
7440-66-6	Zinc	**28**	2.9	

Comments:

US ENVIRONMENTAL PROTECTION AGENCY
NEW ENGLAND LABORATORY

Brunswick Naval Air Station - Brunswick, ME

Metals in Soil Medium Level by ICP

Client Sample ID:	MBR-SB37 D	Lab Sample ID:	AA82443
Date of Collection:	5/1/2008	Matrix	Soil
Date of Digestion:	5/13/2008	Final Volume:	50 mL
Date of Analysis:	05/16/2008	Digestate Dilution:	1
Volume Digested:	N/A	pH:	N/A

CAS Number	Parameter	Concentration mg/Kg	RL mg/Kg	Qualifier
7429-90-5	Aluminum	**8200**	19	
7440-36-0	Antimony	ND	9.7	
7440-38-2	Arsenic	ND	19	
7440-39-3	Barium	**27**	2.9	
7440-41-7	Beryllium	ND	0.97	
7440-43-9	Cadmium	ND	2.9	
7440-70-2	Calcium	**1100**	19	
7440-47-3	Chromium	**13**	2.9	
7440-48-4	Cobalt	**4.5**	2.9	
7440-50-8	Copper	**32**	2.9	
7439-89-6	Iron	**9800**	9.7	
7439-92-1	Lead	**240**	9.7	
7439-95-4	Magnesium	**1800**	19	
7439-96-5	Manganese	**160**	1.9	
7440-02-0	Nickel	**7.8**	5.8	
7782-49-2	Selenium	ND	19	
7440-22-4	Silver	ND	2.9	J2
7440-28-0	Thallium	ND	19	
7440-62-2	Vanadium	**17**	2.9	
7440-66-6	Zinc	**31**	2.9	

Comments:

US ENVIRONMENTAL PROTECTION AGENCY
NEW ENGLAND LABORATORY

Brunswick Naval Air Station - Brunswick, ME

Laboratory Reagent Blank

Client Sample ID:	N/A	Lab Sample ID:	N/A
Date of Collection:	N/A	Matrix	Water
Date of Digestion:	5/13/2008	Final Volume:	50 mL
Date of Analysis:	05/16/2008	Digestate Dilution:	1
Volume Digested:	50 mL	pH:	N/A

CAS Number	Parameter	Concentration ug/L	RL ug/L	Qualifier
7429-90-5	Aluminum	ND	200	
7440-36-0	Antimony	ND	100	
7440-38-2	Arsenic	ND	200	
7440-39-3	Barium	ND	30	
7440-41-7	Beryllium	ND	10	
7440-43-9	Cadmium	ND	30	
7440-70-2	Calcium	ND	200	
7440-47-3	Chromium	ND	30	
7440-48-4	Cobalt	ND	30	
7440-50-8	Copper	ND	30	
7439-89-6	Iron	ND	100	
7439-92-1	Lead	ND	100	
7439-95-4	Magnesium	ND	200	
7439-96-5	Manganese	ND	20	
7440-02-0	Nickel	ND	60	
7782-49-2	Selenium	ND	200	
7440-22-4	Silver	ND	30	
7440-28-0	Thallium	ND	200	
7440-62-2	Vanadium	ND	30	
7440-66-6	Zinc	ND	30	

Comments:

US ENVIRONMENTAL PROTECTION AGENCY
NEW ENGLAND LABORATORY

METALS MATRIX SPIKE (MS) RESULTS

Sample ID: AA82440

PARAMETER	SPIKE ADDED mg/Kg	SAMPLE CONCENTRATION mg/Kg	MS CONCENTRATION mg/Kg	MS % REC	QC LIMITS (% REC)
Antimony	96.0	ND	52.7	55	75 - 125
Arsenic	96.0	ND	87.8	92	75 - 125
Barium	96.0	55	144	93	75 - 125
Beryllium	38.4	ND	36.8	96	75 - 125
Cadmium	48.0	22	61.1	82	75 - 125
Chromium	96.0	170	261	95	75 - 125
Cobalt	96.0	3.5	92.6	93	75 - 125
Copper	96.0	11	102	95	75 - 125
Lead	96.0	490	580	R	75 - 125
Manganese	96.0	160	261	105	75 - 125
Nickel	96.0	ND	93.6	98	75 - 125
Selenium	96.0	ND	85.6	89	75 - 125
Silver	19.2	ND	14.6	76	75 - 125
Thallium	96.0	ND	90.8	95	75 - 125
Vanadium	96.0	14	103	93	75 - 125
Zinc	96.0	46	128	85	75 - 125

Comments:

US ENVIRONMENTAL PROTECTION AGENCY
NEW ENGLAND LABORATORY

Laboratory Duplicate Results

Sample ID: AA82438

PARAMETER	SAMPLE RESULT mg/Kg	SAMPLE DUPLICATE RESULT mg/Kg	PRECISION RPD %	QC LIMITS
Aluminum	5800	5400	7	30
Antimony	ND	ND	NC	30
Arsenic	ND	ND	NC	30
Barium	26	21	21	30
Beryllium	ND	ND	NC	30
Cadmium	4.9	ND	NC	30
Calcium	2600	2500	4	30
Chromium	320	260	21	30
Cobalt	3.3	3.1	6	30
Copper	14	13	7	30
Iron	17000	16000	6	30
Lead	1000	790	23	30
Magnesium	1800	1700	6	30
Manganese	170	150	12	30
Nickel	ND	ND	NC	30
Selenium	ND	ND	NC	30
Silver	ND	ND	NC	30
Thallium	ND	ND	NC	30
Vanadium	14	14	0	30
Zinc	52	49	6	30

Comments:

US ENVIRONMENTAL PROTECTION AGENCY
NEW ENGLAND LABORATORY

Laboratory Fortified Blank (LFB) Results

PARAMETER	LFB AMOUNT SPIKED ug/L	LFB RESULT ug/L	LFB RECOVERY %	QC LIMITS %
Aluminum	1000	973	97	85 - 115
Antimony	1000	920	92	85 - 115
Arsenic	1000	982	98	85 - 115
Barium	1000	1060	106	85 - 115
Beryllium	400	403	101	85 - 115
Cadmium	500	474	95	85 - 115
Calcium	10000	9510	95	85 - 115
Chromium	1000	1000	100	85 - 115
Cobalt	1000	1010	101	85 - 115
Copper	1000	1030	103	85 - 115
Iron	1000	992	99	85 - 115
Lead	1000	945	95	85 - 115
Magnesium	10000	9820	98	85 - 115
Manganese	1000	1020	102	85 - 115
Nickel	1000	1010	101	85 - 115
Selenium	1000	952	95	85 - 115
Silver	200	166	83	85 - 115
Thallium	1000	1010	101	85 - 115
Vanadium	1000	1010	101	85 - 115
Zinc	1000	947	95	85 - 115

Comments:

Samples in Batch: AA82438, AA82439, AA82440, AA82441, AA82442, AA82443

US ENVIRONMENTAL PROTECTION AGENCY
NEW ENGLAND LABORATORY

Solid Laboratory Control Sample (LCS) Results

PARAMETER	LCS RESULTS mg/Kg	CONTROL LIMITS mg/Kg
Aluminum	7630	4820 - 13400
Antimony	98.9	0 - 238
Arsenic	144	116 - 186
Barium	277	223 - 321
Beryllium	102	83.7 - 121
Cadmium	87.1	76.3 - 114
Calcium	6030	5530 - 8170
Chromium	119	96.6 - 143
Cobalt	65.9	54.7 - 79.9
Copper	83.8	64.9 - 101
Iron	14400	8880 - 24400
Lead	112	94.1 - 145
Magnesium	2350	2020 - 3160
Manganese	375	313 - 455
Nickel	99.6	81.0 - 123
Selenium	134	107 - 169
Silver	92.2	64.2 - 129
Thallium	164	132 - 208
Vanadium	125	99.5 - 159
Zinc	106	92.5 - 142

Comments:

ENVIRONMENTAL PROTECTION AGENCY

REGION 1

CHAIN OF CUSTODY RECORD

PROJ. NO.: 08050003

PROJECT NAME: Brunswick Naval Air Station Brunswick, ME

SAMPLERS: (Signature) Dan Granz, Jerry Hoefe

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	ICP Metals (Pb only)							REMARKS
	5/1/08	11:00		✓	MBR-SB01	1	X							
		12:18		✓	MBR-SB12	1	X							
		11:40		✓	MBR-SB22	1	X							
		1500		✓	MBR-SB35	1	X							
		1520		✓	MBR-SB37	1	X							
		1520		✓	MBR-SB37D	1	X							

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Scott [illegible]	5/2/08 0925				
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks	
		[illegible] (ESAT)	05/02/08 09:25	Mike Daly RPM	

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

1-16946

APPENDIX C

SUPPLEMENTAL HISTORICAL INFORMATION

C-1 MACHINE GUN BORESIGHT RANGE

MACHINE GUN BORESIGHT RANGE
(SELECTED PAGES

FINAL
PRELIMINARY ASSESSMENT
NAVAL AIR STATION BRUNSWICK, MAINE

February 2006

Prepared for:

Naval Facilities Engineering Command
Engineering Field Activity, Northeast
10 Industrial Highway
Mail Stop #82
Lester, PA 19113-2090

Prepared by:

MALCOLM PIRNIE, INC.
300 E. Lombard Street
Suite 610
Baltimore, Maryland 21202

Table 5.2-1: Conceptual Site Model Information Profiles – Machine Gun Bore Sight Range

Profile Type	Information Needs	Preliminary Assessment Findings
Range/Site Profile	Installation Name	Naval Air Station Brunswick
	Installation Location	Cumberland County, Maine
	Range/Site Name	Machine Gun Bore Sight Range
	Range/Site Location	The site is located in the eastern portion of the facility adjacent to Building 55 and extends to an open field behind the building.
	Range/Site History	In the 1950s, pilots used the range to calibrate their aircraft mounted machine guns.
	Range/Site Area and Layout	Pilots fired toward the east into a berm after taxing down the runway to the compass rose. The range area encompassed 0.3 acres with an SDZ 1029.4 acres.
	Range/Site Structures	A berm appears on the 1957 aerial photograph of the installation. No other range related structures are discernable from the aerial. Based on the site walk, any structures that existed when the range was active are no longer present.
	Range/Site Boundaries	N: Building 55, Building 52, and containment pond. S: Building 50 E: Open field and forested area. W: Parking lot and Orion Street
	Range/Site Security	There are no security or access restrictions to the site once on the installation.
Munitions/ Release Profile	Munitions Types	Machine gun (small arms) ammunition including .50 caliber and .30 caliber and pistol ammunition including .20 caliber, .22-caliber, and .38 caliber.
	Maximum Probability Penetration Depth	Small arms, less than one foot.
	MEC Density	Small arms, not applicable.
	MEC Scrap/Fragments	No MEC scrap/fragments were found during the visual survey.

Table 5.2-1: Conceptual Site Model Information Profiles – Machine Gun Bore Sight Range

Profile Type	Information Needs	Preliminary Assessment Findings
	Associated Munitions Constituents	MC, including lead from the machine gun ammunition, could potentially be present at the site. Other associated MC (less likely to be of concern) may include: antimony (increases hardness), arsenic (present in lead), nickel (coating on some shot), and lead azide (associated with gunpowder). Sampling data was not available for the area to confirm lead or other potential MC concentrations at the site.
	Migration Routes/Release Mechanisms	Erosion – potential (in non-paved open area) Surface Water runoff – potential (in non-paved open area) Human Intervention – potential (in non-paved open area).
Physical Profile	Climate	Continental climate with three well-defined seasons. Highest temperatures occur in July (79 F or higher). Coldest temperatures occur in January (21 F or lower).
	Topography	The former Machine Gun Bore Sight Range is relatively flat.
	Geology	Characterized by two primary components: unconsolidated sediments and Paleozoic bedrock.
	Soil	Suffield-Buxton-Hollis Association. Deep to shallow, moderately well drained to somewhat poorly drained soils with low permeability.
	Hydrogeology	Groundwater is used as the municipal water supply in the residential area north of NASB. Most of the wells in the vicinity are between 101 and 300 feet deep. There are no wells within the boundary of the range.
	Hydrology	Surface water flows off the paved area away from Building 55.
	Vegetation	The undeveloped portion of the range is grassy with various tree species.
Land Use and Exposure Profile	Current Land Use	A small portion of the range is developed and used as a parking lot for Building 55. The remaining area is undeveloped and unused.
	Current Human Receptors	Current human receptors include: Navy personnel, authorized visitors, and maintenance workers/contractors.

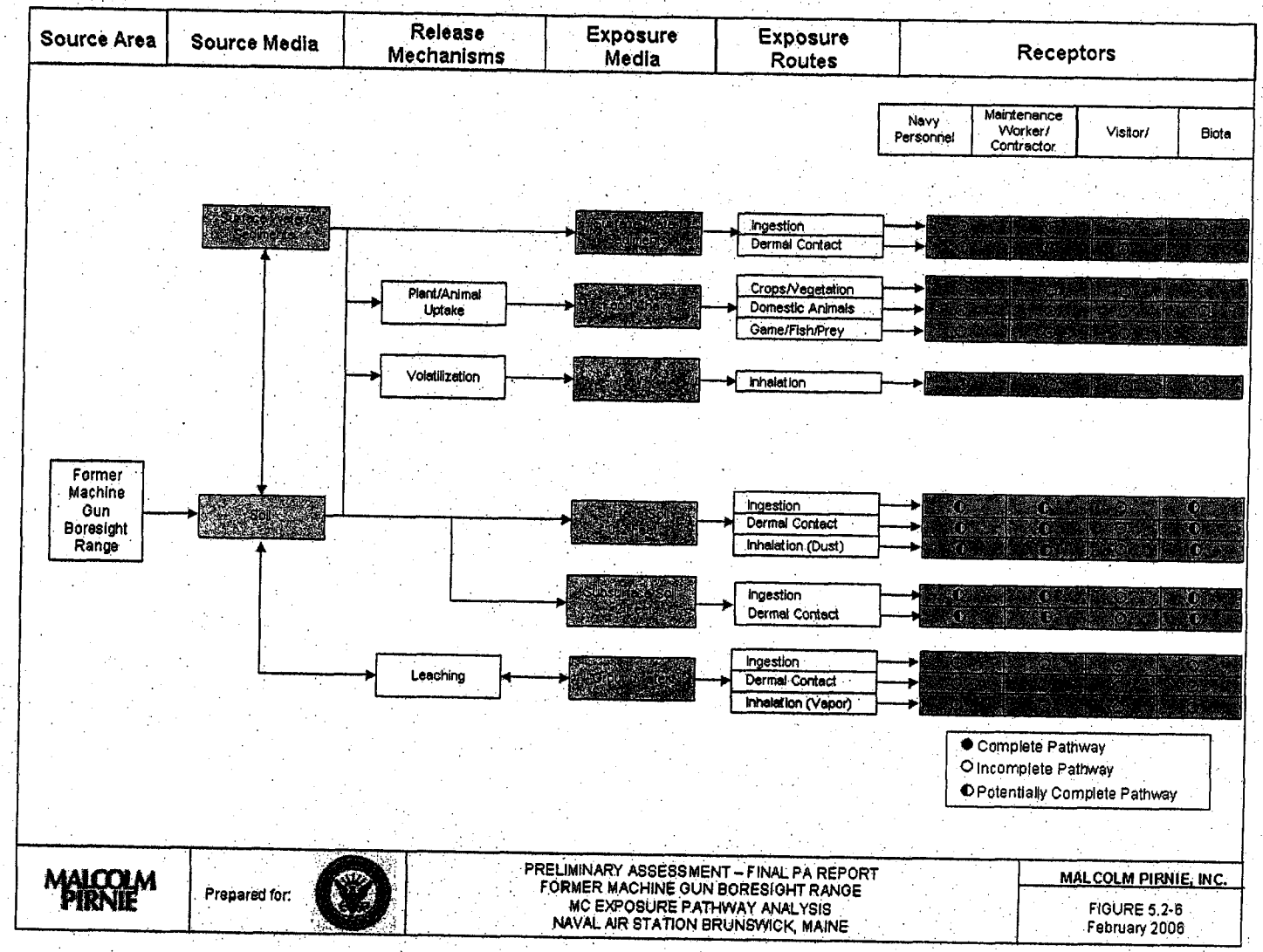
Table 5.2-1: Conceptual Site Model Information Profiles – Machine Gun Bore Sight Range

Profile Type	Information Needs	Preliminary Assessment Findings
	Current Activities (frequency, nature of activity)	Building 55 is located in the area. The remaining portion of the range is undeveloped and unused.
	Potential Future Land Use	Same as current.
	Potential Future Human Receptors	Same as current.
	Potential Future Land Use-Related Activities:	No foreseen change to current use.
	Zoning/Land Use Restrictions	No site specific restrictions or access controls.
	Demographics/Zoning	Cumberland County population density is approximately 50,000 persons per square mile.
	Beneficial Resources	Groundwater is a source of the municipal water supply.
Ecological Profile	Habitat Type	Open field and forested area behind Building 55.
	Degree of Disturbance	Limited to surface activities near Building 55.
	Ecological Receptors and Species of Special Concern	Potential ecological receptors include indigenous species. No species of special concern are known to be present at the site.
	Relationship of MEC/MC Sources to Habitat and Potential Receptors	MEC is not suspected at the site. MC potential migration pathways include leaching to groundwater, plant/animal uptake, and ingestion, dermal contact, and dust inhalation from surface soil.

A key element of the CSM is the exposure pathway analysis. For MEC, a complete or potentially complete exposure pathway must include the following components: 1) a source (e.g., locations where MEC are expected to be found); 2) access (e.g., controlled or uncontrolled access, items on the surface or within the subsurface); 3) an activity (e.g., non-intrusive grounds maintenance or intrusive construction); and 4) receptors (e.g., Navy personnel, construction workers, recreational users, or authorized visitors). It is important to recognize that environmental mechanisms (e.g., erosion) and/or human intervention may result in the repositioning of MEC.

For MC, a complete or potentially complete exposure pathway must include the following components: 1) a source (e.g., locations where MC are expected to be found); 2) an exposure medium (e.g., surface soil); 3) an exposure route (e.g., dermal contact); and 4) receptors (e.g., Navy personnel, construction workers, recreational users, or authorized visitors). If the point of

Figure 5.2-6: MC Exposure Pathway Analysis



MACHINE GUN BORESIGHT RANGE
RANGE DESIGN LETTER
FROM PA APPENDIX C-2

February 26, 1943

MACHINE GUN
RANGE SPECIFICATIONS

IN REPLY ADDRESS
THE BUREAU OF YARDS AND DOCKS
AND REFER TO No.

NOy-5354
NOy-5785

NAVY DEPARTMENT

BUREAU OF YARDS AND DOCKS

WASHINGTON, D.C.

From: Chief of the Bureau of Yards and Docks.
To: Officer-in-Charge of Construction, Contracts NOy-5354 and NOy-5785, 150 Causeway Street, Boston, Mass.

Subject: Revised Layouts of Naval Air Stations, Brunswick, Sanford, Lewiston, and Rockland, Maine - Approach Zones thereat.

Reference: (a) BuY&D ltr dated 22 February 1943 to OinC NOy-5354.

Enclosure:
(A) 1 vandyke and 1 blueprint of Brunswick layout, Y&D Dwg. # 208770, Revised 2/23/43.
(B) 1 vandyke and 1 blueprint of Rockland layout, Y&D Dwg. # 208768, revised 2/18/43.
(C) 3 blueprints of "Sketch showing cleared area and approach zone", for Brunswick, Maine. (Herewith)
(D) 3 copies of "Sketch showing typical cleared area and approach zone" for Air Station. (herewith)
(E) 1 vandyke and 1 blueprint of Y&D Dwg. 219627 - "Schematic Layout for Aircraft fixed Machine Gun Range".
(F) 1 vandyke and 1 blueprint of Y&D Dwg. 153340,- "Tail Mound, Gun Stand and Gun Stop".

1. Enclosures (A) to (F) inclusive are forwarded for the information of the Officer-in-Charge of Construction Contracts NOy-5354 and NOy-5785.

2. It is to be noted that the sewerage disposal plant has been relocated in the area adjoining the incinerator at Brunswick where previously shown on Bureau layouts.

3. Enclosure (E) is forwarded to illustrate graphically the requirements of the cleared areas and approach zones at the end of each runway at Brunswick only.

FOR VICTORY BUY UNITED STATES WAR BONDS AND STAMPS

RG 72, Butler
E. 62B, Gen'l. Corres., 1943-45
Box 341, NA 116

Reproduced at the National Archives

NOy-5354
NOy-5786

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4. Enclosure (D) is forwarded to illustrate graphically the cleared areas and approach zones required at the end of each of the runways at Lewiston, Sanford, and Rockland. The Bureau of Aeronautics has directed that runway clearance lines at each of the above three fields are to be 500 ft. from the edge of the runway paving instead of the edge of the safety strip as called for on Enclosure (D) and in the Bureau of Yards and Docks' Drawing 191523 "Standard plans for runways and warming-up platforms".

5. The radio transmission station at Brunswick is to be located in the S-W sector of the airport where shown on the layout. The Officer-in-Charge will be advised by separate correspondence concerning the details of this installation and the size of the building to be erected.

6. The machine gun ranges at the subject stations will be located as shown on the revised layouts. The machine gun ranges are to be built in accordance with Enclosure (E), except that there is to be no protective shed built over the firing position and the 100 ft. maximum distance between the gun position and the target is to be used. Enclosure (F) is forwarded for the information of the Officer-in-Charge in connection with the construction of the machine gun range. It is also to be noted that only one firing position is to be built and that no intermediate firing positions, as shown on previous layouts, are to be built.

7. Reference (a) advised concerning the ammunition storage requirements at Brunswick. The Officer-in-Charge will note that the layout of ammunition storage facilities has been purposely omitted from Enclosure (A). When the magazine layout is returned by the Officer-in-Charge, and approved by the Bureau of Ordnance, a revised layout of Brunswick will be forwarded.

James S. Kenny
By direction of Chief of Bureau

BuAer (W/blueprint of Encls)
CC - ComOne (W/blueprint of encls)
ROinC NOy-5354, P.O. Box 90,
Brunswick, Maine (W/blueprint of encls)

MACHINE GUN BORESIGHT RANGE

1962 DRAWING
FROM PA APPENDIX C-2

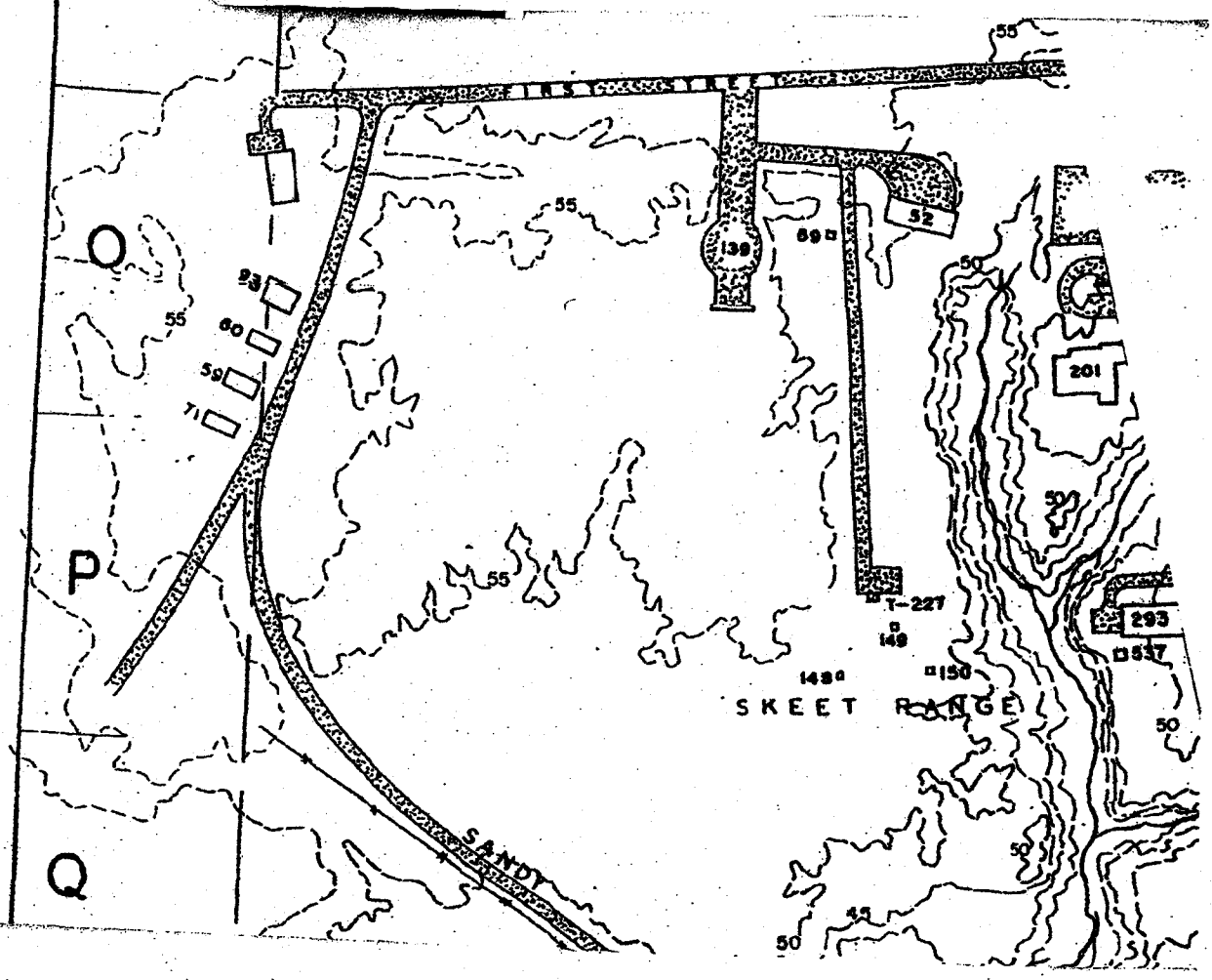
NO.	AMD OR COR	DESCRIPTION OF AMENDMENT OR CORRECTION	DATE	DPWO APPROVAL
2	AMD	ACQUIRED BUILDINGS ADDED; SHEET # CHANGED	11/19/65	
1	COR	401-418, 420-451, 454-466 REMOVED	5/14/65	JN

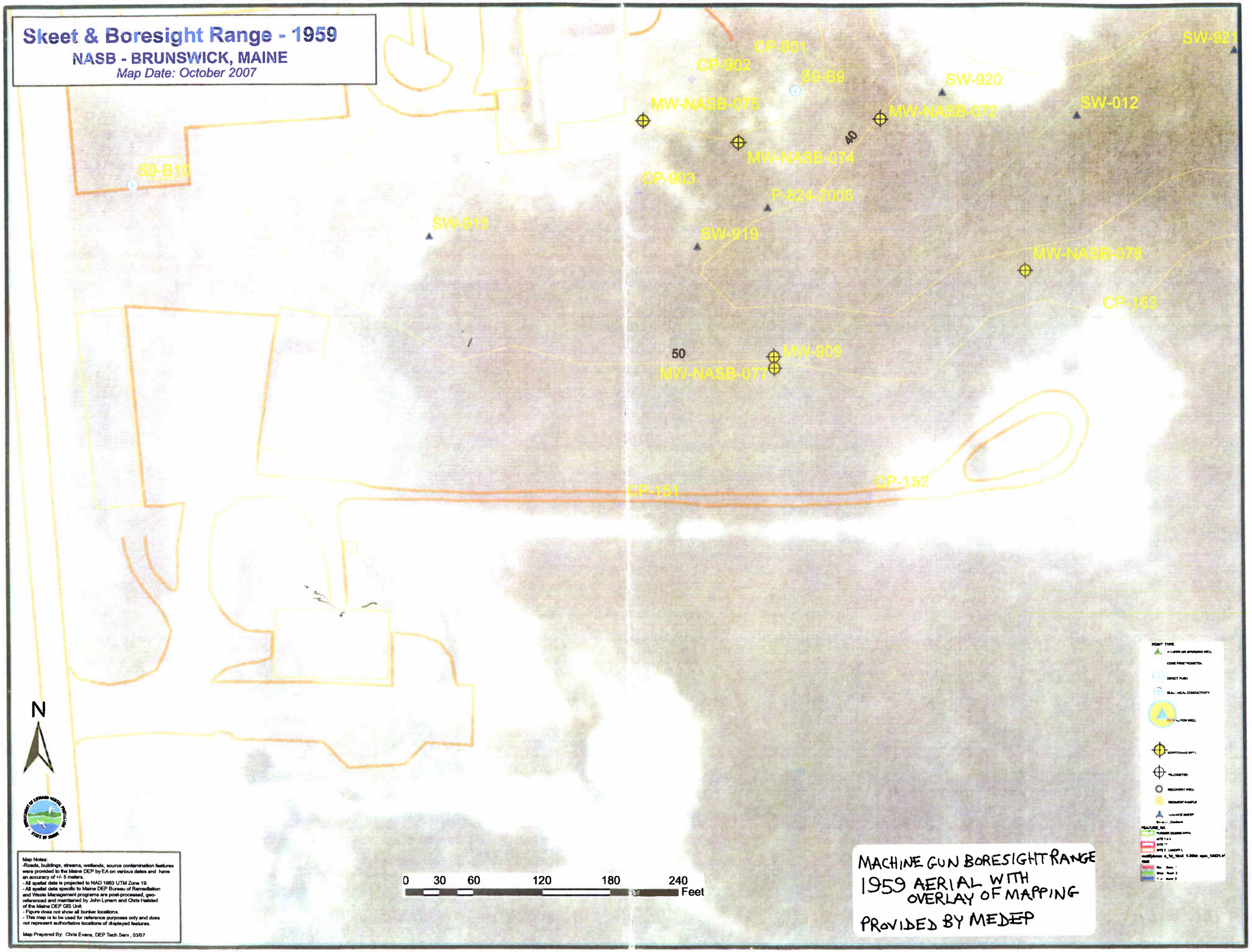
DRAWN BY BABBIDGE/H.C. Henry
BRANCH MGR Nagar
DIRECTOR OF PLANNING MacLEARN
SUBMITTED BY DEP DPWO DATE 4/62
CONCUR CO DATE 4/62
APPROVED BY BUDOCKS

DEPARTMENT OF THE NAVY BUREAU OF YARDS & DOCKS

U.S. NAVAL AIR STATION
BRUNSWICK, MAINE
EXISTING CONDITIONS MAP
OPERATIONS AREA

SCALE: GRAPHIC
SHEET 3 OF 7
Y & D DWG No. 925130

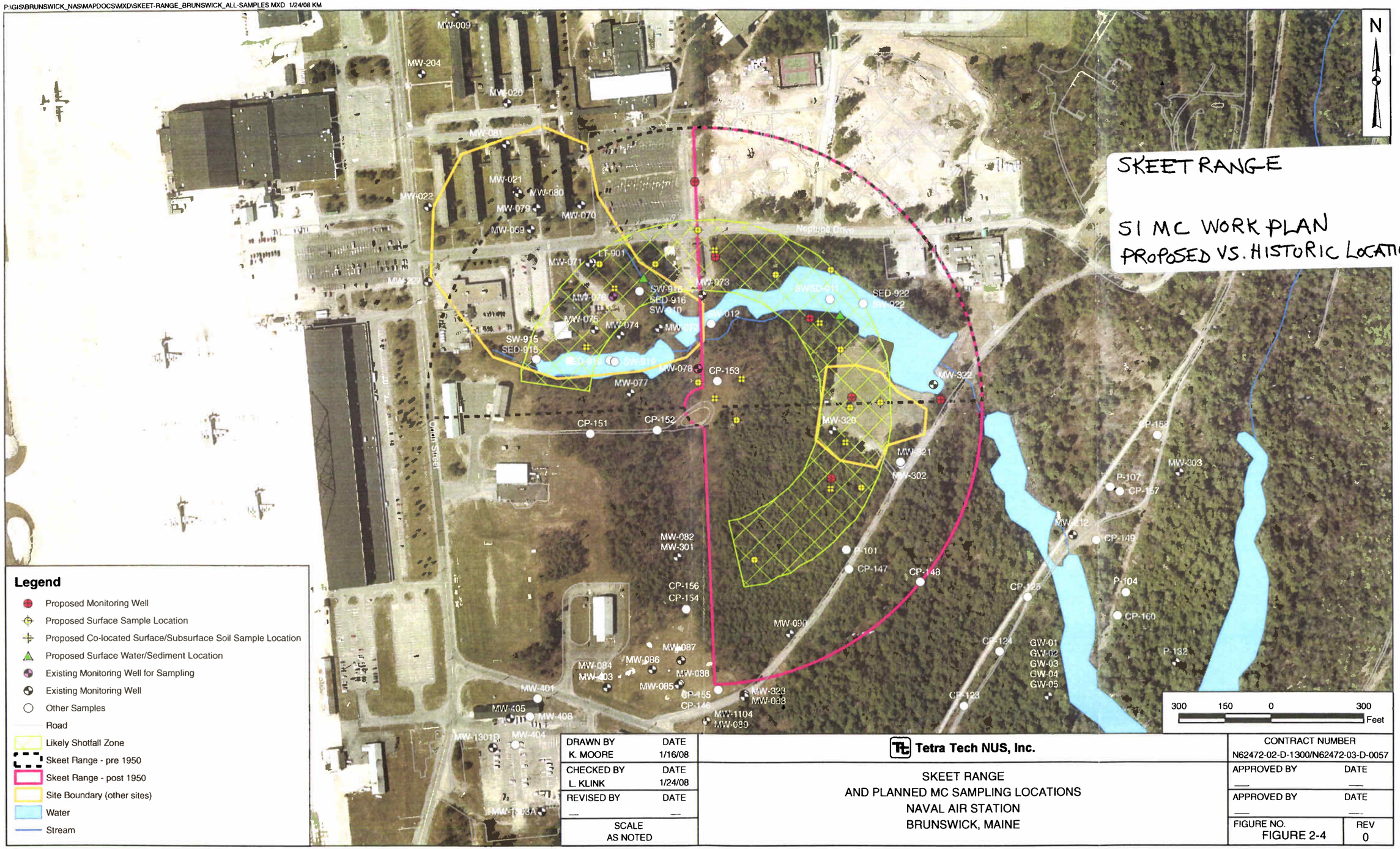




MACHINE GUN BORESIGHT RANGE
1959 AERIAL WITH
OVERLAY OF MAPPING
PROVIDED BY MEDEP

C-2 SKEET RANGE

P:\GIS\BRUNSWICK_NAS\MAPDOCS\MXD\SKEET-RANGE_BRUNSWICK_ALL-SAMPLES.MXD 1/24/08 KM



DRAWN BY K. MOORE	DATE 1/16/08	Tetra Tech NUS, Inc.	CONTRACT NUMBER N62472-02-D-1300/N62472-03-D-0057
CHECKED BY L. KLINK	DATE 1/24/08	SKEET RANGE AND PLANNED MC SAMPLING LOCATIONS NAVAL AIR STATION BRUNSWICK, MAINE	APPROVED BY — DATE —
REVISED BY —	DATE —		APPROVED BY — DATE —
SCALE AS NOTED			FIGURE NO. FIGURE 2-4 / REV 0

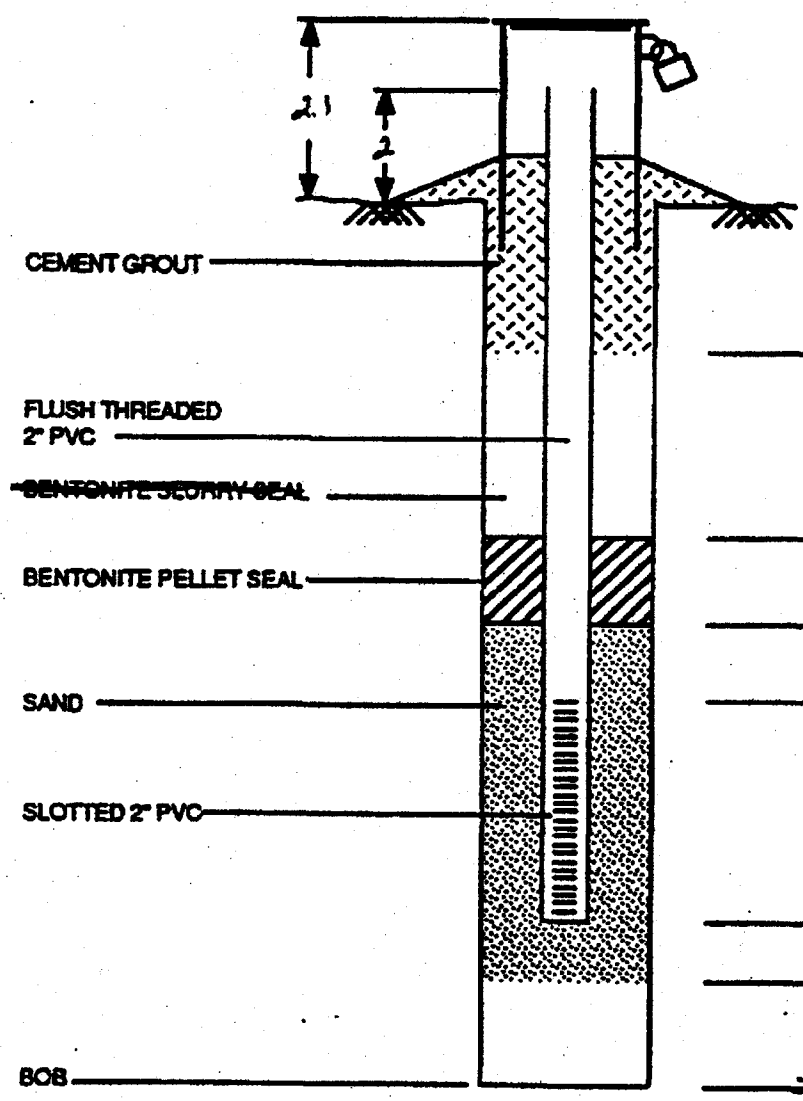
SKEET RANGE
USE OF SITE 9 WELL MW-076

Soil Boring Logs and/or Well Construction Data

Sample Location: MW-NASB-076
Previous Designation: MW-908, MW-76
File Name: MW-NASB-076.pdf

WELL INSTALLATION DETAILS

PROJECT NO.: 4607-31	PROJECT NAME: NAVAL AIR STATION, BRUNSWICK, MAINE	WELL NO.: MW-908 (Redrill)
INSTALLED BY: New England Boring/T.L.	DATE INSTALLED: 10-4-89	BORING DIAMETER: ~10"
WELL DIAMETER: 2"	WELL MATERIAL: PVC	BACKFILL MATERIAL: Marie Silica Sand



DEPTH (feet)	ELEVATION (feet)
0	50.8
2'	48.8
2'	48.8
5'	45.8
7.5	43.3
17.5	33.3
18'	32.8
30'	20.8

E.C.JORDAN CO.

NAVAL AIR STATION, BRUNSWICK			Boring no: MW-908
Client: US. DEPARTMENT OF THE NAVY			Project no: 4807-31
Contractor: NEW ENGLAND BORING CONTRs.		Date started: 8/18/89	Completed: 8/18/89
Method: AUGER	Casing size: 6 5/8" ID	PID: 10.2	Protect'n level: D
Ground el: 50.8'	Soil drilled: 30'	Rock drilled: NA	Total Depth: 30'
Logged by: G. Dawkins	Ch'd by: TL	Date: 10-17-89	Below grnd: 9.0'
			Page: 1 of 2

REDRILLED ON 10-4-89
TO REPLACE DAMAGED WELL

Depth (ft)	PID ambient air	Sample no. and type	Sample	Recovery	CLP	PID headspace (ppm)	Soil/rock description	Soil class	Blows/6-in. and RQD % (0 20 40 60 80 100)	Well data	Elevation (ft)
	1.0	S-1	X	1.8		2.0	Brown fine sand trace silt very loose, Dry — OUTWASH —	SP	1 2 5 6		
2.0											
	1.0	S-2	X	1.6		1.0	Light brown fine sand trace silt very loose, Dry — OUTWASH —	SP	5 5 5 5		
4.0											
	1.0	S-3	X	1.8		1.0	Same as Sample S-2 Collected analytical sample 0915 — OUTWASH —	SP	2 3 3 3		
6.0											
	1.0	S-4	X	1.9		1.5	Same as sample S-2 with oxide stains — OUTWASH —	SP	2 3 2 2		
8.0											
	1.0	S-5	X	1.8		1.0	Same as sample S-4 wet, Loose Collected analytical Sample — OUTWASH —	SP	2 2 3 3		
10.0											
	1.0	S-6	X	1.8		1.0	Same as sample S-4 — OUTWASH —	SP	2 2 3 3		
12.0											
	1.0	S-7	X	1.8		1.0	Brown fine sand trace silt wet, Loose — OUTWASH —	SP	1 2 3 3		
14.0											
	1.0	S-8	X	1.0		1.0	Same as sample S-7 — OUTWASH —	SP	1 2 1 2		
16.0											
	1.0	S-9	X	1.8		1.0	Same as Sample S-7 Collected Analytical Sample 1010 — OUTWASH —	SP	2 3 2 3		
18.0											
	1.0	S-10	X	1.5		1.0	Same as sample S-7 — OUTWASH —	SP	2 2 3 3		
20.0											

*U = Thin wall tube S = Split spoon R = Rock

E.C. JORDAN

NAVAL AIR STATION, BRUNSWICK			Boring no: MW-90B
Client: US. DEPARTMENT OF THE NAVY			Project no: 4807-31
Contractor: NEW ENGLAND BORING CONTRS.		Date started: 8/18/89	Completed: 8/18/89
Method: AUGER	Casing size: 6 5/8" I.D.	PID: 10.2	Protect'n level: D
Ground el: 50.8'	Soil drilled: 30'	Rock drilled: NA	Total Depth: 30'
Logged by: G. Dowkes	Ch'd by:	Date:	▽ Below grnd: 9.0'
			Page 2 of 2

REDRILLED ON 10-4-89 TO REPLACE WELL

Depth (ft)	PID ambient air	Sample no. and type	Sample	Recovery	CLP	PID headspace (ppm)	Soil/rock description	Soil class	Blows/6-in. and RQD % (0 20 40 60 80 100)	Well data	Elevation (ft)
	1.0	S-11	X	1.8		1.5	Same as sample S-7 —OUTWASH—	SP	2 2 3 4		
22.0											
							No SAMPLE				
24.0											
	1.0	S-12	X	1.8		1.0	Same as sample S-7 - OUTWASH	SP	2 2 3 3		
26.0											
	1.0	S-13	X	1.8		1.0	Same as sample S-7 -OUTWASH-	SP	2 2 3 4		
28.0											
	1.0	S-14	X	1.8		1.0	Same as sample S-7 Collected analytical sample 1100 - OUTWASH -	SP	2 2 3 3		
30.0											
							END OF BORING				

*U = Thin wall tube S = Split spoon R = Rock

E.C. JORDAN

SKEET RANGE
USE OF SITE WELL MW-078

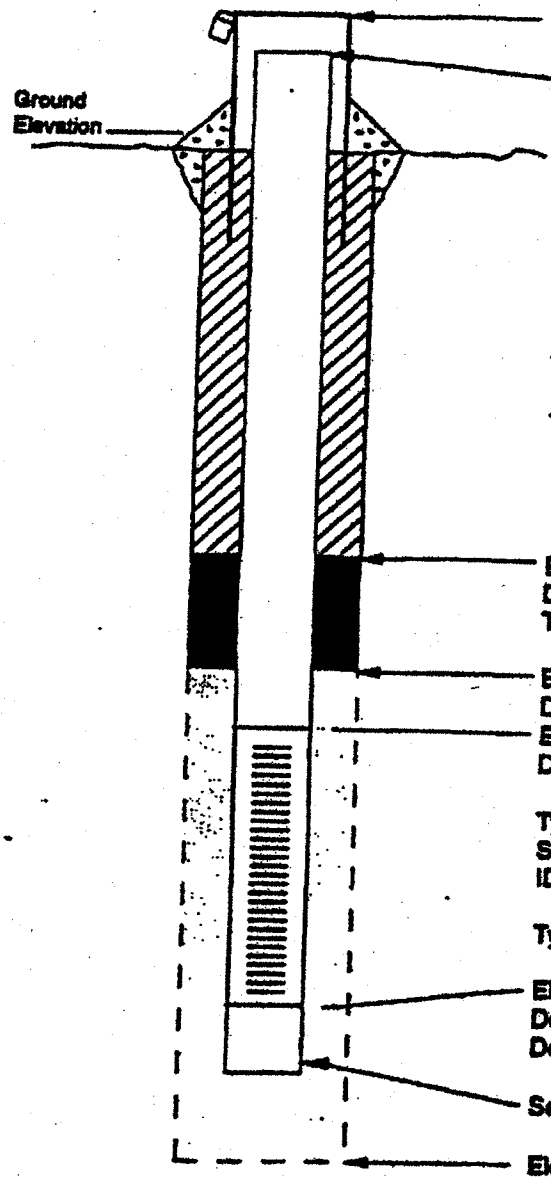
Soil Boring Logs and/or Well Construction Data

Sample Location: MW-NASB-078
Previous Designation: MW-910
File Name: MW-NASB-078.pdf

2/2

MONITORING WELL CONSTRUCTION DIAGRAM

Project NASB Site 9 Driller MARK D'AMBROSIA
Project No. 7127-09 Boring No. MW910 Drilling Method HSA
Elevation TBD Date Installed 1/17/93 Development Method



Elevation of Top of Surface Casing:
Stick-up of Casing Above Ground Surface: ~~[illegible]~~ ?
Elevation of Top of Riser Pipe: 5.4
Type of Surface Seal: CONCRETE
Type of Surface Casing: ~~PVC~~ STEEL

ID of Surface Casing: 4".

Diameter of Borehole: 4.25" ID HSA

Riser Pipe ID: 2 INCH
Type of Riser Pipe: PVC ~~[illegible]~~

Type of Backfill: CONCRETE TO SET WELL BOX

Elevation of Top of Seal:
Depth of Top of Seal: 3 FT
Type of Seal: BENTONITE PELLETS - PURE GOLD

Elevation of Top of Sand:
Depth of Top of Sand: 5 FT
Elevation of Top of Screen:
Depth of Top of Screen: 7'

Type of Screen: PVC
Slot Size x Length: 10 SLOT
ID of Screen: 2 INCH

Type of Sandpack: POOL FILTER SAND

Elevation of Bottom of Screen:
Depth of Bottom of Screen: 12'
Depth of Sediment Sump with Plug:

Sediment Sump with Plug

Elevation of Bottom of Borehole:
Depth of Bottom of Borehole: 13

ABB Environmental Services, Inc.

NASB Field Boring Log

Client: U.S. Navy, Brunswick		Project No. 7127 09	Boring No.: MW 910
Contractor: NHB	Date Started: 1/12/93		Protection: D
Method: HSA/CASING	Casing Size: 4.25 (ID)		Completed: 1/17/93
Ground Elev.: 50.57	Soil Drilled: 17		PI Meter: HNK/TIP
Logged by: K. LaMarre	Checked by:		Total Depth: 17 ft
			Below Ground: 8.7
Screen: 5 (ft.)	Riser: 10.4 (ft.)	Diam: 2" (ID)	Material: PVC
			Page 1 of: 2

DEPTH (FT)	SAMPLE NUMBER	SAMPLE DEPTH	LAB ANALYSES	PEN/RECOVERY	PID (ppm)	SOIL/ROCK DESCRIPTION	SOIL CLASS	BLOWS/6-IN.	WELL DATA	ELEVATION (FT.)
						TOPSOIL - DARK BROWN FINE SANDY SILT W/ORGANICS 1' THICK				
						<SAND>				
	S-1	1-3	LDA	3/1.7		0-0.1 BLACK SILTY FINE SAND				
						0.1-1.7 REDDISH BROWN TO TAN FINE SAND, MOIST, LOOSE	SP	2/2/3/4 (5)		
5	S-2	5-7		2/1.7		0-0.2 SIMILAR TO S-1 (0.1-1.7)				
						0.2-0.4 REDDISH BROWN FINE SAND, MOIST		5/10/16		
						0.4-0.8 DARK BROWN FINE SAND LITTLE SILT, MOIST	SP/SM	(22)		
						0.8-1.7 BLACK FINE SAND, LITTLE SILT, WET MEDIUM DENSE				
10	S-3	10-12		3/1.4				3/2/4/3 (9)		
						<TRANSITIONAL SOIL>				
					BKG	0-0.7 BROWN FINE SAND, SOME SILT				
						0.7-1.4 REDDISH BROWN AND BROWN FINE SAND, LITTLE SILT, WET	SM			
15	S-4	15-17		2/2		<SILTY CLAY>				
						GRAY SILTY CLAY WITH OCCASIONAL FINE SAND LAYERS. BLACK STREAKS AND TRACE SHELL FRAGMENTS, WET, VERY SOFT	MH/CL	1/2		
20						BOE @ 17 ft				
						INSTALL WELL				

PROPORTIONS	(-) AMOUNT (+)	ABBREVIATIONS		
Trace (tr)	0-10%	f = fine	gr = gray	S = Split Spoon
Little (li)	10-20%	m = medium	bn = brown	T = Thin Wall Tube Sample
Some (so)	20-35%	c = coarse	blk = black	V = Vane Shear Test
and	35-50%			

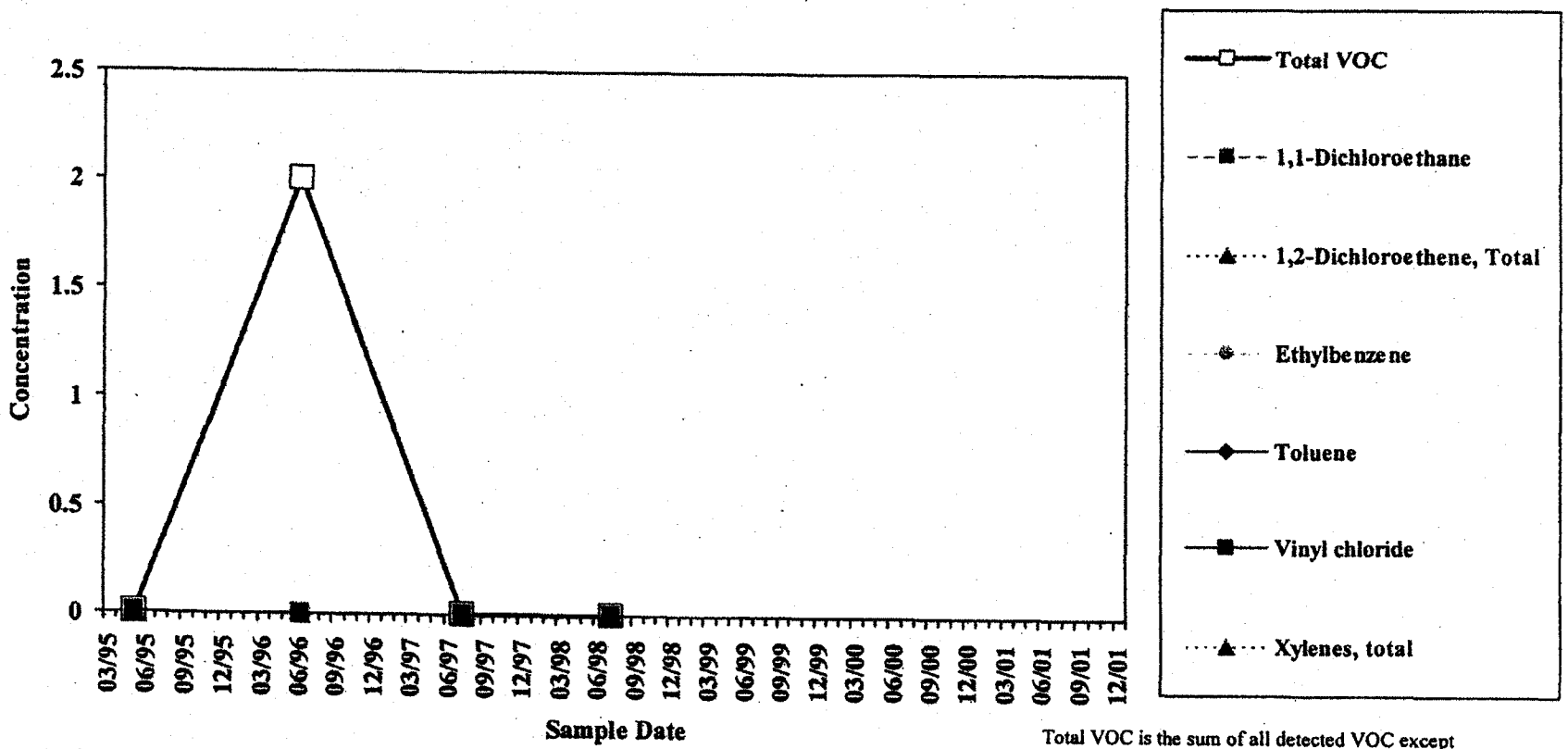
ABB Environmental Services, Inc.

Sample Location:

MW-NASB-078

Site 9
Ground Water

Chemicals of concern: Volatiles



Analyte concentrations less than the sample detection limit are plotted as zero concentration on this chart.

Total VOC is the sum of all detected VOC except Methylene Chloride and Acetone.

Sample concentration units: µg/L

1,1-Dichloroethane		1,2-Dichloroethene, total		Ethylbenzene		Toluene		Vinyl chloride		Xylenes, total	
MEG:	70	MEG:	70	MEG:	700	MEG:	1400	MEG:	0.15	MEG:	600
MCL:	-	MCL:	70	MCL:	700	MCL:	1000	MCL:	2	MCL:	10000

SKEET RANGE
(SELECTED PAGES)

FINAL
PRELIMINARY ASSESSMENT
NAVAL AIR STATION BRUNSWICK, MAINE

February 2006

Prepared for:

Naval Facilities Engineering Command
Engineering Field Activity, Northeast
10 Industrial Highway
Mail Stop #82
Lester, PA 19113-2090

Prepared by:

MALCOLM PIRNIE, INC.
300 E. Lombard Street
Suite 610
Baltimore, Maryland 21202

Table 5.3-1: Conceptual Site Model Information Profiles – Skeet Range		
Profile Type	**Information Needs**	**Preliminary Assessment Findings**
Range/Site Profile	Installation Name	Naval Air Station Brunswick
	Installation Location	Cumberland County, Maine
	Range/Site Name	Skeet Range
	Range/Site Location	Located approximately 75 meters north and 100 meters east of the current Building 55 and adjacent to the Old Machine Gun Range.
	Range/Site History	Shown on installation maps from 1946 and in an aerial photo from 1957
	Range/Site Area and Layout	The Skeet Range encompasses approximately 73.2 acres. On the map from 1946, the range is shown with the firing towards the north; however, in the 1957 aerial photo and on later maps, the range is shown firing to the east.
	Range/Site Structures	Aerial photos show shot house for skeet; however, structures that existed when the range was active are no longer present at the site.
	Range/Site Boundaries	N: Building 211 S: Forested area E: Forested area. W: Orion Street.
	Range/Site Security	No security or access restrictions to the site once on the installation.
Munitions/ Release Profile	Munitions Types	Shotgun ammunition – 12 gauge
	Maximum Probability Penetration Depth	Small arms, one foot or less.
	MEC Density	Not applicable.
	MEC Scrap/Fragments	Not applicable.
	Associated Munitions Constituents	Primary MC is lead, other associated MC (less likely to be of concern) may include: antimony (increases hardness), arsenic (present in lead), nickel (coating on some shot), lead azide (associated with gunpowder) and PAHs.

Table 5.3-1: Conceptual Site Model Information Profiles – Skeet Range

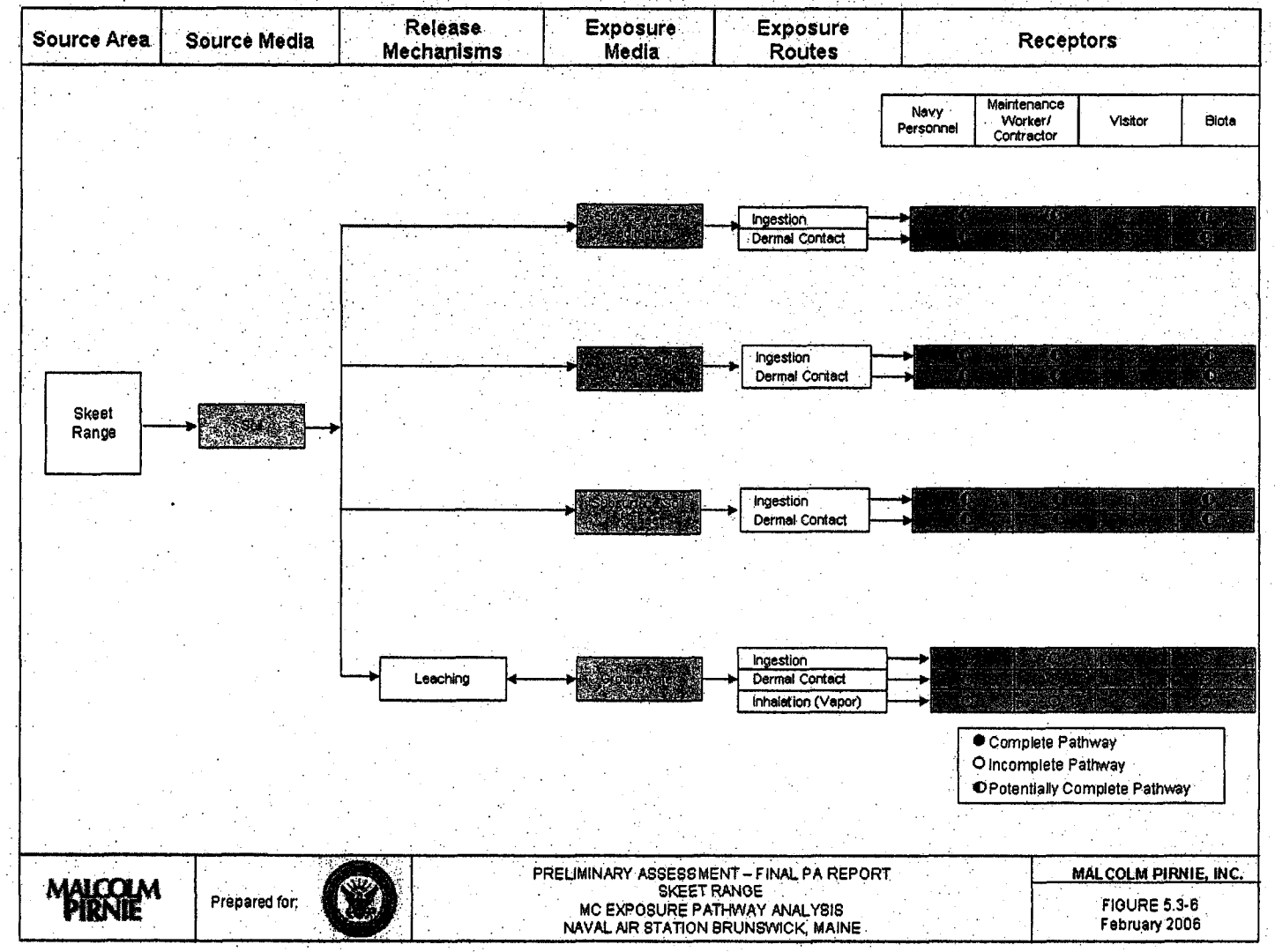
Profile Type	Information Needs	Preliminary Assessment Findings
	Migration Routes/Release Mechanisms	Erosion – The area is flat, so no significant run-off is anticipated. Run-off that does occur will flow into retention pond. Human Intervention – Minimal (current and anticipated future use limited to grounds crew, pedestrian traffic)
Physical Profile	Climate	Continental climate with three well-defined seasons. Highest temperatures occur in July (79 F or higher). Coldest temperatures occur in January (21 F or lower).
	Topography	The Skeet Range is mainly flat.
	Geology	Silty clay with occasional fine sand, trace small fragments, wet soft, very soft) corresponds to the description of the Presumpscot Formation.
	Soil	Soils are from the Suffield-Buxton-Hollis Association which consists of deep to shallow, moderately well drained to somewhat poorly drained soils that are characterized by low permeability. The field boring log for monitoring well 078 lists the topsoil as dark brown, fine, sandy silt with black, silty, fine sand just below the topsoil. From 0.1 to 5 ft the soil is reddish brown to tan moist loose sand.
	Hydrogeology	The yield of bedrock wells in the vicinity of the NASB ranges from less than 10 gpm to over 50 gpm. Most of the wells in the vicinity are between 101 and 300 feet deep.
	Hydrology	Surface water flows towards retention pond.
	Vegetation	The majority of the Skeet Range (post-1950) area is open and grassy with various crops of woodland species of trees (mostly pine). The Skeet Range (pre-1950) is highly developed and vegetation consists mainly of urban landscaping and small trees.
Land Use and Exposure Profile	Current Land Use	The Skeet Range (pre-1950) is highly developed and is covered by several buildings as shown on Map 5.3-2. The Skeet Range (post-1950) is undeveloped except for the northernmost portion which contains a baseball field and is intersected by Neptune Drive.
	Current Human Receptors	Current human receptors include: Navy personnel, maintenance workers/contractors, and visitors.

Table 5.3-1: Conceptual Site Model Information Profiles – Skeet Range		
Profile Type	**Information Needs**	**Preliminary Assessment Findings**
	Current Activities (frequency, nature of activity)	Grounds crew and pedestrian traffic.
	Potential Future Land Use	Same as current.
	Potential Future Human Receptors	Same as current
	Potential Future Land Use-Related Activities:	Same as current.
	Zoning/Land Use Restrictions	No site specific restrictions or access controls.
	Demographics/Zoning	Cumberland County population density is approximately 50,000 persons per square mile.
	Beneficial Resources	Groundwater is the source of the municipal water supply. However, the wells are screened in the deep aquifer (>100 feet below surface).
Ecological Profile	Habitat Type	Scrub/shrub in open field, wetland area nearby along with retention pond.
	Degree of Disturbance	Limited to surface activities including grounds maintenance.
	Ecological Receptors and Species of Special Concern	Potential ecological receptors include indigenous species. No species of special concern are known to be present at the site.
	Relationship of MEC/MC Sources to Habitat and Potential Receptors	MEC not suspected at site. MC potential migration pathways include infiltration to subsurface soil, infiltration and leaching to groundwater, and to a lesser extent dust emissions and volatilization to ambient air.

A key element of the CSM is the exposure pathway analysis. For MEC, a complete or potentially complete exposure pathway must include the following components: 1) a source (e.g., locations where MEC are expected to be found); 2) access (e.g., controlled or uncontrolled access, items on the surface or within the subsurface); 3) an activity (e.g., non-intrusive grounds maintenance or intrusive construction); and 4) receptors (e.g., Navy personnel, construction workers, recreational users, or authorized visitors). It is important to recognize that environmental mechanisms (e.g., erosion) and/or human intervention may result in the repositioning of MEC.

For MC, a complete or potentially complete exposure pathway must include the following components: 1) a source (e.g., locations where MC are expected to be found); 2) an exposure medium (e.g., surface soil); 3) an exposure route (e.g., dermal contact); and 4) receptors (e.g.,

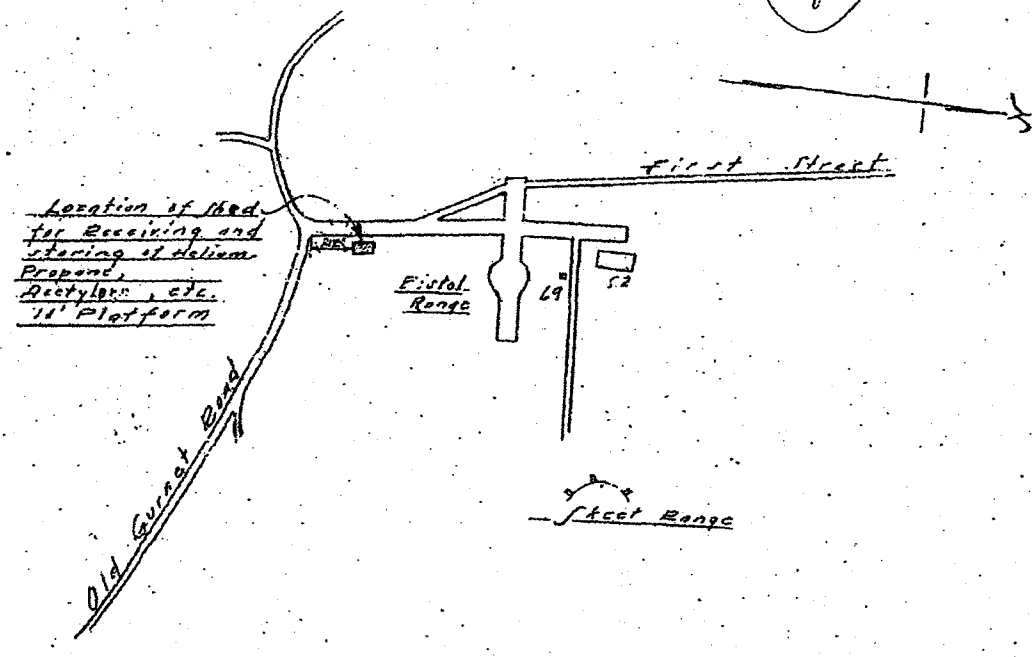
Figure 5.3-6: MC Exposure Pathway Analysis



SKEET RANGE
1957 DRAWING
FROM PA APPENDIX C-2

April 15, 1957

(10)



U.S. NAVAL AIR STATION BRUNSWICK, MAINE
PUBLIC WORKS DEPARTMENT

DESIGN DIV.
DRAWN BY PAD
TRACED BY
CHECKED BY
APPROVED
SUPERVISING ENGR.
DESIGN OFFICER

Shed for Receiving & Issuing of Volatile Gases

APPROVED _____ DATE _____
PUBLIC WORKS OFFICER

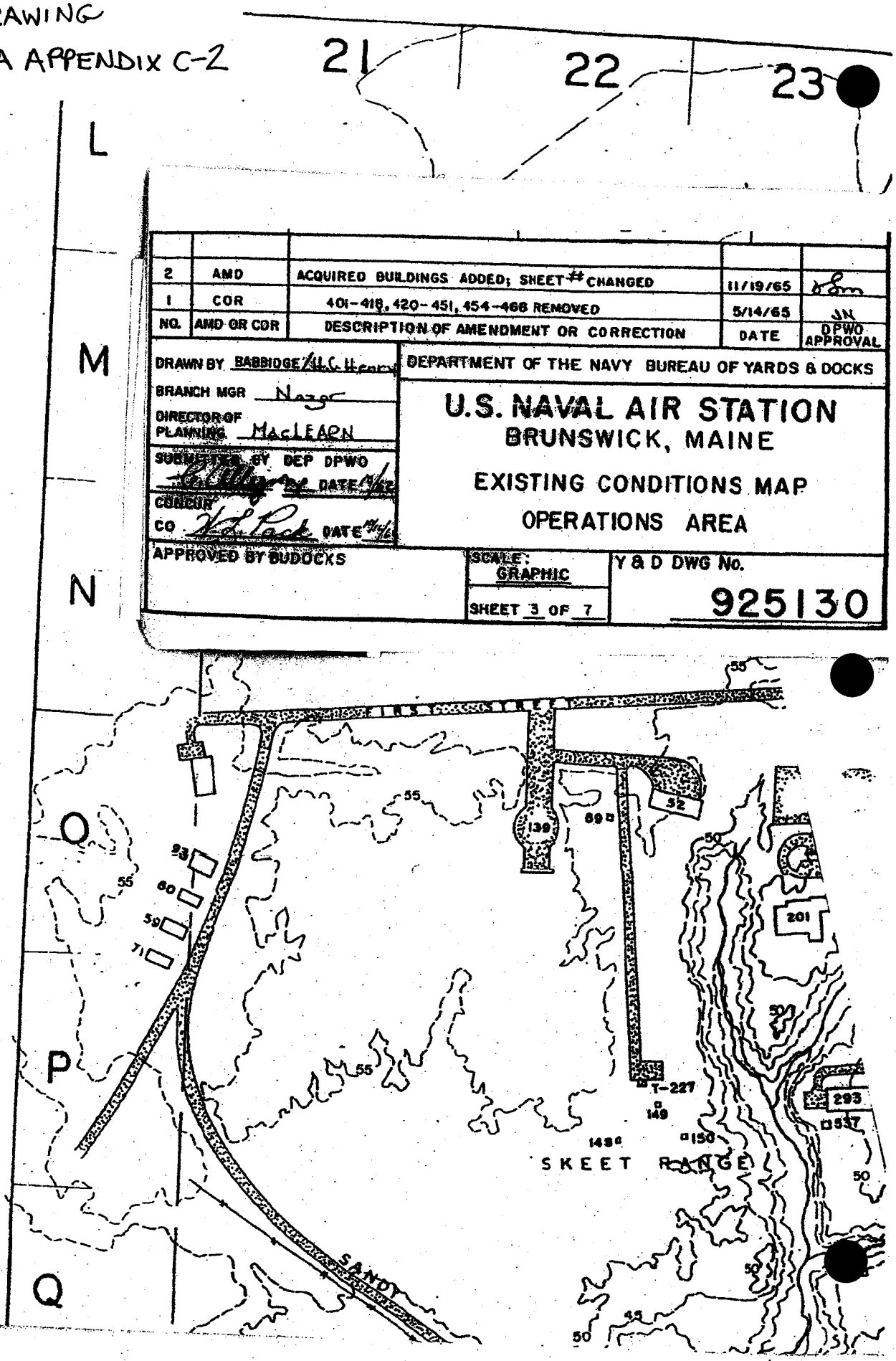
SATISFACTORY TO
TITLE
TITLE

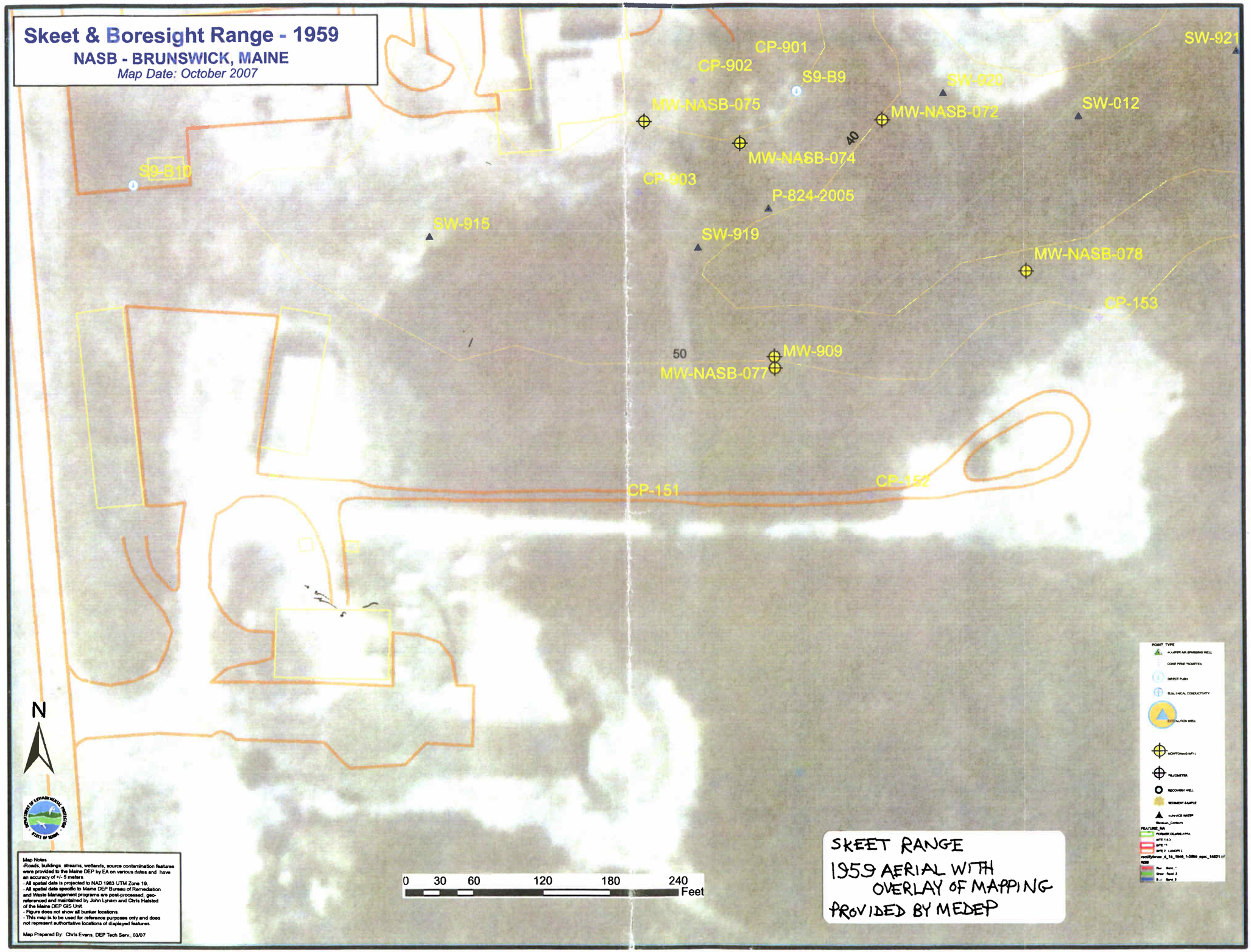
SCALE 1" = 400'-0"
SHEET NO. ____ OF ____
Proj. Iden. No. 14-57

RG 72, Butler
E.62B, Gen'l Corres.
1957, Box 196
NA/Brunswick

REPRODUCED AT THE NATIONAL ARCHIVES

SKEET RANGE
1962 DRAWING
FROM PA APPENDIX C-2




Skeet & Boresight Range - 1959
NASB - BRUNSWICK, MAINE
Map Date: October 2007
CP-901
CP-902
S9-B9
SW-920
SW-921
SW-012
MW-NASB-075
MW-NASB-072
40
MW-NASB-074
S9-B10
CP-903
P-824-2005
SW-915
SW-919
MW-NASB-078
CP-153
50
MW-909
MW-NASB-077
CP-151
CP-152
N
0 30 60 120 180 240
Feet
Map Notes
-Roads, buildings, streams, wetlands, source contamination features were provided to the Maine DEP by EA on various dates and have an accuracy of +/- 5 meters.
- All spatial data is projected to NAD 1983 UTM Zone 19.
- All spatial data specific to Maine DEP Bureau of Remediation and Waste Management programs are post-processed, geo-referenced and maintained by John Lynam and Chris Halsted of the Maine DEP GIS Unit.
- Figure does not show all bunker locations.
- This map is to be used for reference purposes only and does not represent authoritative locations of displayed features.
Map Prepared By: Chris Evans, DEP Tech Serv., 03/07
SKEET RANGE
1959 AERIAL WITH
OVERLAY OF MAPPING
PROVIDED BY MEDEP

Draft
Site 9
Monitoring Event 33 Report
September 2008

Contract No. N62472-02-D-0810
Contract Task Order No. 007

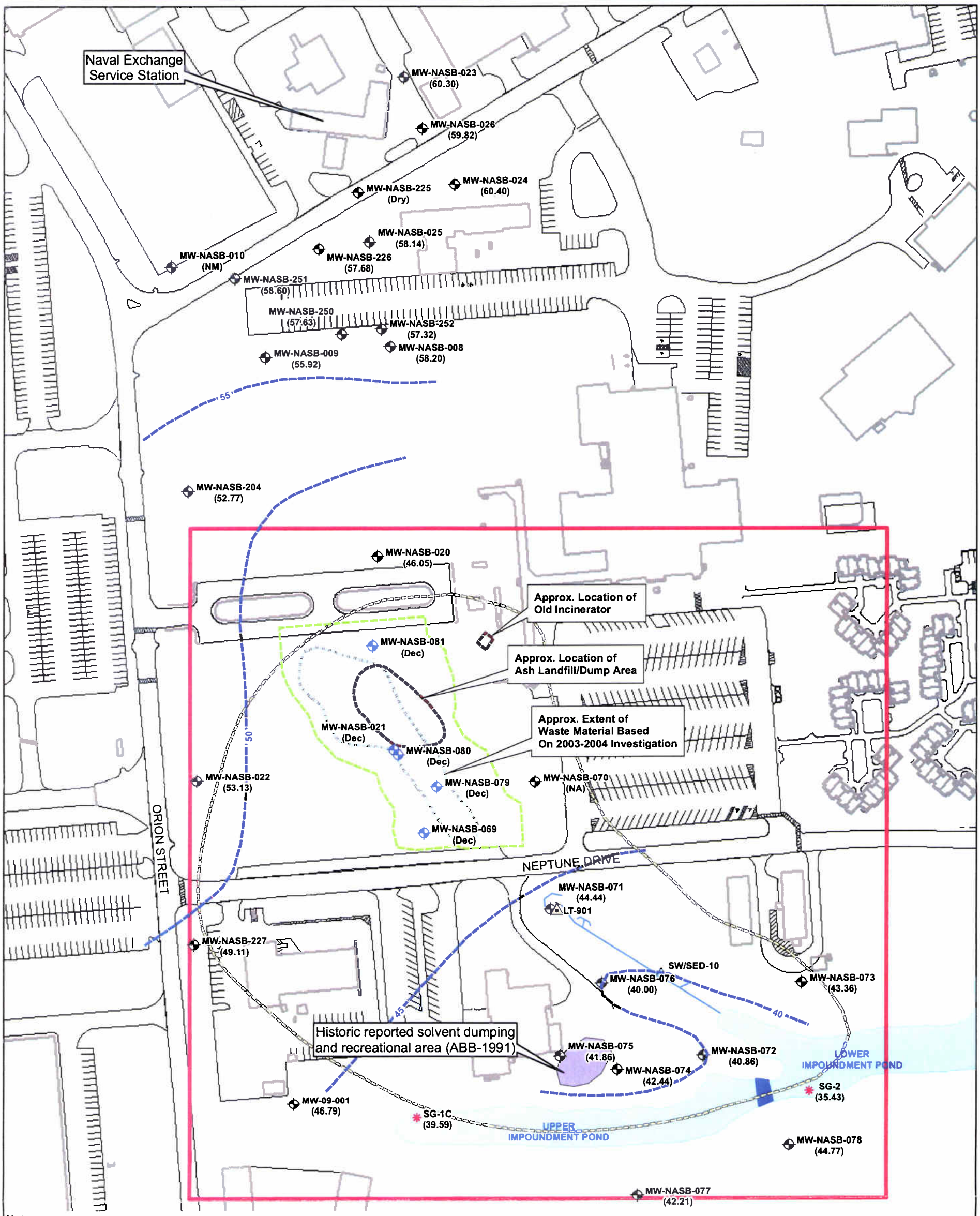
Naval Air Station
Brunswick, Maine

Prepared by:

ECC
33 Boston Post Road West, Suite 340
Marlborough, MA 01752

Prepared for:
Department of the Navy
Naval Facilities Engineering Command
BRAC Program Management Office - Northeast
4911 South Broad Street
Philadelphia, Pennsylvania 19112-1303

December 2008

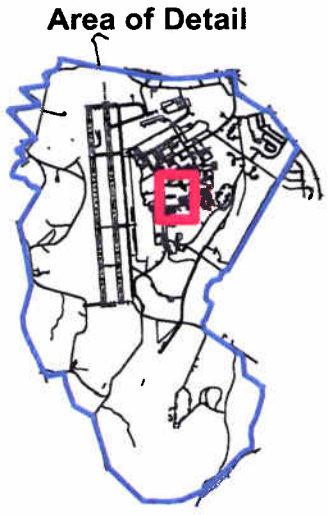


Note:
1. MW-NASB-021, MW-NASB-069, MW-NASB-079, MW-NASB-080, and MW-NASB-081 have been decommissioned and/or destroyed as a result of Removal Action (RA).
2. MW-NASB-070 was buried under a soil pile during the September 2008 sampling event and therefore inaccessible for sampling.
3. NA = Not Available

Contract No.	N62472-02-D-0810
Description	NAS Brunswick, ME
Coordinate system	NAD 1983, UTM, Zone 19N in meters
Sources	Naval Base Boundary provided by Navy.

Date	5-DEC-2008	Rev.	Date	App. By
DB	C. Guido			
CB	M. Johanson			
AB				

Area of Detail



Legend

- 45 — Inferred Groundwater Contour (ft MSL) (Groundwater Elevation, ft MSL)
- MW-NASB-077 (43.40) — Monitoring Well (Groundwater Elevation, ft MSL)
- MW-NASB-021 (Dec) — Decommissioned/Destroyed by Removal Action
- SG-1 (NA) — Stream Gauge
- LT-901 — Leachate Seep Station
- SW/SED-10 — Surface Water/Sediment Station
- Approx. Excavation Limit - June 2008
- Approx. Boundary of Site 9
- Dam
- Institutional Control Boundary
- Impoundment Pond

Figure 1-3

Site 9 Interpreted Groundwater Surface Contour Map September 2008

Naval Air Station Brunswick, Maine

ECC GIS Server
C:\NAVY_GIS\TO07_Brunswick\Site9\MapDocuments\September_2008\Site9_Fig1-3_Sept08_GWcontours.mxd

0 50 100 200 Feet

C-3 TOPSHAM ANNEX SKEET RANGE

TOPSHAM ANNEX SKEET RANGE
(SELECTED PAGES)

FINAL
PRELIMINARY ASSESSMENT
NAVAL AIR STATION BRUNSWICK, TOPSHAM ANNEX, MAINE

September 2005

Prepared for:

Naval Facilities Engineering Command
Engineering Field Activity, Northeast
10 Industrial Highway
Mail Stop #82
Lester, PA 19113-2090

Prepared by:

MALCOLM PIRNIE, INC.
300 E. Lombard Street
Suite 610
Baltimore, Maryland 21202

Table 5-1: Conceptual Site Model Information Profiles – Topsham Annex Skeet Range		
Profile Type	**Information Needs**	**Preliminary Assessment Findings**
Range/Site Profile	**Installation Name**	NASB, Topsham Annex
	Installation Location	Topsham, Sagadahoc County, Maine
	Range/Site Name	Topsham Annex Skeet Range
	Range/Site Location	The Topsham Annex Skeet Range is located in the northern portion of Topsham Annex along the property line and east of the Mt. Ararat Middle School athletic field. The majority of the skeet range extends north of the property line onto private property.
	Range/Site History	The range was identified on historical maps dated from 1957 through 1980. The skeet range was reportedly used by the Air Force during their occupation of Topsham Annex (approximately 1957 to 1970).
	Range/Site Area and Layout	The skeet range is shown on the historic maps as a rectangle with three small squares inside the rectangle. It is assumed that these small squares represent the low and high skeet houses and trap house; however, the direction of fire and the firing arc were not indicated on the maps. Based on observations during the visual survey, it appears that the direction of fire was toward the north. The skeet range, including SDZ, is approximately 29 acres, and approximately 27 acres are located outside of the installation.
	Range/Site Structures	There are no range-related structures currently at the site; however the historic maps indicate the presence of low and high skeet houses and a trap house. Upright wooden posts remain fixed in the ground at two locations within the site boundary. The site also overlaps military housing and an off-base trailer court.
	Range/Site Boundaries	N: Undeveloped woodland S: Recreational fields and undeveloped grassland W: Undeveloped woodland and residential property E: Residential property
	Range/Site Security	There are no access restrictions for the Topsham Annex or the Topsham Annex Skeet Range. There are no access restrictions to the off-base portion of the range.
Munitions/ Release	**Munitions Types**	12-gauge shotguns (small arms) were used at the range for skeet and trap shooting.

Table 5-1: Conceptual Site Model Information Profiles – Topsham Annex Skeet Range

Profile Type	Information Needs	Preliminary Assessment Findings
Profile	**Maximum Probability Penetration Depth**	The penetration depth of small arms on the range floor is one foot or less.
	MEC Density	Only small arms ammunitions were used at the site; therefore, there is no MEC density at the site.
	MEC Scrap/Fragments	No MEC scrap/fragments were found during the visual survey.
	Associated Munitions Constituents	MC may include lead, antimony, copper, zinc, arsenic, and PAHs from bullets, fragments, bullet jackets, and related sporting material (e.g., clay targets).
	Migration Routes/Release Mechanisms	Possible contaminant migration routes include soil surface runoff, groundwater discharge, and any future construction/excavation activities. The drainage ways on the off-base portion of the site also represent possible migration routes.
Physical Profile	**Climate**	Continental climate with four well-defined seasons. Highest temperatures occur in July (79 °F or higher). Coldest temperatures occur in January (21 °F or lower). Based on the climate type, wind and water erosion is common. Frost heave is also common.
	Topography	The elevation at the Topsham Annex Skeet Range is approximately 140 feet above sea level (asl). The site is relatively flat with a small incline to the south
	Geology	The Maine Geological Society has mapped the bedrock beneath the site as Mount Ararat, a member of the Cambrian/Ordovician Cushing Formation, a thinly bedded amphibolite and granofels.
	Soil	Soils in the vicinity of Topsham Annex belong to the Hollis-Sutton-Buxton Association. These soils are described as shallow to deep, medium textured and moderately coarse textured, well drained and moderately well drained, nearly level to steep soils.
	Hydrogeology	A shallow unconfined saturated aquifer lies beneath Topsham Annex. It is assumed that regional groundwater flows west and northwest toward the Cathance River; however, no investigations have been conducted at the site.
	Hydrology	Surface water drains toward the outfall and stream located at the western edge of the site.

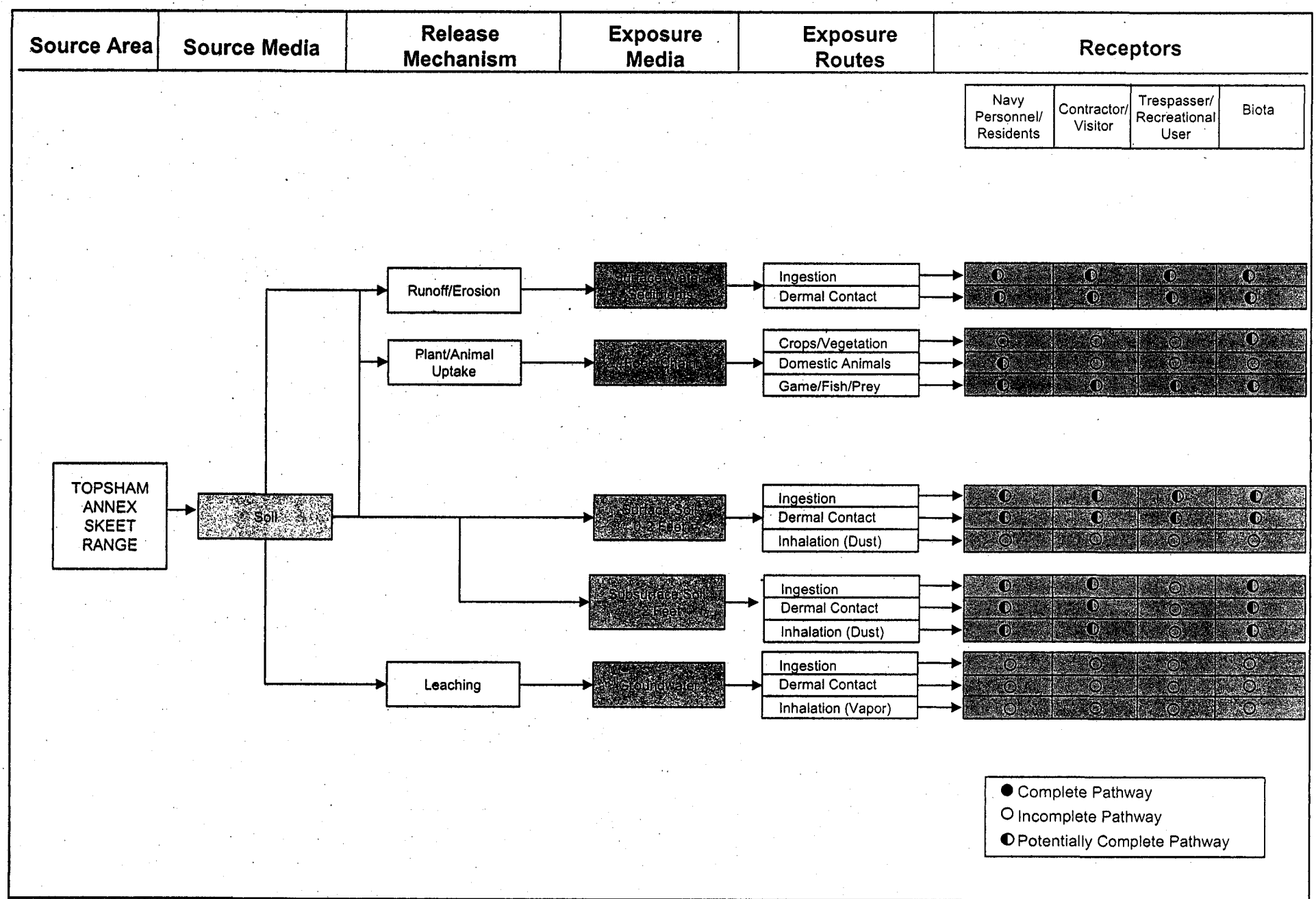
Table 5-1: Conceptual Site Model Information Profiles – Topsham Annex Skeet Range

Profile Type	Information Needs	Preliminary Assessment Findings
	Vegetation	The site is covered with high grass, shrubs, and a few trees.
Land Use and Exposure Profile	**Current Land Use**	The majority of the site is currently undeveloped. Small portions of the range are residential, and another small portion is used as a recreation field by the Mt. Ararat Middle School.
	Current Human Receptors	Navy personnel (military and civilian), residents, visitors, recreational users (including school children), and trespassers.
	Current Activities (frequency, nature of activity)	With the exception of the residences, there are no regular activities conducted at on-base portion of the site. Known activities on the off-base portion of the site include residences and a middle school.
	Potential Future Land Use	There is no foreseen change to current use of the on-base portion of the range. The future use of the off-base portion of the range is unknown.
	Potential Future Human Receptors	Navy personnel (military and civilian), residents, visitors, recreational users (including school children), trespassers, and contractors. These receptors would include construction workers (if intrusive work is necessary) and maintenance and operations workers (if the range use changes). With the exception of school children at the middle school, the potential future human receptors for the off-base portion of the range are unknown.
	Potential Future Land Use-Related Activities:	There are no foreseen changes in the land use-related activities at the on-base portion of the site. However, the Navy does plan to demolish the existing structures and construct new housing units on the same property. The land use-related activities for the off-base portion of the range are unknown.
	Zoning/Land Use Restrictions	The zoning districts for the Topsham Annex and adjacent properties are subject to town ordinances for use regulations and dimensional requirements.
	Demographics	The population of Sagadahoc County is just over 35,000 and the town of Topsham, including Topsham Annex, has an estimated population of approximately 9,100. There are 177 family housing units on Topsham Annex which are located east and south east of the former skeet range.

Table 5-1: Conceptual Site Model Information Profiles – Topsham Annex Skeet Range

Profile Type	Information Needs	Preliminary Assessment Findings
	Beneficial Resources	Evidence of wildlife activity was found on and around the Topsham Annex Skeet Range.
Ecological Profile	**Habitat Type**	The undeveloped site and the surrounding area contain the following habitats: Forest, Grassland, Streams, and wetlands.
	Degree of Disturbance	Minimal – With the exception of the residences, there are no regular activities conducted at the on-base portion of the site, and the residences occupy a small portion of the site. Unknown – Activities on the off-base portion of the site are unknown.
	Ecological Receptors	Biota includes indigenous species of mammals, amphibians, and birds.
	Federal Endangered Species:	None specifically known to be at site. However, species listed for the NASB are presented in Section 3.8.
	Federal Threatened Species:	None specifically known to be at site. However, species listed for the NASB are presented in Section 3.8.
	State Endangered Species:	None specifically known to be at site. However, species listed for the NASB are presented in Section 3.8.
	State Threatened Species:	None specifically known to be at site. However, species listed for the NASB are presented in Section 3.8.
	Other Ecological Receptors:	None observed or reported.
	Relationship of MEC/MC Sources to Habitat and Potential Receptors	There is no potential for MEC (small arms shotgun ammunition only) at the site. MC (including lead) could be at the site. Receptors may have direct or indirect contact with MC that exist in the environment or have been incorporated into the food chain.

A key element of the CSM is the exposure pathway analysis. For MEC, a complete or potentially complete exposure pathway must include the following components: 1) a source (e.g., locations where MEC are expected to be found); 2) access (e.g., controlled access, items on the surface or within the subsurface); 3) an activity (e.g., non-intrusive grounds maintenance or intrusive construction); and 4) receptors (e.g., Navy personnel, construction workers, recreational users, or



MALCOLM PIRNIE

Prepared for:

PRELIMINARY ASSESSMENT – CONCEPTUAL SITE MODEL
TOPSHAM ANNEX SKEET RANGE – MC EXPOSURE PATHWAY ANALYSIS
NASB TOPSHAM ANNEX, MAINE

MALCOLM PIRNIE, INC.

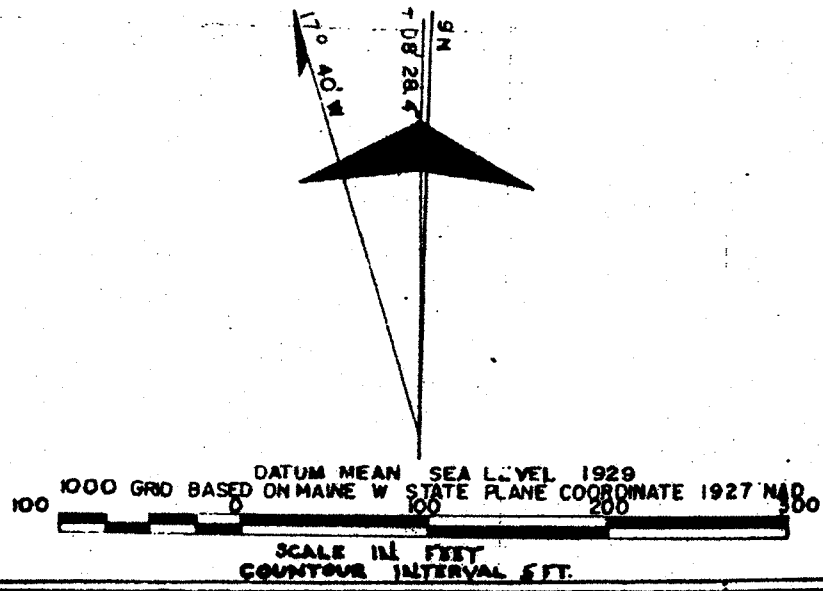
FIGURE 5.1-9 FINAL
SEPTEMBER 2005

TOPSHAM ANNEX
SKEET RANGE
1964 AIR FORCE DRAWING
FROM MALCOLM ARNIE PA - APPX B

10. —X—X— EXISTING FENCE
11. —XX—XX— PROPOSED FENCE

NATURAL FEATURES

12. EXISTING CONTOURS
13. APPROXIMATE CONTOURS
14. EXISTING TREE COVER
15. LAKE OR POND
16. STREAMS OR RIVERS
17. DRAINAGE CHANNEL OR DITCH
18. DAM
19. SWAMP



REV	DATE	DESCRIPTION	INITIAL
	28/4/80	[illegible] BLDG NO. 73	DJA
1	[illegible]	[illegible] NO. 27	DJA

MASTER PLAN

TOPSHAM AIR FORCE STATION

BRUNSWICK MAINE

DRAFTSMAN: JADLOWSKI

SCALE: 1" = 100'

DATE: 28 MARCH 1980

150

160

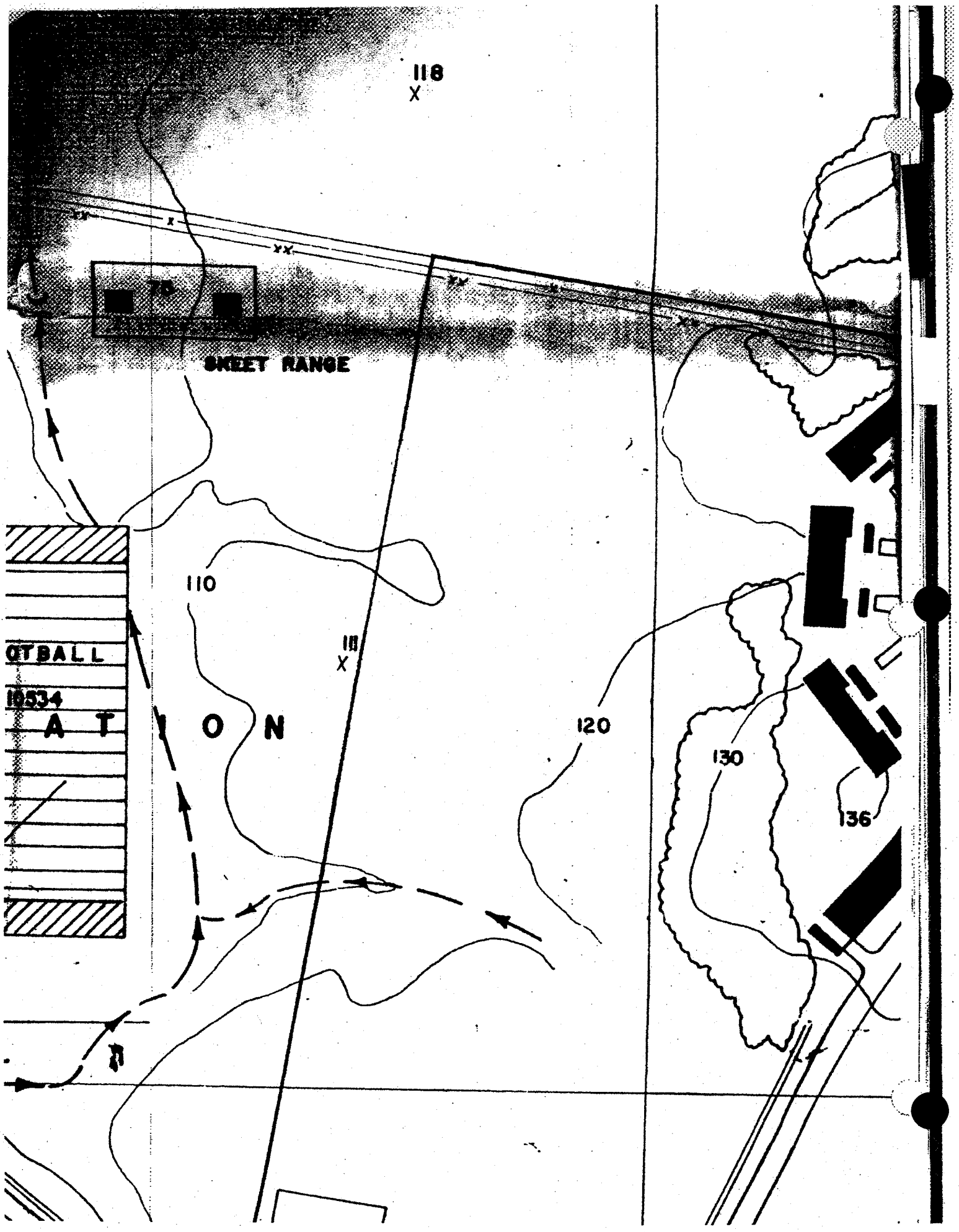
170

N. 405,000

E. 555,000

G 342, USAF Cmds, Acts; 10 rgs
Gen Corr, Jan-Dec, 1970
(ACC 73-A-3205)
Box 39
Fed Rec Ctr, Suitland, Md.

EXHIBIT "A"


118
SKEET RANGE
110
111
OTBALL
10534
A T
O N
120
130
136

2004 Investigation Report
Topsham Annex,
Naval Air Station, Topsham, Maine

Admin Record
#938

Prepared for

Department of the Navy
Engineering Field Activity Northeast
Naval Facilities Engineering Command
North Loop & American Way, Building G
Code 182
Lester, Pennsylvania 19113-2090

Prepared by

ECC
50 D'Angelo Drive
Marlborough, Massachusetts 01752

and

EA Science and Technology
3 Washington Center
The Maple Building
Newburgh, New York 12550

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3. SITE-SPECIFIC INVESTIGATIONS

The groundwater and soil investigations at the Topsham Annex were separated into 8 AOCs:

- Area A (Buildings 337 and 338)
- Area B (Former Building 369)
- Area C (Building 378)
- Areas D (Buildings 1099 and 1114)
- Area E (Building 1108)
- Top – 01 (Filled area)
- Top – 02 (Former oil/water separator)
- Building 380 (Former skeet range).

3.1 AREA A (BUILDINGS 337 AND 338)

Building 337 was used as a vehicle service center (auto hobby shop and car wash) and service station, and Building 338 was used for vehicle storage. Building 337 had a single pump island served by gasoline underground storage tanks (USTs) located to the northeast of the building. Building 337 also had a hydraulic lift and steam pits located within the building footprints, and it was also used as a fire station in the past. To the south of Building 337, a UST and oil/water separator were located. Both buildings previously were served by fuel oil USTs that supplied fuel oil for the heating systems. There is a surface water drain line that travels through this AOC to the northern portion of this area (Figure 3), located north of Building 337 and north and east of Building 338. Currently, these buildings are used for storage by the U.S. Marine Corps.

3.1.1 2004 Investigation Activities

The following investigation activities were completed at Area A from 23 to 24 June, and on 1 July 2004:

- Thirteen direct-push soil borings (labeled BLDG337-01 through BLDG337-07 and BLDG338-01 through BLDG338-06) were completed at the locations shown on Figure 3. Table 1 provides a summary of the samples collected in Area A.
  - Headspace screening was conducted during each soil boring installation with a Foxboro TVA-1000 PID/FID. The PID headspace concentrations ranged from 0 to 10.4 ppm.
  - A total of 26 soil samples were collected at Area A. One soil sample from boring BLDG338-06; 2 samples from boring BLDG337-01, BLDG337-03 through BLDG337-07, BLDG338-01 through BLDG338-05; and 3 samples from boring BLDG337-02.

3.5.6 Recommendations

After a review of historical information, previous investigations, and the 2004 investigation data, EA submits the following recommendations for Area E:

- The groundwater use restriction for potable and non-potable uses should remain in place even though the groundwater at the Topsham Annex and surrounding areas are not used as a water source.

- A focused soil removal should be completed to remove the impacted soils from the areas around 1114-02-GWI, 1114-04-GWI, and 1099-03-GWI.

3.6 TOP-01 (FILLED AREA)

This PAOEC was identified in the 1996 EBS report (Figure 18). The 1996 EBS report indicated that this area is located south of the former skeet range. The EBS provided limited specific site information for this area, other than to identify and recommend that additional investigation efforts be completed to assess this area of fill.

3.6.1 2004 Investigation Activities

The following investigation activities were completed at TOP-01 on 21 June 2004:

- Three direct-push soil borings (labeled TOP-01-01 through TOP-01-03) were advanced at the locations shown on Figure 18.

  - Headspace screening was conducted during each soil boring installation with a Foxboro TVA-1000 PID/FID. PID detections ranged from 0.0 to 124 ppm. FID detections ranged from 2.64 to 150 ppm.

  - A total of 6 soil samples were collected, 2 from each boring. The soil samples were collected at the groundwater interface and from the fill interval. Soil samples were submitted for laboratory analysis of VOCs by Method 8260B, SVOCs by Method 8270C, TPH-DROs by Maine HETL Method 4.1.25, TPH-GROs by Maine HETL 4.1.17, 8 RCRA metals by EPA Method 200/6000/7000 Series, and polychlorinated biphenyls (PCBs) by EPA Method 8082. The TOP-01 sample summary is provided in Table 6.

  - A total of 3 groundwater samples were collected, 1 from each boring. Groundwater samples were submitted for laboratory analysis of VOCs by EPA Method 8260B, SVOCs by EPA Method 8270C, TPH-DROs by Maine HETL Method 4.1.25, TPH-GROs by Maine HETL 4.1.17, and 8 RCRA metals by EPA Method 200/6000/7000 Series. The TOP-01 sample summary is provided in Table 6.

3.6.2 Deviations from the Work Plan

No deviations from the Work Plan occurred at TOP-01.

3.6.3 Summary of Previous Investigations Results and Findings

TOP-01 was a reported area where fill material (i.e., construction rubble, extra soil) was dumped and buried. No further information was available on TOP-01.

3.6.4 Results of the 2004 Field Investigation

A summary of groundwater and soil samples collected is provided in Table 6 and shown on Figure 19. Analytical results for groundwater and soil are summarized in Appendix F, Table 6. The analytical data report (Form 1s) and the chain-of-custody forms are provided in Appendix H.

Groundwater sample results indicated the following:

- No groundwater VOCs were detected above the Maine MEG or Federal MCL at TOP-01.
- One SVOC (bis[2-ethylhexyl] phthalate) was detected above the Maine MEG (25 µg/L) in groundwater at a concentration of 64 µg/L at TOP-01-02, although this is considered to be a sampling artifact.
- TPH-DROs were detected above the Maine MEG (25 µg/L) in 2 of 3 groundwater sample locations at TOP-01-01 at 160B µg/L and TOP-01-02 at 130B µg/L.
- TPH-GROs were not detected above the Maine MEG (50 µg/L) at TOP-01.
- Three RCRA 8 Metals (arsenic, chromium, and lead) were detected in groundwater above the Maine MEG and/or Federal MCL at TOP-01. Arsenic was detected above the Federal MCL (1 µg/L) at TOP-01-01 (7.3B* µg/L) and above both the Maine MEG (10 µg/L) and Federal MCL (1 µg/L) at TOP-01-02 (10.1 µg/L). Chromium was detected above the Maine MEG (40 µg/L) at TOP-01-02 (42.2 µg/L). Lead was detected above both the Maine MEG (10 µg/L) and Federal MCL (15 µg/L) at TOP-01-02 (22.9 µg/L).

Soil sample results indicated the following:

- No VOCs were detected in the soil above Maine RAGs at TOP-01.
- SVOCs were detected at 3 of 6 soil sample locations (TOP-01-02-FILL, TOP-01-02-FILL, and TOP-01-03-FILL). There are no Maine RAGs for SVOCs. However, the toxic equivalent for benzo(a)pyrene of 2,000 µg/kg for residential use was used for comparison (Table 6).

- TPH-DROs were detected above the Maine Petroleum Remediation Goal (10,000 µg/kg) at 5 of 6 soil sample locations with concentrations ranging from 22,000 µg/kg to 290,000 µg/kg (TOP-01-01-FILL at 40,000 µg/kg, TOP-01-01-GWI at 27,000 µg/kg, TOP-01-02-FILL at 180,000 µg/kg, TOP-01-03-GWI at 22,000 µg/kg, and TOP-01-03-FILL at 290,000 µg/kg).

- TPH-GROs were detected above the Maine Petroleum Remediation Goal (10,000 µg/kg) at 1 of 6 soil sample locations (TOP-01-02-GWI at 18,000 µg/kg).

- One RCRA 8 Metal (arsenic) was detected above the Maine RAGs (10,000 µg/kg) at 1 of 6 soil sample locations (TOP-01-03-GWI at 11,700 µg/kg).

- One PCB (Aroclor-1260) was detected in soil at TOP-01-03-FILL at a concentration of 15J µg/kg. However, the residential toxic equivalent of 2,200 µg/kg was used for comparison (Table 6).

EA collected samples for quality assurance and quality control purposes (field duplicate, rinsate, and source water samples) for soil and groundwater samples.

Contamination detected in the quality control blanks is discussed in Appendix G.

3.6.5 Conclusions

TOP-01 is a suspected area where debris was dumped and buried. It is unknown specifically what was disposed of in this area or the dates of use (assumed to be filled with construction debris, extra soil). It is likely that the groundwater and soil impacts observed are from disposal activities undertaken by the Navy. The conclusions provided in this section, and subsequent recommendations, are based on the assumed regional groundwater flow direction and information on groundwater flow from previous investigations (i.e., "the presumed" direction noted in the text). The following conclusions are based on historical information, previous investigations, and results for the 2004 investigation at TOP-01:

- SVOCs were detected at 3 of 6 soil sample locations (TOP-01-02-FILL, TOP-01-02-FILL, and TOP-01-03-FILL). SVOC detections in soil occurred at "fill" sample locations.

- TPH-DROs were detected above the Maine Petroleum Remediation Goal (10,000 µg/kg) in soil at 5 of 6 locations. The highest concentrations were found in "fill" sample locations.

- One PCB (Aroclor-1260) was detected in soil at TOP-01-03-FILL, a "fill" sample location.

- The detections of the SVOCs, TPH-DRO, and PCBs in the groundwater at TOP-01 are most likely contamination from the various debris dumped and buried at the site.

- The concentrations of arsenic, chromium, and lead in groundwater do not appear to be caused by Navy activity on the site based on historical information provided to EA. Potential nearby sources of lead contamination include a former indoor firing range and an abandoned skeet range. However, concentrations of metals in the soil and groundwater do not suggest these sources have impacted this site, as the metal levels are relatively consistent with concentrations at other areas of the Topsham Annex. This will be confirmed during the upcoming investigation planned for the Skeet Range site. The detections of arsenic in soil do not appear to be caused by Navy activity on the site based on historical information provided to EA.

3.6.6 Recommendations

After a review of historical information, previous investigations, and the 2004 investigation data, EA submits the following recommendations for TOP-01:

- A groundwater use restriction for potable and non-potable uses is placed on the site even though the groundwater at the Topsham Annex and surrounding areas are not used as a water source.

- Temporary well points should be installed to determine site-specific groundwater flow.

- Additional borings should be completed in the TOP-01 area to fully delineate the SVOC, TPH-DRO, and PCB contamination in the soil.

3.7 TOP-02 (FORMER OIL/WATER SEPARATOR)

This former oil/water separator was identified in the 1996 EBS report as a PAOEC (Figure 20). Although no closure documentation was found during the 1996 EBS for this PAOEC, the 1999 EBS Addendum noted (visual observation by field personnel) that the former oil/water separator appeared to have been filled with gravel. No sampling of soils and/or groundwater at this PAOEC has been conducted to date. Although the 1999 EBS noted no evidence of release, the proposed boring and sampling are intended to determine if contamination is present, or a release occurred at the former oil/water separator.

3.7.1 2004 Investigation Activities

The following investigation activities were completed at TOP-02 on 21-22 June 2004:

- Three direct-push soil borings (labeled TOP-02-01 through TOP-02-03) were completed at the locations shown on Figure 20.

— Headspace screening was conducted during each soil boring installation with a Foxboro TVA-1000 PID/FID. PID detections were 0.0 ppm. FID detections ranged from 5.21 to 24.32 ppm.

— A total of 3 soil samples were collected, 1 from each boring. The soil samples were collected at the groundwater interface. Soil samples were submitted for laboratory analysis of TPH-DROs by Maine HETL Method 4.1.25, TPH-GROs by Maine HETL 4.1.17, and 8 RCRA metals by EPA Method 200/6000/7000 Series (Table 7).

— A total of 3 groundwater samples were collected, 1 from each boring. Groundwater samples were submitted for laboratory analysis of TPH-DROs by Maine HETL Method 4.1.25, TPH-GROs by Maine HETL 4.1.17, and 8 RCRA metals by EPA Method 200/6000/7000 Series (Table 7).

3.7.2 Deviations from the Work Plan

No deviations from the Work Plan occurred at TOP-02.

3.7.3 Summary of Previous Investigations Results and Findings

TOP-02 (former oil/water separator) was identified in the November 1996 EBS by HRP as an AOC in need of further investigation.

3.7.4 Results of the 2004 Field Investigation

A summary of groundwater and soil samples collected is provided in Table 7 and shown on Figure 21. Analytical results for groundwater and soil are summarized in Appendix F, Table 7. The analytical data report (Form 1s) and the chain-of-custody forms are provided in Appendix H.

Groundwater sample results indicated the following:

- TPH-DROs were detected at or above the Maine MEG (50 µg/L) at 3 of 3 groundwater sample locations (TOP-02-01 at 89B µg/L, TOP-02-02 at 60B µg/L, and TOP-02-03 at 50B µg/L).

- TPH-GROs were not detected in groundwater above the Maine MEG (50 µg/L) at TOP-02.

- No RCRA 8 Metals were detected in groundwater above Maine MEG or Federal MCL at TOP-02.

Soil sample results indicated the following:

- TPH-DROs were not detected in soil above the Maine Petroleum Goal (10,000 μg/kg).
- TPH-GROs were not detected in soil above the Maine Petroleum Goal (10,000 μg/kg).
- No RCRA 8 Metals were in soil detected above Maine RAGs at TOP-02.

EA collected samples for quality assurance and quality control purposes (field duplicate, rinsate, and source water samples) for soil and groundwater samples.

Contamination detected in the quality control blanks is discussed in Appendix G.

3.7.5 Conclusions

TOP-02 is the location of a former oil/water separator. The conclusions provided in this section, and subsequent recommendations, are based on the assumed regional groundwater flow direction and information on groundwater flow from previous investigations (i.e., "the presumed" direction noted in the text). The following conclusions are based on historical information, previous investigations, and the results for the 2004 investigation at TOP-02:

- TPH-DROs were detected in groundwater at or slightly above Maine MEGs at 3 locations. TPH-DROs were not detected in soil above the Maine Petroleum Remediation Goal (10,000 μg/kg). These detections are likely residual low-level contamination from the oil/water separator.
- No concentrations of TPH-GRO or RCRA-8 metals were detected in groundwater or soil above the applicable standard: Maine MEG, Federal MCL, or Maine Petroleum Remediation Goal.

3.7.6 Recommendations

After a review of historical information, previous investigations, and the 2004 investigation data, EA submits the following recommendations for TOP-02:

- A groundwater use restriction for potable and non-potable uses is placed on the site even though the groundwater at the Topsham Annex and surrounding areas is not used as a water source.

3.8 BUILDING 380 (FORMER SKEET RANGE)

Building 380 was formerly located at a skeet range at the Topsham Annex (Figure 18). Currently, the area of the former skeet range is overgrown with vegetation; however, there are three posts in the area that are reportedly the launching points of the skeet targets. The field personnel made every effort to determine where the firing line is to identify the area most likely

to be the shotfall area. As reported in the January 2003 Interstate Technology Regulatory Cooperation's Small Range Guidance Document, typical lead skeet loads can range as far out as 680 ft from the firing line.

3.8.1 2004 Investigation Activities

The following investigation activities were completed at Building 380 on 21 June 2004:

- Three composite soil samples (labeled SS-01-0604 through SS-03-0604) were collected at locations shown on Figure 18. The locations selected were chosen from what is believed to be the former range floor (targeting the shotfall area of the range floor) of the skeet range. No skeet shot was encountered, however, skeet pigeon chips were found, indicating samples were collected in a location where skeet shot could have fallen.

  — Each composite soil sample was collected from 3 distinct sample locations. Each composite soil sample area consisted of 3 distinct sample locations, which were combined into 1 composite sample (i.e., composite by area). At each sample location, soil was collected from 0 to 12 in. below the ground surface with a stainless steel trowel and transferred into a stainless steel mixing bowl. The composite soil sample was mixed in a stainless steel bowl to homogenize the entire sample. After mixing was complete, a portion of the soil was transferred from the bowl to the sample container using a stainless steel trowel and/or scoop. Soil samples were submitted for laboratory analysis of lead by EPA Method 3050B/6010B (Table 8).

3.8.2 Deviations from the Work Plan

No deviations from the Work Plan occurred at Building 380.

3.8.3 Summary of Previous Investigations Results and Findings

Building 380, the former skeet range, was identified in the November 1996 EBS by HRP as an AOC in need of further investigation.

3.8.4 Results of the 2004 Field Investigation

A summary of soil samples collected is provided in Table 8. Analytical results for soil are summarized in Appendix F, Table 8. The analytical data report (Form 1s) and chain-of-custody forms are provided in Appendix H.

- Lead was not detected above the Maine RAGs (375,000 µg/kg) in the 3 composite soil samples collected at Building 380 (SS-01-0604 at 202,000 µg/kg, SS-02-0604 at 82,900 µg/kg, and SS-03-0604 at 49,900 µg/kg).

EA collected samples for quality assurance and quality control purposes (field duplicate, rinsate, and source water samples) for soil samples.

3.8.5 Conclusions

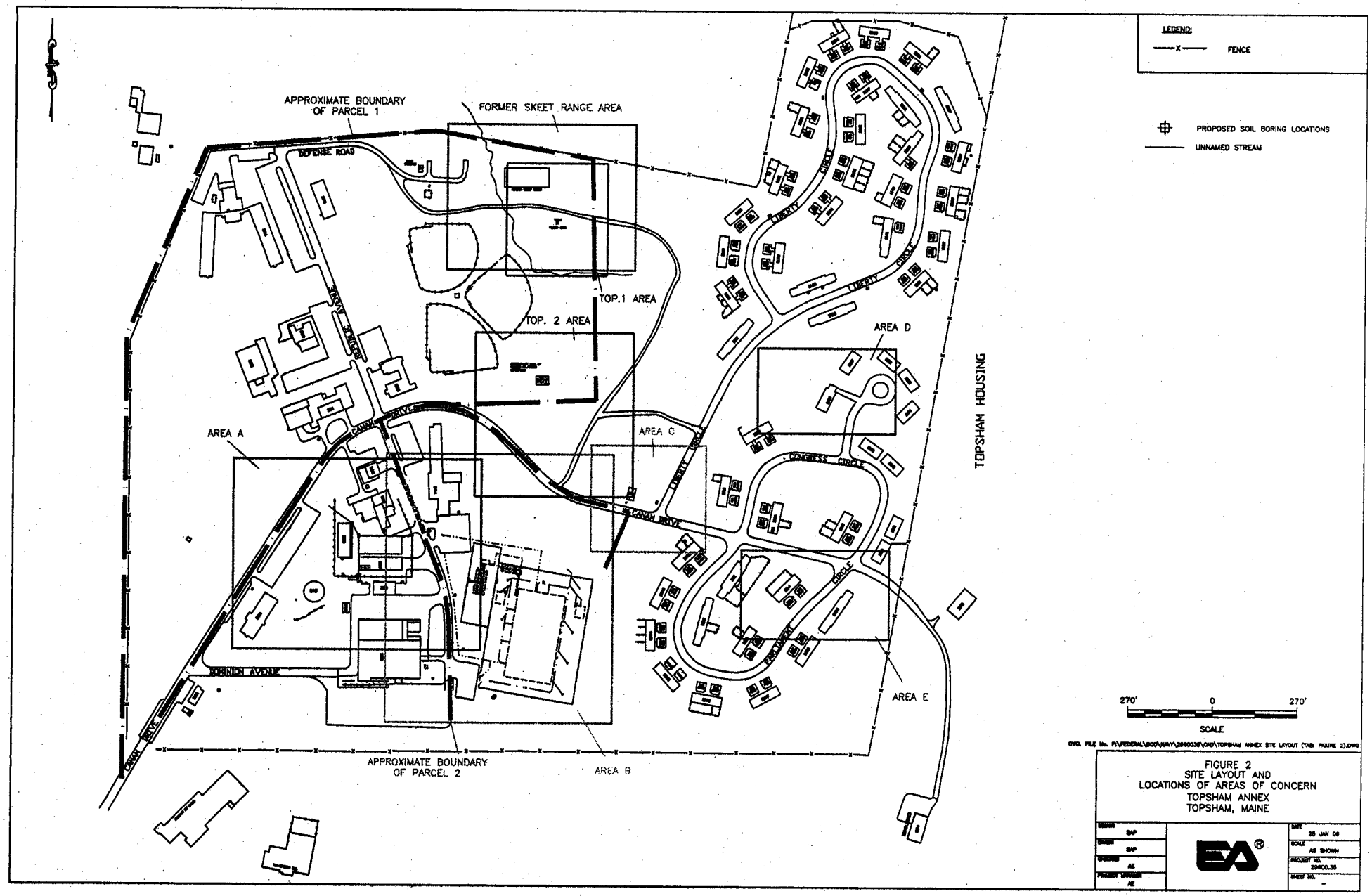
The following conclusions are based on historical information, previous investigations, and the results for the 2004 investigation at Building 380:

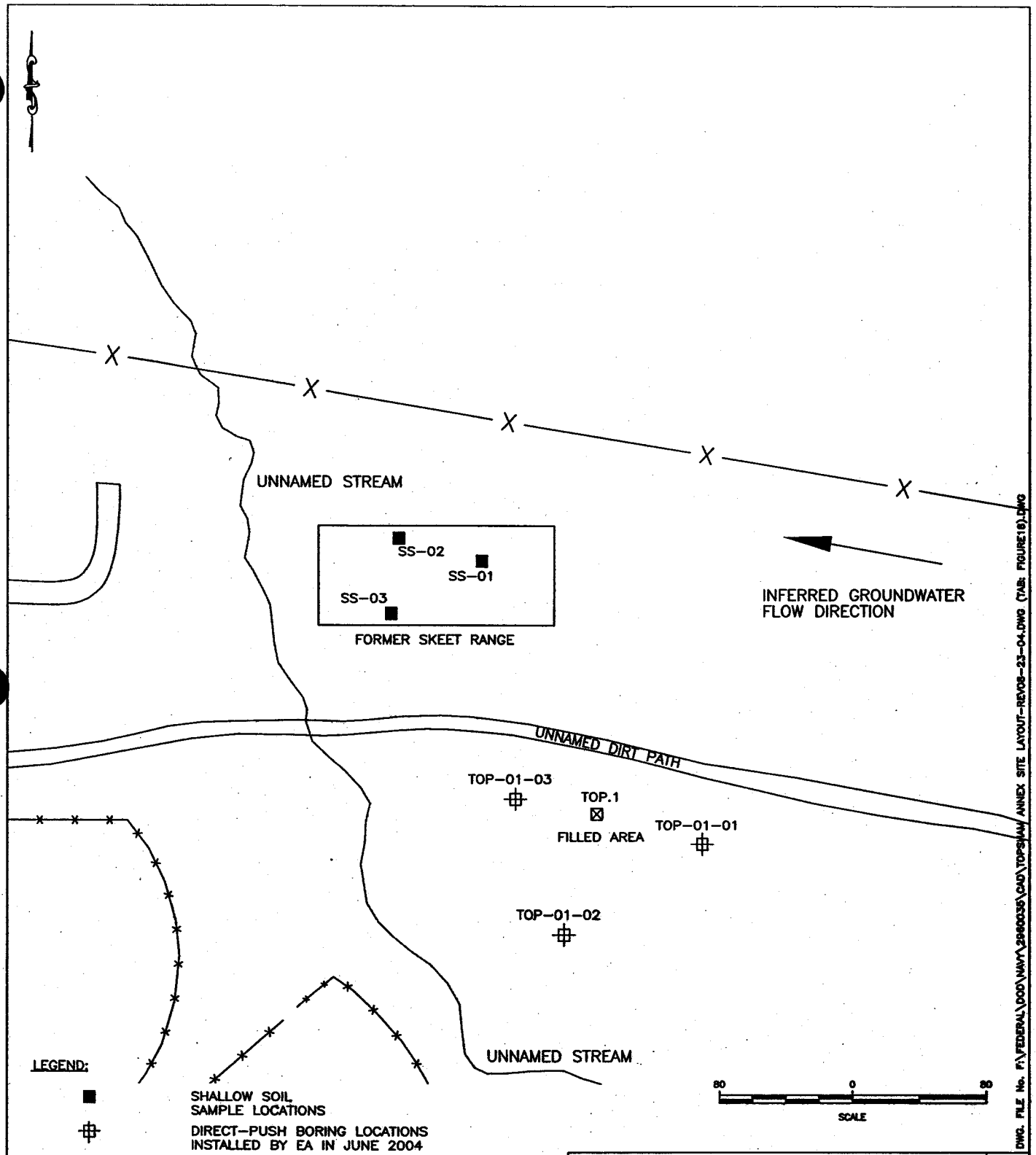
- Lead was detected in soil at all 3 sample locations at Building 380. The detections of lead are below the Maine RAGs.
- Since the beginning of this study, a contract has been awarded for a Preliminary Assessment of the Skeet Range under the Munitions Response Program.

Building 380 is the location of a former skeet range. No elevated concentrations of lead were detected.

3.8.6 Recommendations

Recommendations for the site are contained in the Preliminary Assessment Report.


APPROXIMATE BOUNDARY OF PARCEL 1
FORMER SKEET RANGE AREA
DEFENSE ROAD
TOP.1 AREA
TOP. 2 AREA
AREA A
AREA B
AREA C
AREA D
AREA E
CANAM DRIVE
DOMINION AVENUE
LIBERTY CIRCLE
CONGRESS CIRCLE
PARLIAMENT CIRCLE
TOPSHAM HOUSING
APPROXIMATE BOUNDARY OF PARCEL 2
LEGEND:
FENCE
PROPOSED SOIL BORING LOCATIONS
UNNAMED STREAM
270'
0
270'
SCALE
DWG. FILE No. P:\FEDERAL\2007\NAVY\2840038\CAD\TOPSHAM ANNEX SITE LAYOUT (TAB FIGURE 2).DWG
FIGURE 2
SITE LAYOUT AND
LOCATIONS OF AREAS OF CONCERN
TOPSHAM ANNEX
TOPSHAM, MAINE
DRAWN SAP
DESIGN SAP
CHECKED AE
PROJECT MANAGER AE
EA®
DATE 25 JAN 08
SCALE AS SHOWN
PROJECT NO. 29400.38
SHEET NO. -



LEGEND:

- SHALLOW SOIL SAMPLE LOCATIONS
- DIRECT-PUSH BORING LOCATIONS INSTALLED BY EA IN JUNE 2004
- TOP.1 AREA
- CHAIN LINK FENCE
- INFERRED GROUNDWATER FLOW DIRECTION IS BASED ON REGIONAL GROUNWATER FLOW

NOTES:

1. THE LOCATION OF FORMER SKEET RANGE WAS TAKEN FROM AN OLD VICINITY PLAN AND IS APPROXIMATE.
2. FOSTER WHEELER ENVIRONMENTAL CORPORATION, FINAL REMOVAL ACTION TOPSHAM ANNEX PARCEL 2, TOPSHAM MAINE, MAY 2001.
3. INFERRED GROUNDWATER FLOW DIRECTION IS BASED ON REGIONAL GROUNDWATER FLOW.

FIGURE 18
TOP-01 (FILLED AREA) AND
FORMER BUILDING 380 (SKEET RANGE)
TOPSHAM ANNEX
TOPSHAM, MAINE

DESIGN SAP	EA®	DATE 25 JAN 06
DRAWN SAP		SCALE AS SHOWN
CHECKED AE		PROJECT NO. 29600.35
PROJECT MANAGER AE		SHEET NO. --

NOTES:

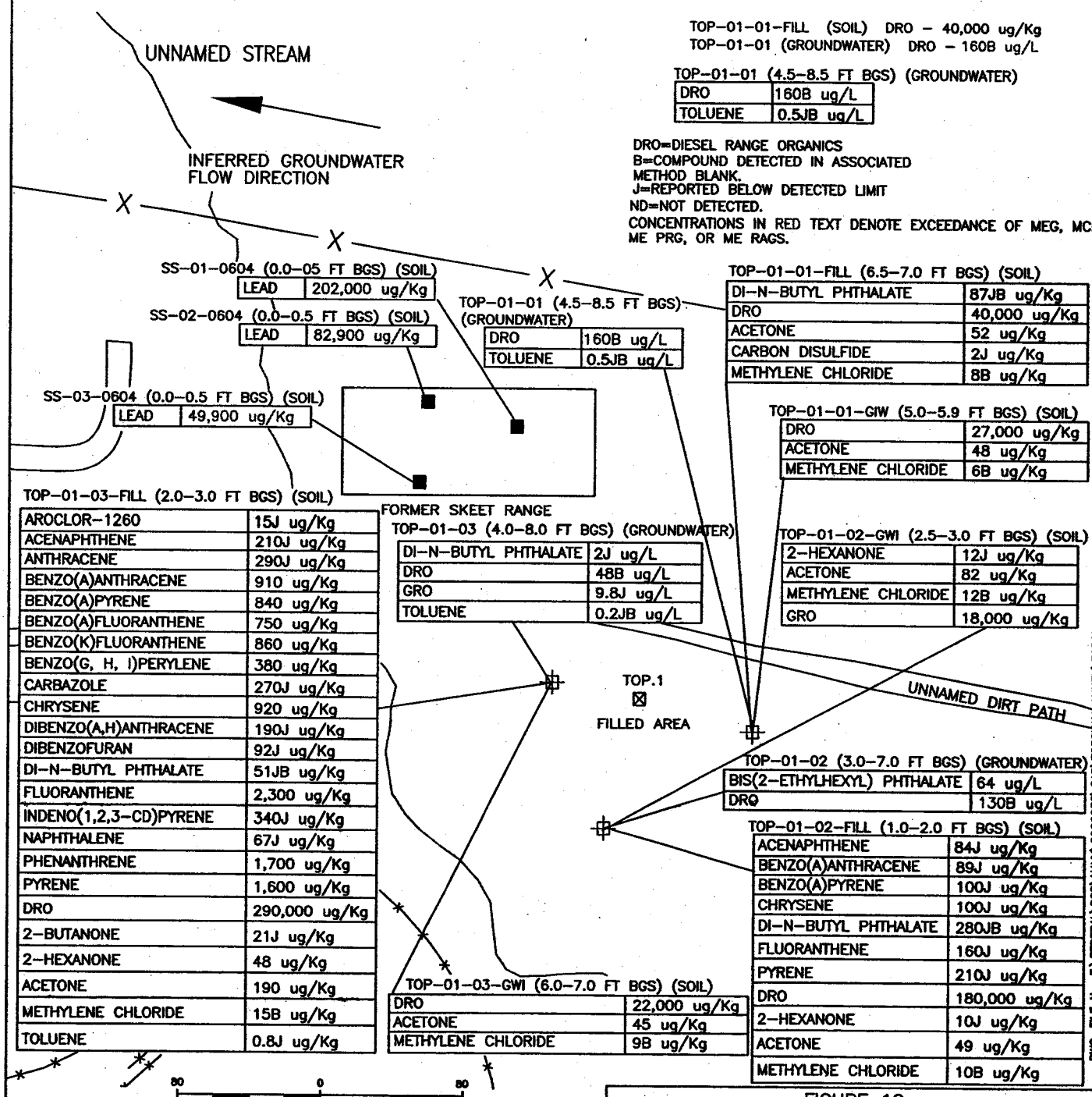
1. THE LOCATION OF FORMER SKEET RANGE WAS TAKEN FROM AN OLD VICINITY PLAN AND IS APPROXIMATE.
2. FOSTER WHEELER ENVIRONMENTAL CORPORATION, FINAL REMOVAL ACTION TOPSHAM ANNEX PARCEL 2, TOPSHAM MAINE, MAY 2001.
3. INFERRED GROUNDWATER FLOW DIRECTION IS BASED ON REGIONAL GROUNDWATER FLOW.

TOP-01-01-FILL (SOIL) DRO – 40,000 ug/Kg
TOP-01-01 (GROUNDWATER) DRO – 160B ug/L

TOP-01-01 (4.5-8.5 FT BGS) (GROUNDWATER)

DRO	160B ug/L
TOLUENE	0.5JB ug/L

DRO=DIESEL RANGE ORGANICS
B=COMPOUND DETECTED IN ASSOCIATED METHOD BLANK.
J=REPORTED BELOW DETECTED LIMIT
ND=NOT DETECTED.
CONCENTRATIONS IN RED TEXT DENOTE EXCEEDANCE OF MEG, MCL, ME PRG, OR ME RAGS.



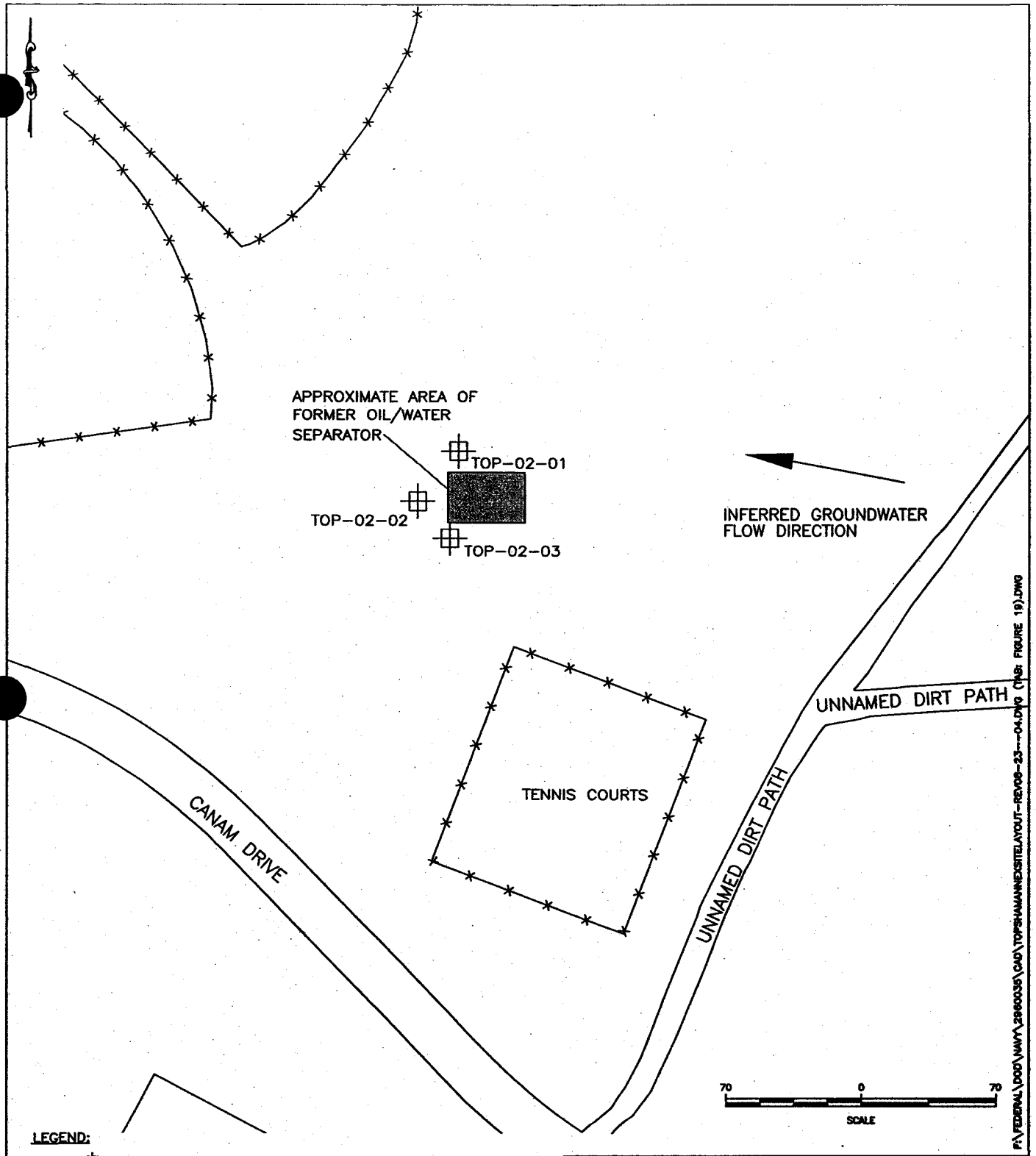
LEGEND:

- SHALLOW SOIL SAMPLE LOCATIONS
- DIRECT-PUSH BORING LOCATIONS INSTALLED BY EA IN JUNE 2004
- TOP.1 AREA
- CHAIN LINK FENCE
- INFERRED GROUNDWATER FLOW DIRECTION IS BASED ON REGIONAL GROUNWATER FLOW

FIGURE 19
TOP-01 (FILLED AREA) AND FORMER BUILDING 380 (SKEET RANGE) SOIL AND GROUNDWATER CONCENTRATIONS
TOPSHAM ANNEX
TOPSHAM, MAINE

DESIGN SAP	EA®	DATE 25 JAN 06
DRAWN SAP		SCALE AS SHOWN
CHECKED AE		PROJECT NO. 29600.35
PROJECT MANAGER AE		SHEET NO. –

DWG. FILE No. F:\FEDERAL\DOD\NAVY\29600.35\CAD\TOPSHAM_ANNEX\TOPSHAM ANNEX SITE - FIGURE19_21.DWG (TAB: FIGURE19).DWG



LEGEND:

DIRECT-PUSH BORING LOCATIONS INSTALLED BY EA IN JUNE 2004

TOP-02 AREA (FORMER OIL/WATER SEPARATOR)

CHAIN LINK FENCE

INFERRED GROUNDWATER FLOW DIRECTION IS BASED ON REGIONAL GROUNWATER FLOW

NOTES:

1. FOSTER WHEELER ENVIRONMENTAL CORPORATION, FINAL REMOVAL ACTION TOPSHAM ANNEX PARCEL 2, TOPSHAM MAINE, MAY 2001.
2. INFERRED GROUNDWATER FLOW DIRECTION IS BASED ON REGIONAL GROUNDWATER FLOW.

FIGURE 20
TOP-02 (FORMER OIL/WATER SEPARATOR)
TOPSHAM ANNEX
TOPSHAM, MAINE

DESIGN SAP	EA®	DATE 12 JULY 04
DRAWN SAP		SCALE AS SHOWN
CHECKED AE		PROJECT NO. 29600.35
PROJECT MANAGER AE		SHEET NO. –

NOTES:

1. FOSTER WHEELER ENVIRONMENTAL CORPORATION, FINAL REMOVAL ACTION TOPSHAM ANNEX PARCEL 2, TOPSHAM MAINE, MAY 2001.
2. INFERRED GROUNDWATER FLOW DIRECTION IS BASED ON REGIONAL GROUNDWATER FLOW.

TOP-02-01 (SOIL) DRO – 9,200 ug/Kg
TOP-02-01 (GROUNDWATER) DRO – 89B ug/L

TOP-02-03 (2.0-6.0 FT BGS) (GROUNDWATER)

DRO	50B ug/L
GRO	2.1J ug/L

DRO=DIESEL RANGE ORGANICS
B=COMPOUND DETECTED IN ASSOCIATED METHOD BLANK.
J=REPORTED BELOW DETECTED LIMIT
ND=NOT DETECTED.
CONCENTRATIONS IN RED TEXT DENOTE EXCEEDANCE OF MEG, MCL, ME PRG, OR ME RAGS.

APPROXIMATE AREA OF FORMER OIL/WATER SEPARATOR

TOP-02-01 (1.0-5.0 FT BGS) (GROUNDWATER)

DRO	89B ug/L

TOP-02-01-GWI (5.0-6.0 FT BGS) (SOIL)

DRO	9,200 ug/Kg

TOP-02-02 (5.0-9.0FT BGS) (GROUNDWATER)

DRO	60B ug/L

TOP-02-02-GWI (5.0-6.0 FT BGS) (SOIL)

DRO	8,200 ug/Kg

INFERRED GROUNDWATER FLOW DIRECTION

TOP-02-03 (5.0 FT BGS) (SOIL)

DRO	3,600J ug/Kg
GRO	3,100 ug/Kg

TOP-02-03 (2.0-6.0 FT BGS) (GROUNDWATER)

DRO	50B ug/L
GRO	2.1J ug/L

TENNIS COURTS

CANAM DRIVE

UNNAMED DIRT PATH

UNNAMED DIRT PATH

70 0 70
SCALE

F:\FEDERAL\DOD\NAVY\29600.35\CAD\TOPSHAM_ANNEX\TOPSHAM ANNEX SITE - FIGURE 21.DWG (TAB: FIGURE 21).DWG

LEGEND:

DIRECT-PUSH BORING LOCATIONS INSTALLED BY EA IN JUNE 2004

TOP-02 AREA (FORMER OIL/WATER SEPARATOR)

CHAIN LINK FENCE

INFERRED GROUNDWATER FLOW DIRECTION IS BASED ON REGIONAL GROUNWATER FLOW

FIGURE 21
TOP-02 (FORMER OIL/WATER SEPARATOR)
SOIL AND GROUNDWATER CONCENTRATIONS
TOPSHAM ANNEX
TOPSHAM, MAINE

DESIGN SAP	EA®	DATE 28 OCTOBER 04
DRAWN SAP		SCALE AS SHOWN
CHECKED AE		PROJECT NO. 29600.35
PROJECT MANAGER AE		SHEET NO. –

TABLE 6 TOP-01 SAMPLE SUMMARY

Boring Sample Identification	Matrix	Date	Time	Total Depth of Boring (ft bgs)	Soil Sample Depth (ft bgs)	Groundwater Sample Interval (ft bgs)	Maximum PID/FID Response (ppm)	Analytical Parameters Collected
Top-01-01	Groundwater	21 JUN 2004	1030	13.0	NA	4.5-8.5	0.5/820	VOC, GRO, DRO, SVOC, RCRA 8 Metals
Top-01-02	Groundwater	21 JUN 2004	1315	10.0	NA	3.0-7.0	0.0/28.24	VOC, GRO, DRO, SVOC, RCRA 8 Metals
Top-01-03	Groundwater	21 JUN 2004	1600	10.0	NA	4.0-8.0	124/121	VOC, GRO, DRO, SVOC, RCRA 8 Metals
Top-01-01-GWI	Soil	21 JUN 2004	1030	13.0	5.0-5.9	NA	0.5/820	VOC, GRO, DRO, PCB, SVOC, RCRA 8 Metals
Top-01-01-Fill	Soil	21 JUN 2004	1045	13.0	6.5-7.0	NA	0.5/820	VOC, GRO, DRO, PCB, SVOC, RCRA 8 Metals
Top-01-02-GWI	Soil	21 JUN 2004	1110	10.0	2.5-3.0	NA	0.0/28.24	VOC, GRO, DRO, PCB, SVOC, RCRA 8 Metals
Top-01-02-Fill	Soil	21 JUN 2004	1120	10.0	1.0-2.0	NA	0.0/28.24	VOC, GRO, DRO, PCB, SVOC, RCRA 8 Metals
Top-01-03-GWI	Soil	21 JUN 2004	1250	10.0	6.0-7.0	NA	124/121	VOC, GRO, DRO, PCB, SVOC, RCRA 8 Metals
Top-01-03-FILL	Soil	21 JUN 2004	1230	10.0	2.0-3.0	NA	124/121	VOC, GRO, DRO, PCB, SVOC, RCRA 8 Metals

NOTE:
PID = Photoionization detector.
FID = Flame ionization detector.
NA = Not applicable.
VOC = Volatile organic compound.
GRO = Gasoline range organic.
DRO = Diesel range organic.
SVOC = Semivolatile organic compound.
RCRA = Resource Conservation and Recovery Act.
GWI = Sample collected at groundwater interval.
PCB = Polychlorinated biphenyl.
Fill = Sample collected from fill material.

TABLE 7 TOP-02 SAMPLE SUMMARY

Boring Sample Identification	Matrix	Date	Time	Total Depth of Boring (ft bgs)	Soil Sample Depth (ft bgs)	Groundwater Sample Interval (ft bgs)	Maximum PID/FID Response (ppm)	Analytical Parameters Collected
Top-02-01	Groundwater	22 JUN 2004	0630	9.5	NA	1.0-5.0	0.0/24.32	GRO, DRO, RCRA 8 Metals
Top-02-02	Groundwater	22 JUN 2004	0715	15.0	NA	5.0-9.0	0.0/23.24	GRO, DRO, RCRA 8 Metals
Top-02-03	Groundwater	22 JUN 2004	1045	10.0	NA	2.0-6.0	0.0/7.13	GRO, DRO, RCRA 8 Metals
Top-02-01-GWI	Soil	21 JUN 2004	1415	9.5	5.0-6.0	NA	0.0/24.32	GRO, DRO, RCRA 8 Metals
Top-02-02-GWI	Soil	21 JUN 2004	1445	15.0	5.0-6.0	NA	0.0/23.24	GRO, DRO, RCRA 8 Metals
Top-02-03-GWI	Soil	21 JUN 2004	1600	10.0	5.0	NA	0.0/7.13	GRO, DRO, RCRA 8 Metals

NOTE: PID = Photoionization detector.
FID = Flame ionization detector.
NA = Not applicable.
GRO = Gasoline range organic.
DRO = Diesel range organic.
RCRA = Resource Conservation and Recovery Act.
GWI = Sample collected at groundwater interval.

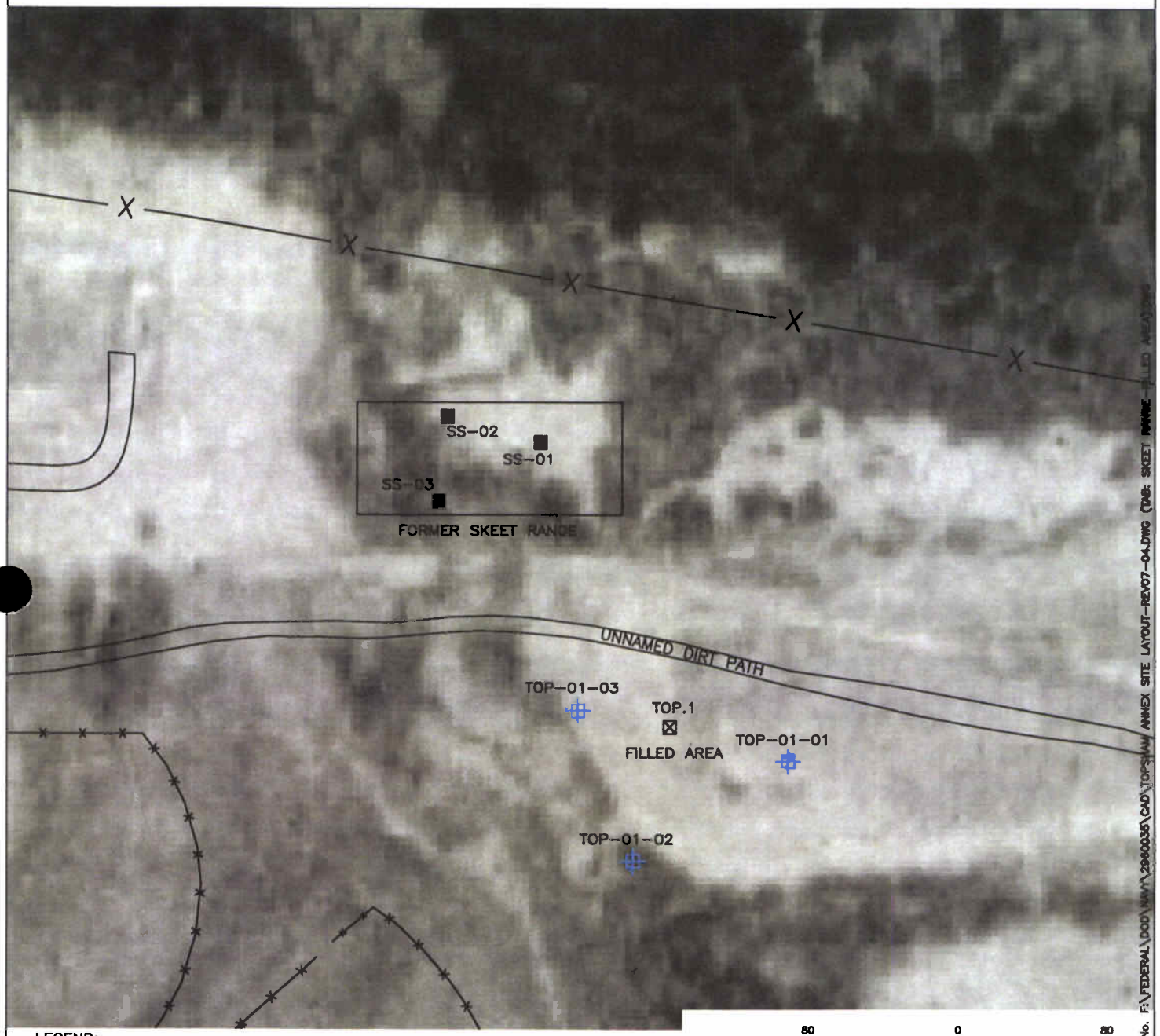
TABLE 8 BUILDING 380 (SKEET AREA) SAMPLE SUMMARY

Sample ID	Matrix	Date	Time	Soil Sample Depth (ft bgs)	Groundwater Sample Interval (ft bgs)	Maximum PID/FID Response (ppm)	Analytical Parameters Collected
SS-01-0604	Soil	21 JUN 2004	0930	0.0-0.5	NA	NA/NA	Lead
SS-02-0604	Soil	21 JUN 2004	0945	0.0-0.5	NA	NA/NA	Lead
SS-03-0604	Soil	21 JUN 2004	0955	0.0-0.5	NA	NA/NA	Lead

NOTE: PID = Photoionization detector.
FID = Flame ionization detector.
SS = Shallow soil samples – 0-6 in. below ground surface.
NA = Not applicable.

Appendix B

Aerial Photographs of Areas of Concern



80 0 80
SCALE

DWG. FILE No. F:\FEDERAL\DOD\NAVY\29600.35\CAD\TOPSHAM ANNEX SITE LAYOUT-REV07-04.DWG (TAB: SKEET RANGE FILLED AREA)

LEGEND:

- ■ SHALLOW SOIL SAMPLE LOCATIONS
- ⌖ DIRECT-PUSH BORING LOCATIONS INSTALLED BY EA IN JUNE 2004
- ⊠ TOP.1 AREA
- ×—×—× CHAIN LINK FENCE

NOTES:

1. THE LOCATION OF FORMER SKEET RANGE WAS TAKEN FROM AN OLD VICINITY PLAN AND IS APPROXIMATE.
2. FOSTER WHEELER ENVIRONMENTAL CORPORATION, FINAL REMOVAL ACTION TOPSHAM ANNEX PARCEL 2, TOPSHAM MAINE, MAY 2001.

FIGURE B-6
TOP-01 (FILLED AREA) AND
FORMER BLDG. 380 (SKEET RANGE)
TOPSHAM ANNEX
TOPSHAM, MAINE

DESIGN SAP	EA®	DATE 12 JULY 04
DRAWN SAP		SCALE AS SHOWN
CHECKED AE		PROJECT NO. 29600.35
PROJECT MANAGER AE		SHEET NO. –

Appendix C

Soil Boring Logs

EA Engineering, Science, and Technology, Inc.

LOG OF SOIL BORING

Coordinates:
Surface Elevation:
Well Riser Elevation:

Job. No.	Client:	Location:
29600.35.6874	U.S. Navy, EFANE	Topsham Annex, Topsham ME

Drilling Method: Direct Push Soil Borings; GeoProbe 6400 DT track mounted drill rig

Sampling Method: 5 ft Macro Core / 4 ft Screen Point 16 Sampler

Boring No. Top-02-01

Sheet 1 of 1

Drilling Water Level		
Date		
Time		

Start	Drilling Date/Times	Finish
6/21/04		6/21/04

Surface Conditions: Grass

Sample Type	Feet Driven/ft. Recvrd	Depth Boring (ft)	Samp # / depth (ft)	PID / FID (ppm)	Ft bgs	USCS Log	SOIL DESCRIPTION
MC	5/3.1	5	S-1/0-5	0.0/6.24	0		0.0-0.1 organic
						SW	0.1-0.7 Dk Brn Very Fine SAND some silt
					1	SW	0.7-1.1 Brn V. Fine SAND, LT silt
							Tr. small Gravel
					2	GW	1.1-1.8 Crushed Rock
							Pink → white Quartz / other
					3	SW	1.8-3.1 LT Brn V. Fine SAND
							LT. Clay, Small → Med Gravel
					4		
MC	4.5/1.3	9.5	S-2/5-9.5	0.0/2432	5	SW	5-6.3 Same as above very wet
							@ 5'
					6		
							Refusal @ 9.5'
					7		
					8		Soil Sample:
							Top-02-01-GWI @ 5.0-6.0'
					9		
					10		
					11		
					12		
					13		
					14		
							NR - Not Recorded

Logged by: C. Springer
Drilling Contractor: ADH Env.
Date: 21 June 2004
Driller: Hatfield

EA Engineering, Science, and Technology, Inc.

LOG OF SOIL BORING

Coordinates:
Surface Elevation:
Well Riser Elevation:

Job. No.	Client:	Location:
29600.35.6874	U.S. Navy, EFANE	Topsham Annex Topsham ME

Drilling Method: Direct Push Soil Borings; GeoProbe 6400 DT track mounted drill rig

Sampling Method: 5 ft Macro Core / 4 ft Screen Point 16 Sampler

Boring No. Top-02-02

Sheet 1 of

Drilling Water Level		
Date		
Time		

Start	Drilling Date/Times	Finish
6/21/04		6/21/04

Surface Conditions: Grass

Sample Type	Feet Driven/ft. Recvrd	Depth Boring (ft)	Samp # / depth (ft)	PID / FID (ppm)	Ft bgs	USCS Log	SOIL DESCRIPTION
MC	5/2.5	5	S-1/0-5	0.0/5.21	0		0.0 - 0.1 organic
						SW	0.1 - 1.2 Brn V. Fine SAND, Lt. Clay
					1	GW	1.2 - 1.5 Crushed Rock, Quartz, Biotite
						GW	1.5 - 2.0 Crushed Rock, Quartz chips
					2	SW	2.0 - 2.5 LT Brn V. Fine SAND, Lt Clay
							Lt. Small Gravel
					3		
					4		
MC	5/1.1	10	S-2/5-10	0.0/23.24	5	SW	5.0 - 6.0 Same wet @ 5'
					6	GW	6.0 - 6.1 Crushed Rock, Biotite
					7		Soil Sample: Top-02-02-GW1 @ 5.0-6.0'
					8		
					9		
MC	5/5	15	S-3/10-15	0.0/6.23	10	GW	10.0 - 11.1 Same
					11	SW	11.1 - 13.2 Brn Gray F SAND, Biotite chips
					12		
					13	MH	13.2 - 13.7 Dark Gray Organics, Silt and Clay Saturated
					14	CL	13.7 - 15 Gray Green CLAY, Lt Silt, Tight
							End of Boring @ 15'

Logged by: C. Sponsler
Date: 6-21-04
Drilling Contractor: ADH ENV
Driller: Hat Field

EA Engineering, Science, and Technology, Inc.

LOG OF SOIL BORING

Coordinates:
Surface Elevation:
Well Riser Elevation:

Job. No.	Client:	Location:
29600.35.6874	U.S. Navy, EFANE	Topsham Annex, Topsham ME

Drilling Method: Direct Push Soil Borings; GeoProbe 6400 DT track mounted drill rig

Sampling Method: 5 ft Macro Core / 4 ft Screen Point 16 Sampler

Boring No. Top-02-03

Sheet 1 of 1

Drilling Water Level		
Date		
Time		

Start	Drilling Date/Times	Finish
6/21/04		6/21/04

Surface Conditions: Grass

Sample Type	Feet Driven/ft. Recvrd	Depth Boring (ft)	Samp # / depth (ft)	PID / FID (ppm)	Ft bgs	USCS Log	SOIL DESCRIPTION
MC	5/3.3	5	S-1/0-5	0.0/6.28	0		0.0-0.1 organic
						ML	0.1-1.0 Brn SILT, trace clay
					1	GW	1.0-1.4 Crushed Rock w/ white Quartz
						SM	1.4-3.3 Dark Brn SILT, trace clay
					2		Trace S→M Gravel
					3		
					4		water 5'
MC	5/2.6	10	S-2/5-10	0.0/7.13	5	GP	5.0-6.1 Orange Brn S-M Gravel LT. Fine Sand
					6	CL	6.1-7.6 Gray CLAY, LT Silt very soft
					7		End of Boring @ 10'
					8		
					9		Soil Sample: Top-02-03-GWI @ 5.0-6.0'
					10		
					11		
					12		
					13		
					14		

Logged by: C. Spanger
Drilling Contractor: ADH Env.
Date: 6-21-04
Driller: Hatfield

EA Engineering, Science, and Technology, Inc.

LOG OF SOIL BORING

Coordinates:
Surface Elevation:
Well Riser Elevation:

Job. No.	Client:	Location:
29600.35.6874	U.S. Navy, EFANE	Topsham Annex Topsham ME

Drilling Method: Direct Push Soil Borings; GeoProbe 6400 DT track mounted drill rig

Sampling Method: 5 ft Macro Core / 4 ft Screen Point 16 Sampler

Boring No. Top-01-01

Sheet 1 of 1

Drilling Water Level		
Date		
Time		

Start	Drilling Date/Times	Finish
21 June 2004		21 June 2004

Surface Conditions: Grass

Sample Type	Feet Driven/ft. Recvrd	Depth Boring (ft)	Samp # / depth (ft)	PID / FID (ppm)	Ft bgs	USCS Log	SOIL DESCRIPTION
MC	5/3.7	5	S-1/0-5	0.0/11.5	0		0-0.1 organic
						ML	0.1-2.1 Brn SILT, Little Clay DRY Trace Fine Sand
					1		
					2	GW	2.1-2.4 Crushed Quartz
						ML	2.4-3.4 Brn SILT, LT Clay, LT Fine Sand Damp
					3	SW	3.4-3.7 Brn Grey Fine Sand Damp LT Clay, Trace Silt
					4		
MC	5/2.0	10	S-2/5-10	0.5/150	5	ML	5.0-5.2 Brn SILT, Some Clay Wet @ 5.3'
						SW	5.2-5.9 Brn Grey Fine SAND Some Clay, Trace Silt
					6	GW	5.9-6.5 Crushed ROCK, Quartz/Biotite
					7	SW	6.5-7.0 Grey Brn Fine SAND Wet Little Clay, Crushed Rock Quartz/Biotite
					8		
					9		
MC	3/3	13	S-3/10-13	0.0/820	10	SW	10-11.5 SAME As Above
						ML	11.5-13 Grey Brn SILT, LT Clay Stiff
					11		End of Boring @ 13'
					12		
					13		Soil Sample: Top-01-01-GWI @ 5.0-5.9' Top-01-01-Fill @ 6.5-7.0'
					14		FID BKGD 3.95 ppm

Logged by: C. Springer
Date: 21 June 2004
Drilling Contractor: ADH Env.
Driller: Peter Hatfield

EA Engineering, Science, and Technology, Inc.

LOG OF SOIL BORING

Coordinates:
Surface Elevation:
Well Riser Elevation:

Job. No.	Client:	Location:
29600.35.6874	U.S. Navy, EFANE	Topsham Annex, Topsham ME

Drilling Method: Direct Push Soil Borings; GeoProbe 6400 DT track mounted drill rig

Sampling Method: 5 ft Macro Core / 4 ft Screen Point 16 Sampler

Boring No. Top-01-02

Sheet 1 of

Drilling Water Level		
Date		
Time		

Start	Drilling Date/Times	Finish
6/21/04		6/21/04

Surface Conditions: Tall Grass

Sample Type	Feet Driven/ft. Recvrd	Depth Boring (ft)	Samp # / depth (ft)	PID / FID (ppm)	Ft bgs	USCS Log	SOIL DESCRIPTION
MC	5/3	5	S-1 / 0-5	0.0 / 26.9	0		0-1.0 Brn SILT Dry
					1	SW	1.0-2.5 Brn V. Fine SAND, LT Si, LT
					2	SW	2.5-3.0 Brn Fine SAND, LT Clay, Tr. Si, LT Tr. Small Gravel
					3		WET @ 3.0'
					4		
MC	5/5	10	S-2 / 5-10	0.0 / 28.24	5	SW	5.0-5.9 Dvr Brn Fine SAND WET LT. Clay, Tr. Si LT
					6	CL	5.9-7.5 Dvr Brn CLAY STIFF LT. Si LT
					7	CL	7.5-8.0 Gray Clay, LT. Si LT STIFF
					8	CL	8.0-10.0 Gray Brn SILT, Tr. Clay STIFF
					9		End of Boring @ 10'
					10		
					11		Soil Sample: Top01-02-GWI @ 2.5-3.0; Top01-02-Fill @ 1.0-2.0
					12		
					13		
					14		

Logged by: C. Springer
Drilling Contractor: ADH ENV.
Date: 21 June 2004
Driller: Peter Hatfield

EA Engineering, Science, and Technology, Inc.

LOG OF SOIL BORING

Coordinates:
Surface Elevation:
Well Riser Elevation:

Job. No.	Client:	Location:
29600.35.6874	U.S. Navy, EFANE	Topsham Annex, Topsham ME

Drilling Method: Direct Push Soil Borings, GeoProbe 6400 DT track mounted drill rig

Sampling Method: 5 ft Macro Core / 4 ft Screen Point 16 Sampler

Boring No. Top-01-03

Sheet 1 of 1

Drilling Water Level		
Date		
Time		

Start	Drilling Date/Times	Finish
6/21/04		6/21/04

Surface Conditions: Tall Grass

Sample Type	Feet Driven/ft. Recvrd	Depth Boring (ft)	Samp # / depth (ft)	PID / FID (ppm)	Ft bgs	USCS Log	SOIL DESCRIPTION
MC	5/3.5	5	S-1/0-5	0/0.21	0		0.0-0.1 Organic
						ML	0.1-1.3 Brn SILT, Concrete Rubble, DRY
					1	SW	1.3-3.5 Brn V. Fine SAND, Tr. Clay, LT. Small Gravel
					2		
					3		
					4		
MC	5/3.2	10	S-2/5-10	124/121	5	SW	5.0-6.0 SAME AS above Damp
					6	SW	6.0-7.1 Brn V. Fine SAND Some clay
					7		wet @ 6.0'
						CL	7.1-8.0 DRK Brn CLAY, Some SILT
					8	CL	8.0-8.2 Gray CLAY, Some SILT
					9		
					10		End of Boring @ 10'
					11		Soil Samples
							Top-01-03-GWI @ 6.0-7.0'
					12		Top-01-03-FILL @ 2.0-3.0'
					13		
					14		

Logged by: C. Springer
Drilling Contractor: ADH ENV.
Date: 21 June 2004
Driller: HATFIELD

Appendix E

Survey Data

Area E	BLDG1099-01	405411.5693	3007417.5993	159.89	194
	BLDG1099-02	405426.1798	3007396.3195	159.25	193
	BLDG1099-03	405405.4928	3007385.8672	160.45	195
	BLDG1099-04	405380.1545	3007350.5856	161.19	196
	BLDG1099-05	405426.7503	3007381.2301	159.31	197
	BLDG1099-06	405434.4363	3007356.3750	159.97	198
	BLDG1114-01	405454.1598	3007311.4128	158.78	199
	BLDG1114-02	405402.4417	3007295.9411	160.90	201
	BLDG1114-03	405394.0371	3007209.3813	158.14	183
	BLDG1114-04	405449.1261	3007192.0838	152.83	182
	BLDG1114-05	405474.4359	3007266.5069	158.14	205
	BLDG1114-06	405418.4882	3007319.3501	159.70	200
	BLDG1114-07	405389.5121	3007312.7541	161.97	203
	BLDG1114-08	405384.0080	3007284.9178	161.59	202
	BUILDING 1114 NORTH CORNER	405482.6674	3007287.9405	158.35	204
	CHAIN LINK FENCE CORNER	405492.6728	3007242.4039	153.45	206
	CHAIN LINK FENCE CORNER	405439.4625	3007187.1670	153.49	207
TOP 1	TOP 01 01	406571.3390	3006587.5054	118.48	174
	TOP 01 02	406517.5170	3006504.8269	114.49	173
	TOP 01 03	406598.8183	3006475.7583	117.07	172
TOP 2	TOP 02 01	406117.6254	3006468.1810	121.48	171
	TOP 02 02	406092.2602	3006446.8414	121.62	170
	TOP 02 03	406073.2783	3006463.6851	122.40	169
Bldg. 380	SS 01	406741.2121	3006455.3912	108.57	175
	SS 02	406755.0860	3006405.7987	107.85	176
	SS 03	406709.8709	3006401.2289	107.27	177

Notes:

1) Brian Smith Surveying, Inc completed the survey on 8 July 2004
2) ft msl = Feet Mean Sea Level
3) Locations are in Maine State Plane NADA 83 West (based on previous survey done by Summit Environmental (2002) and Malcolm Pirnie (2001)

Bldg 380 is the Skeet Range

Appendix F

Data Summary Tables

TABLE 6 SUMMARY OF ANALYTICAL RESULTS FOR SAMPLES COLLECTED FROM TOP-01

GROUNDWATER SAMPLES

VOLATILE ORGANIC COMPOUNDS BY U.S. ENVIRONMENTAL PROTECTION AGENCY METHOD 8260B (µg/L)

		TOP-01-01	TOP-01-02	TOP-01-03	TOP-01-03-(DUP)
Analyte	**MEG/MCL**				
1,1-DICHLOROETHENE	0.6/7	(<1U)	(<1U)	(<1U)	(<1U)
1,2-DICHLOROETHANE	4/5	(<1U)	(<1U)	(<1U)	(<1U)
1,2-DICHLOROETHYLENE	---/---	(<2U)	(<2U)	(<2U)	(<2U)
2-BUTANONE	1,440/---	(<5U)	(<5U)	(<5U)	(<5U)
2-HEXANONE	---/---	(<5U)	(<5U)	(<5U)	(<5U)
ACETONE	700/---	(<5U)	(<5U)	(<5U)	(<5U)
BENZENE	12/5	(<1U)	(<1U)	(<1U)	(<1U)
BROMOMETHANE	10/---	(<2U)	(<2U)	(<2U)	(<2U)
CARBON DISULFIDE	---/---	(<1U)	(<1U)	(<1U)	(<1U)
CHLOROFORM	57/---	(<1U)	(<1U)	(<1U)	(<1U)
CHLOROMETHANE	3/---	(<2U)	(<2U)	(<2U)	(<2U)
CIS-1,2-DICHLOROETHENE	70/70	(<1U)	(<1U)	(<1U)	(<1U)
ETHYLBENZENE	70/700	(<1U)	(<1U)	(<1U)	(<1U)
M-,P-XYLENE	---/---	(<2U)	(<2U)	(<2U)	(<2U)
METHYLENE CHLORIDE	47/5	(<2U)	(<2U)	(<2U)	(<2U)
O-XYLENE	---/---	(<1U)	(<1U)	(<1U)	(<1U)
TOLUENE	1,400/1,000	0.5JB	(<1U)	0.2JB	0.8JB
TRANS-1,2-DICHLOROETHENE	140/100	(<1U)	(<1U)	(<1U)	(<1U)
TRICHLOROETHENE	32/5	(<1U)	(<1U)	(<1U)	(<1U)
VINYL CHLORIDE	0.2/2	(<2U)	(<2U)	(<2U)	(<2U)
XYLENES (TOTAL)	14,000/10,000	(<3U)	(<3U)	(<3U)	(<3U)

SEMIVOLATILE ORGANIC COMPOUNDS BY U.S. ENVIRONMENTAL PROTECTION AGENCY METHOD 8270C (µg/L)

		TOP-01-01	TOP-01-02	TOP-01-03	TOP-01-03-(DUP)
Analyte	**MEG/MCL**				
4-METHYLPHENOL	3.5/---	(<10U)	(<10U)	(<10U)	(<10U)
ACENAPHTHENE	---/---	(<10U)	(<10U)	(<10U)	(<10U)
ANTHRACENE	---/---	(<10U)	(<10U)	(<10U)	(<10U)
BENZO(A)ANTHRACENE	---/---	(<10U)	(<10U)	(<10U)	(<10U)
BENZO(A)PYRENE	0.05/0.2	(<10U)	(<10U)	(<10U)	(<10U)
BENZO(B)FLUORANTHENE	---/---	(<10U)	(<10U)	(<10U)	(<10U)
BENZO(K)FLUORANTHENE	---/---	(<10U)	(<10U)	(<10U)	(<10U)
BENZO[G,H,I]PERYLENE	---/---	(<10U)	(<10U)	(<10U)	(<10U)
BIS(2-ETHYLHEXYL) PHTHALATE	25/---	(<10U)	[illegible]	(<10U)	(<10U)
CARBAZOLE	---/---	(<10U)	(<10U)	(<10U)	(<10U)
CHRYSENE	---/---	(<10U)	(<10U)	(<10U)	(<10U)
DIBENZO(A,H)ANTHRACENE	---/---	(<10U)	(<10U)	(<10U)	(<10U)

SEMIVOLATILE ORGANIC COMPOUNDS BY U.S. ENVIRONMENTAL PROTECTION AGENCY METHOD 8270C (µg/L)

		TOP-01-01	TOP-01-02	TOP-01-03	TOP-01-03-(DUP)
DIBENZOFURAN	---/---	(<10U)	(<10U)	(<10U)	(<10U)
DI-N-BUTYL PHTHALATE	700/---	(<10U)	(<10U)	2J	(<10U)
FLUORANTHENE	---/---	(<10U)	(<10U)	(<10U)	(<10U)
FLUORENE	---/---	(<10U)	(<10U)	(<10U)	(<10U)
INDENO(1,2,3-CD)PYRENE	---/---	(<10U)	(<10U)	(<10U)	(<10U)
NAPHTHALENE	14/---	(<10U)	(<10U)	(<10U)	(<10U)
PHENANTHRENE	---/---	(<10U)	(<10U)	(<10U)	(<10U)
PHENOL	4,000/---	(<10U)	(<10U)	(<10U)	(<10U)
PYRENE	---/---	(<10U)	(<10U)	(<10U)	(<10U)

TPH-DRO BY MDHS-HETL LOP 4.1.25 (µg/L)

		TOP-01-01	TOP-01-02	TOP-01-03	TOP-01-03-(DUP)
Analyte	**MEG/MCL**				
DIESEL RANGE ORGANICS	50/---	[redacted]	[redacted]	48B	50B

TPH-GRO BY MDHS-HETL LOP 4.2.17 (µg/L)

		TOP-01-01	TOP-01-02	TOP-01-03	TOP-01-03-(DUP)
Analyte	**MEG/MCL**				
GASOLINE RANGE ORGANICS	50/---	(<10U)	(<10U)	9.8J	2.2J

TARGET ANALYTE LIST ELEMENTS BY U.S. ENVIRONMENTAL PROTECTION AGENCY 6000/7000 SERIES METHODS (µg/L)

		TOP-01-01	TOP-01-02	TOP-01-03	TOP-01-03-(DUP)
Analyte	**MEG/MCL**				
ARSENIC	10/1	[redacted]	[redacted]	(<3.03U)	(<3.03U)
BARIUM	2,000/2,000	19.9	89.5	16.3	17
CADMIUM	3.5/5	(<0.3U)	(<0.3U)	(<0.3U)	(<0.3U)
CHROMIUM	40/100	1.2B*	[redacted]	1.2B*	1.8B*
LEAD	10/15	(<1.7U)	[redacted]	(<1.7U)	(<1.7U)
MERCURY	---/---	(<0.01U)	(<0.01U)	(<0.01U)	(<0.01U)
SELENIUM	35/50	(<3.71U)	(<3.71U)	(<3.71U)	(<3.71U)
SILVER	35/---	(<0.7U)	(<0.7U)	(<0.7U)	(<0.7U)

SOIL SAMPLES

VOLATILE ORGANIC COMPOUNDS BY U.S. ENVIRONMENTAL PROTECTION AGENCY METHOD 8260B (µg/Kg)

		TOP-01-01-FILL	TOP-01-01-GWI	TOP-01-02-FILL	TOP-01-02-GWI	TOP-01-03-FILL	TOP-01-03-GWI	TOP-01-03-GWI-(DUP)
Analyte	**ME RAGS**							
1,1-DICHLOROETHENE	2000	(<5U)	(<5U)	(<5U)	(<5U)	(<6U)	(<5U)	(<5U)
1,2-DICHLOROETHANE	---	(<5U)	(<5U)	(<5U)	(<5U)	(<6U)	(<5U)	(<5U)
1,2-DICHLOROETHYLENE	---	(<10U)	(<10U)	(<10U)	(<10U)	(<12U)	(<10U)	(<11U)

VOLATILE ORGANIC COMPOUNDS BY U.S. ENVIRONMENTAL PROTECTION AGENCY METHOD 8260B (µg/Kg)

		TOP-01-01-FILL	TOP-01-01-GWI	TOP-01-02-FILL	TOP-01-02-GWI	TOP-01-03-FILL	TOP-01-03-GWI	TOP-01-03-GWI-(DUP)
2-BUTANONE	---	(<26U)	(<25U)	(<25U)	(<26U)	21J	(<25U)	(<27U)
2-HEXANONE	---	(<26U)	(<25U)	10J	12J	48	(<25U)	(<27U)
ACETONE	475000	52	48	49	82	190	45	64
BENZENE	5000	(<5U)	(<5U)	(<5U)	(<5U)	(<6U)	(<5U)	(<5U)
BROMOMETHANE	---	(<10U)	(<10U)	(<10U)	(<10U)	(<12U)	(<10U)	(<11U)
CARBON DISULFIDE	---	2J	(<5U)	(<5U)	(<5U)	(<6U)	(<5U)	(<5U)
CHLOROFORM	---	(<5U)	(<5U)	(<5U)	(<5U)	(<6U)	(<5U)	(<5U)
CHLOROMETHANE	---	(<10U)	(<10U)	(<10U)	(<10U)	(<12U)	(<10U)	(<11U)
CIS-1,2-DICHLOROETHENE	---	(<5U)	(<5U)	(<5U)	(<5U)	(<6U)	(<5U)	(<5U)
ETHYLBENZENE	1670000	(<5U)	(<5U)	(<5U)	(<5U)	(<6U)	(<5U)	(<5U)
M-,P-XYLENE	---	(<10U)	(<10U)	(<10U)	(<10U)	(<12U)	(<10U)	(<11U)
METHYLENE CHLORIDE	13000	8B	6B	10B	12B	15B	9B	8
O-XYLENE	---	(<5U)	(<5U)	(<5U)	(<5U)	(<6U)	(<5U)	(<5U)
TOLUENE	2390000	(<5U)	(<5U)	(<5U)	(<5U)	0.8J	(<5U)	(<5U)
TRANS-1,2-DICHLOROETHENE	135000	(<5U)	(<5U)	(<5U)	(<5U)	(<6U)	(<5U)	(<5U)
TRICHLOROETHENE	19000	(<5U)	(<5U)	(<5U)	(<5U)	(<6U)	(<5U)	(<5U)
VINYL CHLORIDE	400	(<10U)	(<10U)	(<10U)	(<10U)	(<12U)	(<10U)	(<11U)
XYLENES (TOTAL)	10	(<15U)	(<15U)	(<15U)	(<16U)	(<17U)	(<15U)	(<16U)

SEMIVOLATILE ORGANIC COMPOUNDS BY U.S. ENVIRONMENTAL PROTECTION AGENCY METHOD 8270C (µg/Kg)

		TOP-01-01-FILL	TOP-01-01-GWI	TOP-01-02-FILL	TOP-01-02-GWI	TOP-01-03-FILL	TOP-01-03-GWI	TOP-01-03-GWI-(DUP)
Analyte	**ME RAGS**							
4-METHYLPHENOL	---	(<380U)	(<390U)	(<430U)	(<400U)	(<380U)	(<380U)	(<430U)
ACENAPHTHENE	---	(<380U)	(<390U)	84J	(<400U)	210J	(<380U)	140J
ANTHRACENE	---	(<380U)	(<390U)	(<430U)	(<400U)	290J	(<380U)	110J
BENZO(A)ANTHRACENE	---	(<380U)	(<390U)	89J	(<400U)	910	(<380U)	560
BENZO(A)PYRENE	2,000*	(<380U)	(<390U)	100J	(<400U)	840	(<380U)	610
BENZO(B)FLUORANTHENE	---	(<380U)	(<390U)	(<430U)	(<400U)	750	(<380U)	570
BENZO(K)FLUORANTHENE	---	(<380U)	(<390U)	(<430U)	(<400U)	860	(<380U)	660
BENZO[G,H,I]PERYLENE	---	(<380U)	(<390U)	(<430U)	(<400U)	380	(<380U)	250J
BIS(2-ETHYLHEXYL) PHTHALATE	---	(<380U)	(<390U)	(<430U)	(<400U)	(<380U)	(<380U)	(<430U)
CARBAZOLE	---	(<380U)	(<390U)	(<430U)	(<400U)	270J	(<380U)	110J
CHRYSENE	---	(<380U)	(<390U)	100J	(<400U)	920	(<380U)	650
DIBENZO(A,H)ANTHRACENE	---	(<380U)	(<390U)	(<430U)	(<400U)	190J	(<380U)	76J
DIBENZOFURAN	---	(<380U)	(<390U)	(<430U)	(<400U)	92J	(<380U)	(<430U)
DI-N-BUTYL PHTHALATE	---	87JB	(<390U)	280JB	(<400U)	51JB	(<380U)	(<430U)
FLUORANTHENE	---	(<380U)	(<390U)	160J	(<400U)	2300	(<380U)	1200
FLUORENE	---	(<380U)	(<390U)	(<430U)	(<400U)	140J	(<380U)	50J
INDENO(1,2,3-CD)PYRENE	---	(<380U)	(<390U)	(<430U)	(<400U)	340J	(<380U)	250J
NAPHTHALENE	---	(<380U)	(<390U)	(<430U)	(<400U)	67J	(<380U)	70J

SEMIVOLATILE ORGANIC COMPOUNDS BY U.S. ENVIRONMENTAL PROTECTION AGENCY METHOD 8270C (µg/Kg)

		TOP-01-01-FILL	TOP-01-01-GWI	TOP-01-02-FILL	TOP-01-02-GWI	TOP-01-03-FILL	TOP-01-03-GWI	TOP-01-03-GWI-(DUP)
PHENANTHRENE	---	(<380U)	(<390U)	(<430U)	(<400U)	1700	(<380U)	830
PHENOL	---	(<380U)	(<390U)	(<430U)	(<400U)	(<380U)	(<380U)	(<430U)
PYRENE	---	(<380U)	(<390U)	210J	(<400U)	1600	(<380U)	1400

TPH-DRO BY MDHS-HETL LOP 4.1.25 (µg/Kg)

		TOP-01-01-FILL	TOP-01-01-GWI	TOP-01-02-FILL	TOP-01-02-GWI	TOP-01-03-FILL	TOP-01-03-GWI	TOP-01-03-GWI-(DUP)
Analyte	**ME PRG**							
DIESEL RANGE ORGANICS	10000	[redacted]	[redacted]	[redacted]	(<6000U)	[redacted]	[redacted]	[redacted]

TPH-GRO BY MDHS-HETL LOP 4.2.17 (µg/Kg)

		TOP-01-01-FILL	TOP-01-01-GWI	TOP-01-02-FILL	TOP-01-02-GWI	TOP-01-03-FILL	TOP-01-03-GWI	TOP-01-03-GWI-(DUP)
Analyte	**ME PRG**							
GASOLINE RANGE ORGANICS	10000	(<2500U)	(<2500U)	(<3000U)	[redacted]	(<2500U)	(<4600U)	(<2700U)

TARGET ANALYTE LIST ELEMENTS BY U.S. ENVIRONMENTAL PROTECTION AGENCY 6000/7000 SERIES METHODS (µg/Kg)

		TOP-01-01-FILL	TOP-01-01-GWI	TOP-01-02-FILL	TOP-01-02-GWI	TOP-01-03-FILL	TOP-01-03-GWI	TOP-01-03-GWI-(DUP)
Analyte	**ME RAGS**							
ARSENIC	10000	5300	5000	9100	5000	4800	[redacted]	5600
BARIUM	10000000	50200	59100	69800	45900	23600	33700	30500
CADMIUM	27000	(<20U)	(<30U)	(<50U)	(<30U)	(<20U)	(<30U)	(<30U)
CHROMIUM	950000	25400	30200	37400	21600	13200	32500	17300
LEAD	375000	7000	8600	12200	7400	15000	8200	20800
MERCURY	60000	20B*	20B*	30B*	20B*	40	20B*	70
SELENIUM	950000	(<290U)	(<390U)	(<660U)	(<340U)	(<280U)	(<360U)	(<410U)
SILVER	950000	(<60U)	(<70U)	(<60U)	(<60U)	(<50U)	(<70U)	(<80U)

POLYCHLORINATED BIPHENYLS BY EPA METHOD 8082 (µg/Kg)

		TOP-01-01-FILL	TOP-01-01-GWI	TOP-01-02-FILL	TOP-01-02-GWI	TOP-01-03-FILL	TOP-01-03-GWI	TOP-01-03-GWI-(DUP)
Analyte	**ME RAGS**							
AROCLOR-1260	2,200*	(<20U)	(<20U)	(<22U)	(<20U)	15J	(<20U)	(<22U)

FIELD QA/QC SAMPLES

VOLATILE ORGANIC COMPOUNDS BY U.S. ENVIRONMENTAL PROTECTION AGENCY METHOD 8260B (µg/L)

		TOP-RINSE	Top-Trip-01-(GW)	Top-Trip-01-(Soil)
Analyte	**MEG/MCL**			
1,1-DICHLOROETHENE	---	(<1U)	(<1U)	(<5U)
1,2-DICHLOROETHANE	---	(<1U)	(<1U)	(<5U)
1,2-DICHLOROETHYLENE	---	(<2U)	(<2U)	(<10U)
2-BUTANONE	---	(<5U)	(<5U)	(<25U)

VOLATILE ORGANIC COMPOUNDS BY U.S. ENVIRONMENTAL PROTECTION AGENCY METHOD 8260B (µg/L)

		TOP-RINSE	Top-Trip-01-(GW)	Top-Trip-01-(Soil)
2-HEXANONE	---	(<5U)	(<5U)	(<25U)
ACETONE	---	70	(<5U)	(<25U)
BENZENE	---	(<1U)	(<1U)	(<5U)
BROMOMETHANE	---	(<2U)	(<2U)	(<10U)
CARBON DISULFIDE	---	(<1U)	(<1U)	(<5U)
CHLOROFORM	---	(<1U)	(<1U)	(<5U)
CHLOROMETHANE	---	5	(<2U)	(<10U)
CIS-1,2-DICHLOROETHENE	---	(<1U)	(<1U)	(<5U)
ETHYLBENZENE	---	5	(<1U)	(<5U)
M-,P-XYLENE	---	22	(<2U)	(<10U)
METHYLENE CHLORIDE	---	(<2U)	(<2U)	18B
O-XYLENE	---	8	(<1U)	(<5U)
TOLUENE	---	2	(<1U)	(<5U)
TRANS-1,2-DICHLOROETHENE	---	(<1U)	(<1U)	(<5U)
TRICHLOROETHENE	---	(<1U)	(<1U)	(<5U)
VINYL CHLORIDE	---	(<2U)	(<2U)	(<10U)
XYLENES (TOTAL)	---	31	(<3U)	(<15U)

SEMIVOLATILE ORGANIC COMPOUNDS BY U.S. ENVIRONMENTAL PROTECTION AGENCY METHOD 8270C (µg/L)

		TOP-RINSE
Analyte	**MEG/MCL**	
4-METHYLPHENOL	---	(<11U)
ACENAPHTHENE	---	(<11U)
ANTHRACENE	---	(<11U)
BENZO(A)ANTHRACENE	---	(<11U)
BENZO(A)PYRENE	---	(<11U)
BENZO(B)FLUORANTHENE	---	(<11U)
BENZO(K)FLUORANTHENE	---	(<11U)
BENZO[G,H,I]PERYLENE	---	(<11U)
BIS(2-ETHYLHEXYL) PHTHALATE	---	(<11U)
CARBAZOLE	---	(<11U)
CHRYSENE	---	(<11U)
DIBENZO(A,H)ANTHRACENE	---	(<11U)
DIBENZOFURAN	---	(<11U)
DI-N-BUTYL PHTHALATE	---	4J
FLUORANTHENE	---	(<11U)
FLUORENE	---	(<11U)
INDENO(1,2,3-CD)PYRENE	---	(<11U)
NAPHTHALENE	---	(<11U)
PHENANTHRENE	---	(<11U)

SEMIVOLATILE ORGANIC COMPOUNDS BY U.S. ENVIRONMENTAL PROTECTION AGENCY METHOD 8270C (µg/L)

		TOP-RINSE
PHENOL	---	(<11U)
PYRENE	---	(<11U)

TPH-DRO BY MDHS-HETL LOP 4.1.25 (µg/L)

		TOP-RINSE
Analyte	**MEG/MCL**	
DIESEL RANGE ORGANICS	---	60B

TPH-GRO BY MDHS-HETL LOP 4.2.17 (µg/L)

		TOP-RINSE
Analyte	**MEG/MCL**	
GASOLINE RANGE ORGANICS	---	26

TARGET ANALYTE LIST ELEMENTS BY U.S. ENVIRONMENTAL PROTECTION AGENCY 6000/7000 SERIES METHODS (µg/L)

		TOP-RINSE
Analyte	**MEG/MCL**	
ARSENIC	---	(<3.03U)
BARIUM	---	1.5B*
CADMIUM	---	(<0.3U)
CHROMIUM	---	2.7B*
LEAD	---	(<1.7U)
MERCURY	---	(<0.01U)
SELENIUM	---	4B*
SILVER	---	(<0.7U)

MEG (Maximum Exposure Guideline) obtained from State of Maine Department of Human Services Maximum Exposure Guidelines, memorandum dated 23 October 1992. Dashes (---) indicate compound has no applicable MEG.
MCL (Maximum Contaminant Level) obtained from 40 CFR Parts 141 and 142 (U.S. EPA 1998). Dashes (---) indicate compound has no applicable MCL.
ME RAGS obtained from Remedial Action Guidelines for Contaminated Soils provided by the State of Maine Department of Environmental Protection. Residential Guidelines are used for the Project Action Level. Dashes (---) indicate compound has no applicable guideline.
* indicates Residential Toxic Equivalent
ME PRG (Maine Petroleum Remediation Goal) taken from Procedural Guidelines for Establishing Action Levels and Remediation Goals for the Remediation of Oil Contaminated Soil and Groundwater in Maine, State of Maine Department of Environmental Protection, Rev 13, March 2000

NOTE:
DUP indicates a duplicate sample.
U = Not detected at or above the sample quantitation limit. Shown as (<____U).
B = Compound detected in associated method blank.
B* = Analyte concentration is between the Instrument Detection Limit and the Contract Required Detection Limit.
J = Estimated concentration.

Only those analytes detected in at least one of the samples are shown on this table.
Concentrations highlighted with gray and bold type denote exceedance of MEG, MCL, ME PRG, or ME RAGS.

TABLE 7 SUMMARY OF ANALYTICAL RESULTS FOR SAMPLES COLLECTED FROM TOP-02

GROUNDWATER SAMPLES

TPH-DRO BY MDHS-HETL LOP 4.1.25 (µg/L)

Analyte	MEG/MCL	TOP-02-01	TOP-02-02	TOP-02-03
DIESEL RANGE ORGANICS	50/---	[redacted]	[redacted]	50B

TPH-GRO BY MDHS-HETL LOP 4.2.17 (µg/L)

Analyte	MEG/MCL	TOP-02-01	TOP-02-02	TOP-02-03
GASOLINE RANGE ORGANICS	50/---	(<10U)	(<10U)	2.1J

TARGET ANALYTE LIST ELEMENTS BY U.S. ENVIRONMENTAL PROTECTION AGENCY 6000/7000 SERIES METHODS (µg/L)

Analyte	MEG/MCL	TOP-02-01	TOP-02-02	TOP-02-03
ARSENIC	10/1	(<3.03U)	(<3.03U)	(<3.03U)
BARIUM	2,000/2,000	49.8	27.6	9.1
CADMIUM	3.5/5	(<0.3U)	(<0.3U)	(<0.3U)
CHROMIUM	40/100	1.8B*	(<0.8U)	(<0.8U)
LEAD	10/15	(<1.7U)	(<1.7U)	(<1.7U)
MERCURY	---/---	(<0.01U)	(<0.01U)	(<0.01U)
SELENIUM	35/50	(<3.71U)	(<3.71U)	(<3.71U)
SILVER	35/---	(<0.7U)	(<0.7U)	(<0.7U)

SOIL SAMPLES

TPH-DRO BY MDHS-HETL LOP 4.1.25 (µg/Kg)

Analyte	ME PRG	TOP-02-01-GWI	TOP-02-02-GWI	TOP-02-03-GWI
DIESEL RANGE ORGANICS	10000	9200	8200	3600J

TPH-GRO BY MDHS-HETL LOP 4.2.17 (µg/Kg)

Analyte	ME PRG	TOP-02-01-GWI	TOP-02-02-GWI	TOP-02-03-GWI
GASOLINE RANGE ORGANICS	10000	(<2500U)	(<2500U)	3100

TARGET ANALYTE LIST ELEMENTS BY U.S. ENVIRONMENTAL PROTECTION AGENCY 6000/7000 SERIES METHODS (µg/Kg)

Analyte	ME RAGS	TOP-02-01-GWI	TOP-02-02-GWI	TOP-02-03-GWI
ARSENIC	10000	1600	2000	6200
BARIUM	10000000	16000	29800	21700
CADMIUM	27000	(<30U)	(<30U)	(<30U)
CHROMIUM	950000	7600	32600	10400

TARGET ANALYTE LIST ELEMENTS BY U.S. ENVIRONMENTAL PROTECTION AGENCY 6000/7000 SERIES METHODS (µg/Kg)

		TOP-02-01-GWI	TOP-02-02-GWI	TOP-02-03-GWI
LEAD	375000	2800	2400	5800
MERCURY	60000	20B*	20B*	340
SELENIUM	950000	(<380U)	(<350U)	(<330U)
SILVER	950000	(<70U)	(<70U)	(<60U)

FIELD QA/QC SAMPLES

VOLATILE ORGANIC COMPOUNDS BY U.S. ENVIRONMENTAL PROTECTION AGENCY METHOD 8260B (µg/L)

		Top-Trip-01-(GW)	Top-Trip-01-(Soil)
Analyte	**MEG/MCL**		
1,1-DICHLOROETHENE	---	(<1U)	(<5U)
1,2-DICHLOROETHANE	---	(<1U)	(<5U)
1,2-DICHLOROETHYLENE	---	(<2U)	(<10U)
2-BUTANONE	---	(<5U)	(<25U)
2-HEXANONE	---	(<5U)	(<25U)
ACETONE	---	(<5U)	(<25U)
BENZENE	---	(<1U)	(<5U)
BROMOMETHANE	---	(<2U)	(<10U)
CARBON DISULFIDE	---	(<1U)	(<5U)
CHLOROFORM	---	(<1U)	(<5U)
CHLOROMETHANE	---	(<2U)	(<10U)
CIS-1,2-DICHLOROETHENE	---	(<1U)	(<5U)
ETHYLBENZENE	---	(<1U)	(<5U)
M-,P-XYLENE	---	(<2U)	(<10U)
METHYLENE CHLORIDE	---	(<2U)	18B
O-XYLENE	---	(<1U)	(<5U)
TOLUENE	---	(<1U)	(<5U)
TRANS-1,2-DICHLOROETHENE	---	(<1U)	(<5U)
TRICHLOROETHENE	---	(<1U)	(<5U)
VINYL CHLORIDE	---	(<2U)	(<10U)
XYLENES (TOTAL)	---	(<3U)	(<15U)

TPH-DRO BY MDHS-HETL LOP 4.1.25 (µg/L)

		TOP-RINSE
Analyte	**MEG/MCL**	
DIESEL RANGE ORGANICS	---	60B

TPH-GRO BY MDHS-HETL LOP 4.2.17 (µg/L)

		TOP-RINSE
Analyte	**MEG/MCL**	
GASOLINE RANGE ORGANICS	---	26

TARGET ANALYTE LIST ELEMENTS BY U.S. ENVIRONMENTAL PROTECTION AGENCY 6000/7000 SERIES METHODS (µg/L)

		SS-SOURCE	TOP-RINSE
Analyte	**MEG/MCL**		
ARSENIC	---		(<3.03U)
BARIUM	---		1.5B*
CADMIUM	---		(<0.3U)
CHROMIUM	---		2.7B*
LEAD	---	(<1.7U)	(<1.7U)
MERCURY	---		(<0.01U)
SELENIUM	---		4B*
SILVER	---		(<0.7U)

MEG (Maximum Exposure Guideline) obtained from State of Maine Department of Human Services Maximum Exposure Guidelines, memorandum dated 23 October 1992. Dashes (---) indicate compound has no applicable MEG.
MCL (Maximum Contaminant Level) obtained from 40 CFR Parts 141 and 142 (U.S. EPA 1998). Dashes (---) indicate compound has no applicable MCL.
ME RAGS obtained from Remedial Action Guidelines for Contaminated Soils provided by the State of Maine Department of Environmental Protection. Residential Guidelines are used for the Project Action Level. Dashes (---) indicate compound has no applicable guideline.
ME PRG (Maine Petroleum Remediation Goal) taken from Procedural Guidelines for Establishing Action Levels and Remediation Goals for the Remediation of Oil Contaminated Soil and Groundwater in Maine, State of Maine Department of Environmental Protection, Rev 13, March 2000 .

NOTE:
DUP indicates a duplicate sample.
U = Not detected at or above the sample quantitation limit. Shown as (<____U).
B = Compound detected in associated method blank.
B* = Analyte concentration is between the Instrument Detection Limit and the Contract Required Detection Limit.
J = Estimated concentration.

Only those analytes detected in at least one of the samples are shown on this table.
Concentrations highlighted with gray and bold type denote exceedance of MEG, MCL, ME PRG, or ME RAGS.

TABLE 8 SUMMARY OF ANALYTICAL RESULTS FOR SAMPLES COLLECTED FROM BUILDING 380

SOIL SAMPLES

TARGET ANALYTE LIST ELEMENTS BY U.S. ENVIRONMENTAL PROTECTION AGENCY 6000/7000 SERIES METHODS (µg/Kg)

		SS-01-0604	SS-01-0604-(DUP)	SS-02-0604	SS-03-0604
Analyte	**ME RAGS**				
LEAD	375000	202000	190000	82900	49900

FIELD QA/QC SAMPLES

TARGET ANALYTE LIST ELEMENTS BY U.S. ENVIRONMENTAL PROTECTION AGENCY 6000/7000 SERIES METHODS (µg/L)

		SS-RINSE	SS-SOURCE
Analyte	**MEG/MCL**		
LEAD	---	(<1.7U)	(<1.7U)

ME RAGS obtained from Remedial Action Guidelines for Contaminated Soils provided by the State of Maine Department of Environmental Protection. Residential Guidelines are used for the Project Action Level. Dashes (---) indicate compound has no applicable guideline.

NOTE:
DUP indicates a duplicate sample.
U = Not detected at or above the sample quantitation limit. Shown as (<____U).

Only those analytes detected in at least one of the samples are shown on this table.

TOPSHAM ANNEX
2006 INVESTIGATION
DRAFT REPORT (SELECTED PAGES)

3.3.3 TOP-1 Investigation

Five direct-push soil borings were advanced within the TOP-1 area to collect soil samples for visual observation, field screening with a PID, and identification of site geology. The soil boring locations are shown on Appendix A, Figure 10 along with the locations of a previous soil investigation. The five direct push soil boring locations are identified as TOP-01-04 through TOP-01-09. The proposed locations were staked in the field and were chosen by uniformly expanding the data points for the TOP-1 area in all directions. A MEDEP representative conducted a site visit and he, along with the TtEC site geologist, determined the actual locations of the soil borings and monitoring wells prior to any installation. The actual locations are shown on Appendix A, Figure 10 and were determined with discussions between the site geologist and the MEDEP representative.

Two soil samples were collected for laboratory analysis from each of the direct-push soil boring locations, for a total of ten laboratory samples. One soil sample was collected from within the fill interval, and a second sample was collected at the groundwater interface. Each soil sample was analyzed for PCBs by USEPA Method 8082, VOC by Method 8260B, SVOC by Method 8270C, TPH-DRO by Maine Method 4.1.25 and 8 RCRA metals by USEPA Method 200/6000/7000 Series. For a summary of the laboratory results for the soil samples collected from the TOP-1 soil borings, refer to Table 3.

Three monitoring wells were also installed in the TOP-1 Area. The locations of the three monitoring wells are shown on Appendix A, Figure 10. They are identified as TOP-01-MW-01 through TOP-01-MW-03. These locations were chosen to optimize the ability to determine the groundwater flow direction and also to expand the overall data points for the TOP-1 area. The proposed locations were staked in the field and the actual locations as shown on Appendix A, Figure 10 were determined based upon site conditions, chemical and geologic information obtained from the soil borings, and discussions between the site geologist and MEDEP representative. The monitoring wells were completed to a depth determined in the field by the geologist during installation. The wells were extended to a depth that places the well just into the top of the marine clay that had been previously identified to exist on site. The monitoring wells were completed flush to grade and were constructed with five feet of schedule 40 polyvinyl chloride (PVC) 20-slot screen and solid PVC riser to the ground surface. The gravel pack that was utilized was sized to be compatible with the 20-slot screen.

The newly installed monitoring wells were developed according to TtEC's well development SOPs which are included in the Final Work Plan (R1) for TPH Soil Remediation and Investigation Activities (July 19, 2006) as Attachment A. The monitoring wells were developed no sooner than 24 hours after installation to remove sediments and drill cuttings from the area around the screened interval of the well. The monitoring wells were developed by pumping with a peristaltic pump.

One groundwater sample was collected from each of the three monitoring well locations for laboratory analysis. Each groundwater sample was analyzed for VOC by Method 8260B, SVOC by Method 8270C, TPH-DRO by Maine Method 4.1.25 and 8 RCRA metals by USEPA Method 200/6000/7000 Series. Due to the possible presence of vinyl chloride within the groundwater, VOC samples were analyzed on a 48-hour turn around time utilizing the standard 8260B method. If the immediate analysis yielded non-detect results for vinyl chloride, the remaining sample quantity was then analyzed by 8260B Select Ion Monitoring (SIM) methods for vinyl chloride to confirm that sample results were below MEDEP standards. For a summary of the laboratory results for the groundwater samples collected from the TOP-1 monitoring wells, refer to Table 4.

Results s/b in Ice

Groundwater flow direction was determined from water levels obtained during the monitoring well construction. Based upon these water levels and the survey data, the groundwater in the TOP-1 areas flows in a southeasterly direction.

TABLE 3
SOIL BORING
ANALYTICAL RESULTS
TOP-1 AREA
NASB - TOPSHAM ANNEX
TOPSHAM, MAINE

Parameter	TA-SB-04-03	TA-SB-04-08	TA-SB-05-10	TA-SB-05-15	TA-SB-06-05	TA-SB-06-09	TA-SB-07-05	TA-SB-07-08	TA-SB-08-05	TA-SB-08-08
	9/5/2006 (2.0' - 3.0')	9/5/2006 (7.0' - 8.0')	9/5/2006 (9.0' - 10.0')	9/5/2006 (14.0' - 15.0')	9/5/2006 (4.0' - 5.0')	9/5/2006 (8.0' - 9.0')	9/5/2006 (4.0' - 5.0')	9/5/2006 (7.0' - 8.0')	9/5/2006 (4.0' - 5.0')	9/5/2006 (7.0' - 9.0')
1,1,1,2-TETRACHLOROETHANE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,1,1-TRICHLOROETHANE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,1,2,2-TETRACHLOROETHANE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,1,2-TRICHLOROETHANE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,1-DICHLOROETHANE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,1-DICHLOROETHENE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,1-DICHLOROPROPENE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,2,3-TRICHLOROBENZENE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,2,3-TRICHLOROPROPANE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,2,4-TRICHLOROBENZENE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,2,4-TRIMETHYLBENZENE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,2-DIBROMO-3-CHLOROPROPANE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,2-DIBROMOETHANE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,2-DICHLOROBENZENE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,2-DICHLOROETHANE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,2-DICHLOROETHYLENE (TOTAL)	10 U	10 U	10 U	11 U	10 U	10 U	11 U	12 U	10 U	12 U
1,2-DICHLOROPROPANE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,3,5-TRICHLOROBENZENE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,3,5-TRIMETHYLBENZENE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,3-DICHLOROBENZENE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,3-DICHLOROPROPANE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
1,4-DICHLOROBENZENE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
2,2-DICHLOROPROPANE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
2-BUTANONE	25 U	25 U	26 U	26 U	25 U	25 U	28 U	31 UJ	26 U	25 U
2-CHLOROTOLUENE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
2-HEXANONE	25 U	25 U	26 U	26 U	25 U	25 U	28 U	31 U	26 U	30 U
4-CHLOROTOLUENE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
4-METHYL-2-PENTANONE	25 U	25 U	26 U	26 U	25 U	25 U	28 U	31 U	26 U	30 U
ACETONE	25 U	10 J	26 U	28 U	25 U	25 U	28 U	19 J	26 U	130
BENZENE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
BROMOBENZENE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
BROMOCHLOROMETHANE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
BROMODICHLOROMETHANE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
BROMOFORM	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
BROMOMETHANE	10 U	10 U	10 U	11 U	10 U	10 U	11 U	12 U	10 U	12 U
CARBON DISULFIDE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
CARBON TETRACHLORIDE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
CHLOROBENZENE	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
CHLOROETHANE	10 U	10 U	10 U	11 U	10 U	10 U	11 U	12 U	10 U	12 U
CHLOROFORM	5 U	5 U	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U
CHLOROMETHANE	10 U	10 U	10 U	11 U	10 U	10 U	11 U	12 U	10 U	12 U

TABLE 3
SOIL BORING
ANALYTICAL RESULTS
TOP-1 AREA
NASB - TOPSHAM ANNEX
TOPSHAM, MAINE

Parameter	TA-SB-04-03	TA-SB-04-08	TA-SB-05-10	TA-SB-05-15	TA-SB-06-05	TA-SB-06-09	TA-SB-07-05	TA-SB-07-08	TA-SB-08-05	TA-SB-08-08
	9/5/2006 (2.0' - 3.0')	9/5/2006 (7.0' - 8.0')	9/5/2006 (9.0' - 10.0')	9/5/2006 (14.0' - 15.0')	9/5/2006 (4.0' - 5.0')	9/5/2006 (8.0' - 9.0')	9/5/2006 (4.0' - 5.0')	9/5/2006 (7.0' - 8.0')	9/5/2006 (4.0' - 5.0')	9/5/2006 (7.0' - 8.0')
CIS-1,2-DICHLOROETHENE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
CIS-1,3-DICHLOROPROPENE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
DIBROMOCHLOROMETHANE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
DIBROMOMETHANE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
DICHLORODIFLUOROMETHANE	10 U	10 U	10 U	12 U	10 U	10 U	11 U	12 U	10 U	12 U
DIETHYL ETHER	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
ETHYLBENZENE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
HEXACHLOROBUTADIENE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
ISOPROPYLBENZENE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
M+P-XYLENES	10 U	10 U	10 U	12 U	10 U	10 U	11 U	12 U	10 U	12 U
METHYL TERT-BUTYL ETHER	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
METHYLENE CHLORIDE	**5 JB**	**7 JB**	**5 JB**	**6 JB**	**6 JB**	**6 JB**	**8 JB**	**17 JB**	**6 JB**	**6 JB**
NAPHTHALENE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
N-BUTYLBENZENE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
N-PROPYLBENZENE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
O-XYLENE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
P-ISOPROPYLTOLUENE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	11
SEC-BUTYLBENZENE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
STYRENE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
TERT-BUTYLBENZENE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
TETRACHLOROETHENE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
TETRAHYDROFURAN	50 U	50 U	52 U	65 U	50 U	50 U	56 U	60 U	52 U	60 U
TOLUENE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
TRANS-1,2-DICHLOROETHENE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
TRANS-1,3-DICHLOROPROPENE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
TRICHLOROETHENE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
TRICHLOROFLUOROMETHANE	**9**	**17**	**13**	**17**	**12**	**17**	**15**	**17**	**13**	**13**
VINYL ACETATE	5 U	5 U	5 U	6 U	5 U	5 U	6 U	6 U	5 U	6 U
VINYL CHLORIDE	10 U	10 U	10 U	12 U	10 U	10 U	11 U	12 U	10 U	12 U
XYLENES (TOTAL)	15 U	15 U	15 U	18 U	15 U	15 U	17 U	17 U	16 U	18 U

TABLE 3
SOIL BORING
ANALYTICAL RESULTS
TOP-1 AREA
NASB - TOPSHAM ANNEX
TOPSHAM, MAINE

Parameter	TA-SB-04-03	TA-SB-04-08	TA-SB-05-10	TA-SB-05-15	TA-SB-06-05	TA-SB-06-09	TA-SB-07-05	TA-SB-07-08	TA-SB-08-05	TA-SB-08-09
	9/5/2006 (2.0' - 3.0')	9/5/2006 (7.0' - 8.0')	9/5/2006 (9.0' - 10.0')	9/5/2006 (14.0' - 15.0')	9/5/2006 (4.0' - 5.0')	9/5/2006 (8.0' - 9.0')	9/5/2006 (4.0' - 5.0')	9/5/2006 (7.0' - 8.0')	9/5/2006 (4.0' - 5.0')	9/5/2006 (7.0' - 8.0')
1,2,4-TRICHLOROBENZENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
1,2-DICHLOROBENZENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
1,3-DICHLOROBENZENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	430 U
1,4-DICHLOROBENZENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
2,2'-OXYBIS(1-CHLOROPROPANE)	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
2,4,5-TRICHLOROPHENOL	980 U	980 U	1000 U	1000 U	870 U	990 U	920 U	1100 U	1000 U	NA
2,4,6-TRICHLOROPHENOL	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
2,4-DICHLOROPHENOL	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
2,4-DIMETHYLPHENOL	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
2,4-DINITROPHENOL	980 U	980 U	1000 U	1000 U	870 U	990 U	920 U	1100 U	1000 U	NA
2,4-DINITROTOLUENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
2,6-DINITROTOLUENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
2-CHLORONAPHTHALENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
2-CHLOROPHENOL	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	430 U
2-METHYLNAPHTHALENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
2-METHYLPHENOL	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
2-NITROANILINE	980 U	980 U	1000 U	1000 U	870 U	990 U	920 U	1100 U	1000 U	NA
2-NITROPHENOL	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
3&4-METHYLPHENOL	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
3,3'-DICHLOROBENZIDINE	980 U	980 U	1000 U	1000 U	870 U	990 U	920 U	1100 U	1000 U	NA
3-NITROANILINE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
4,6-DINITRO-2-METHYLPHENOL	980 U	980 U	1000 U	1000 U	870 U	990 U	920 U	1100 U	1000 U	NA
4-BROMOPHENYL-PHENYLETHER	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
4-CHLORO-3-METHYLPHENOL	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
4-CHLOROANILINE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
4-CHLOROPHENYL-PHENYLETHER	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
4-NITROANILINE	980 U	980 U	1000 U	1000 U	870 U	990 U	920 U	1100 U	1000 U	NA
4-NITROPHENOL	980 U	980 U	1000 U	1000 U	870 U	990 U	920 U	1100 U	1000 U	NA
ACENAPHTHENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
ACENAPHTHYLENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
ANTHRACENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
BENZO(A)ANTHRACENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
BENZO(A)PYRENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
BENZO(B)FLUORANTHENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
BENZO(G,H,I)PERYLENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
BENZO(K)FLUORANTHENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
BIS(2-CHLOROETHOXY)METHANE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA

TABLE 3
SOIL BORING
ANALYTICAL RESULTS
TOP-1 AREA
NASB - TOPSHAM ANNEX
TOPSHAM, MAINE

Parameter	TA-SB-04-03	TA-SB-04-08	TA-SB-05-10	TA-SB-06-15	TA-SB-06-05	TA-SB-06-09	TA-SB-07-05	TA-SB-07-08	TA-SB-08-05	TA-SB-08-08
	9/5/2006 (2.0' - 3.0')	9/5/2006 (7.0' - 8.0')	9/5/2006 (9.0' - 10.0')	9/5/2006 (14.0' - 15.0')	9/5/2006 (4.0' - 5.0')	9/5/2006 (8.0' - 9.0')	9/5/2006 (4.0' - 5.0')	9/5/2006 (7.0' - 8.0')	9/5/2006 (4.0' - 5.0')	9/5/2006 (7.0' - 8.0')
BIS(2-CHLOROETHYL)ETHER	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	430 U
BIS(2-ETHYLHEXYL)PHTHALATE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
BUTYLBENZYLPHTHALATE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
CARBAZOLE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
CHRYSENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
DIBENZO(A,H)ANTHRACENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
DIBENZOFURAN	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
DIETHYL PHTHALATE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
DIMETHYL PHTHALATE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
DI-N-BUTYL PHTHALATE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
DI-N-OCTYL PHTHALATE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
FLUORANTHENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
FLUORENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
HEXACHLOROBENZENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
HEXACHLOROBUTADIENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
HEXACHLOROCYCLOPENTADIENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
HEXACHLOROETHANE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
INDENO(1,2,3-CD)PYRENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
ISOPHORONE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
NAPHTHALENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
NITROBENZENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
N-NITROSO-DI-N-PROPYLAMINE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
N-NITROSODIPHENYLAMINE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
PENTACHLOROPHENOL	940 U	980 U	1000 U	1000 U	940 U	980 U	940 U	1100 U	1000 U	NA
PHENANTHRENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
PHENOL	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	430 U
PYRENE	380 U	400 U	410 U	420 U	350 U	400 U	370 U	430 U	410 U	NA
NAPHTHALENE	23 U	24 U	25 U	25 U	21 U	24 U	22 U	26 U	25 U	26 U
2-METHYLNAPHTHALENE	23 U	24 U	25 U	25 U	21 U	24 U	22 U	26 U	25 U	26 U
ACENAPHTHYLENE	23 U	24 U	25 U	25 U	21 U	24 U	22 U	26 U	25 U	26 U
ACENAPHTHENE	23 U	24 U	25 U	25 U	21 U	24 U	22 U	26 U	25 U	26 U
FLUORENE	23 U	24 U	25 U	25 U	21 U	24 U	22 U	26 U	25 U	26 U
PHENANTHRENE	3.8 J	5.8 J	25 U	25 U	5.6 J	6.5 J	22 U	26 U	4.4 J	16 J
ANTHRACENE	23 U	24 U	25 U	25 U	21 U	24 U	22 U	26 U	25 U	26 U
FLUORANTHENE	23 U	5.8 J	25 U	25 U	5.5 J	24 U	22 U	26 U	25 U	26
PYRENE	23 U	24 U	25 U	25 U	21 U	24 U	22 U	26 U	25 U	14 J
BENZO(A)ANTHRACENE	11 J	12 J	25 U	10 J	9.7 J	10 J	22 U	26 U	10 J	23 J

TABLE 3
SOIL BORING
ANALYTICAL RESULTS
TOP-1 AREA
NASB - TOPSHAM ANNEX
TOPSHAM, MAINE

Parameter	TA-SB-04-03	TA-SB-04-08	TA-SB-05-10	TA-SB-05-15	TA-SB-06-05	TA-SB-06-09	TA-SB-07-05	TA-SB-07-08	TA-SB-08-05	TA-SB-08-08
	9/5/2006 (2.0' - 3.0')	9/5/2006 (7.0' - 8.0')	9/5/2006 (9.0' - 10.0')	9/5/2006 (14.0' - 15.0')	9/5/2006 (4.0' - 5.0')	9/5/2006 (8.0' - 9.0')	9/5/2006 (4.0' - 5.0')	9/5/2006 (7.0' - 8.0')	9/5/2006 (4.0' - 5.0')	9/5/2006 (7.0' - 8.0')
CHRYSENE	4.3 J	24 U	25 U	25 U	3.9 J	24 U	22 U	26 U	25 U	26 U
BENZO(B)FLUORANTHENE	23 U	24 U	25 U	25 U	21 U	24 U	22 U	26 U	25 U	26 U
BENZO(K)FLUORANTHENE	23 U	24 U	25 U	25 U	21 U	24 U	22 U	26 U	25 U	26 U
BENZO(A)PYRENE	23 U	24 U	25 U	25 U	21 U	24 U	22 U	26 U	25 U	26 U
INDENO(1,2,3-CD)PYRENE	23 U	24 U	25 U	5.9 J	21 U	24 U	22 U	26 U	5.8 J	5.11 J
BENZO(G,H,I)PERYLENE	4.3 J	24 U	25 U	25 U	21 U	24 U	22 U	26 U	25 U	5.4 J
DIBENZO(A,H)ANTHRACENE	23 U	24 U	25 U	25 U	21 U	24 U	22 U	26 U	25 U	26 U
4,4'-DDD	3.8 U	4.0 U	4.1 U	4.2 U	3.5 U	4.0 U	3.7 U	4.3 U	4.1 U	4.3 U
4,4'-DDE	3.8 U	4.0 U	4.1 U	4.2 U	3.5 U	4.0 U	3.7 U	4.3 U	4.1 U	4.3 U
4,4'-DDT	3.8 U	4.0 U	4.1 U	4.2 U	3.5 U	4.0 U	3.7 U	4.3 U	4.1 U	4.3 U
ALDRIN	1.9 U	2.0 U	2.1 U	2.1 U	1.8 U	2.0 U	1.9 U	2.2 U	2.1 U	2.2 U
ALPHA-BHC	1.9 U	2.0 U	2.1 U	2.1 U	1.8 U	2.0 U	1.9 U	2.2 U	2.1 U	2.2 U
ALPHA-CHLORDANE	1.9 U	2.0 U	2.1 U	2.1 U	1.8 U	2.0 U	1.9 U	2.2 U	2.1 U	2.2 U
AROCLOR-1016	19 U	20 U	21 U	21 U	18 U	20 U	19 U	22 U	21 U	22 U
AROCLOR-1221	19 U	20 U	21 U	21 U	18 U	20 U	19 U	22 U	21 U	22 U
AROCLOR-1232	19 U	20 U	21 U	21 U	18 U	20 U	19 U	22 U	21 U	22 U
AROCLOR-1242	19 U	20 U	21 U	21 U	18 U	20 U	19 U	22 U	21 U	22 U
AROCLOR-1248	19 U	20 U	21 U	21 U	18 U	20 U	19 U	22 U	21 U	22 U
AROCLOR-1254	19 U	20 U	21 U	21 U	18 U	20 U	19 U	22 U	21 U	22 U
AROCLOR-1260	19 U	20 U	21 U	21 U	18 U	20 U	19 U	22 U	21 U	22 U
BETA-BHC	1.9 U	2.0 U	2.1 U	2.1 U	1.8 U	2.0 U	1.9 U	2.2 U	2.1 U	2.2 U
DELTA-BHC	1.9 U	2.0 U	2.1 U	2.1 U	1.8 U	2.0 U	1.9 U	2.2 U	2.1 U	2.2 U
DIELDRIN	3.8 U	4.0 U	4.1 U	4.2 U	3.5 U	4.0 U	3.7 U	4.3 U	4.1 U	4.3 U
ENDOSULFAN I	1.9 U	2.0 U	2.1 U	2.1 U	1.8 U	2.0 U	1.9 U	2.2 U	2.1 U	2.2 U
ENDOSULFAN II	3.8 U	4.0 U	4.1 U	4.2 U	3.5 U	4.0 U	3.7 U	4.3 U	4.1 U	4.3 U
ENDOSULFAN SULFATE	3.8 U	4.0 U	4.1 U	4.2 U	3.5 U	4.0 U	3.7 U	4.3 U	4.1 U	4.3 U
ENDRIN	3.8 U	4.0 U	4.1 U	4.2 U	3.5 U	4.0 U	3.7 U	4.3 U	4.1 U	4.3 U
ENDRIN ALDEHYDE	3.8 U	4.0 U	4.1 U	4.2 U	3.5 U	4.0 U	3.7 U	4.3 U	4.1 U	4.3 U
ENDRIN KETONE	3.8 U	4.0 U	4.1 U	4.2 U	3.5 U	4.0 U	3.7 U	4.3 U	4.1 U	4.3 U
GAMMA-BHC	1.9 U	2.0 U	2.1 U	2.1 U	1.8 U	2.0 U	1.9 U	2.2 U	2.1 U	2.2 U
GAMMA-CHLORDANE	1.9 U	2.0 U	2.1 U	2.1 U	1.8 U	2.0 U	1.9 U	2.2 U	2.1 U	2.2 U
HEPTACHLOR	1.9 U	2.0 U	2.1 U	2.1 U	1.8 U	2.0 U	1.9 U	2.2 U	2.1 U	2.2 U
HEPTACHLOR EPOXIDE	1.9 U	2.0 U	2.1 U	2.1 U	1.8 U	2.0 U	1.9 U	2.2 U	2.1 U	2.2 U
METHOXYCHLOR	19 U	20 U	21 U	21 U	18 U	20 U	19 U	22 U	21 U	22 U
TOXAPHENE	38 U	40 U	41 U	42 U	35 U	40 U	37 U	43 U	41 U	43 U

TABLE 3
SOIL BORING
ANALYTICAL RESULTS
TOP-1 AREA
NASB - TOPSHAM ANNEX
TOPSHAM, MAINE

Parameter	TA-SB-04-03	TA-SB-04-08	TA-SB-05-10	TA-SB-05-15	TA-SB-06-05	TA-SB-06-09	TA-SB-07-05	TA-SB-07-08	TA-SB-08-05	TA-SB-08-09
	9/5/2006 (2.0' - 3.0')	9/5/2006 (7.0' - 8.0')	9/5/2006 (9.0' - 10.0')	9/5/2006 (14.0' - 15.0')	9/5/2006 (4.0' - 5.0')	9/5/2006 (8.0' - 9.0')	9/5/2006 (4.0' - 5.0')	9/5/2006 (7.0' - 8.0')	9/5/2006 (4.0' - 5.0')	9/5/2006 (7.0' - 8.0')
ARSENIC, TOTAL	9.0	[illegible]	16.6	[illegible]	2.1	[illegible]	1.7	[illegible]	10.2	[illegible]
BARIUM, TOTAL	92.8	[illegible]	117	[illegible]	20.1	[illegible]	16.8	[illegible]	128	75.0
BERYLLIUM, TOTAL	0.67	[illegible]	1.0	[illegible]	0.26 B	[illegible]	0.26 B	[illegible]	0.84	0.89
CADMIUM, TOTAL	0.09 U	[illegible]	0.09 U	[illegible]	0.04 U	[illegible]	0.04 B	[illegible]	0.09 U	0.27 B
CHROMIUM, TOTAL	43.3	[illegible]	65.9	[illegible]	9.9	[illegible]	8.1	[illegible]	52.6	36.5
LEAD, TOTAL	9.2	[illegible]	15.1	[illegible]	3.3	[illegible]	2.5	[illegible]	10.4	14.7
MERCURY, TOTAL	0.01 U	[illegible]	0.01 U	[illegible]	0.01 U	[illegible]	0.01 U	[illegible]	0.01 U	0.01 B
SELENIUM, TOTAL	1.09 U	[illegible]	1.00 U	[illegible]	0.42 U	[illegible]	0.41 U	[illegible]	1.04 U	0.53 U
SILVER, TOTAL	0.14 U	[illegible]	0.13 U	[illegible]	0.11 U	[illegible]	0.10 U	[illegible]	0.13 U	0.10 U
ORO	12	[illegible]	2.8 J	[illegible]	3.5 J	[illegible]	3.2 J	[illegible]	5.1 J	[illegible]
Total Solids (%)	87	[illegible]	81	[illegible]	94	[illegible]	89	[illegible]	80	70

Notes:

U = Indicates the compound was analyzed for but not detected above the laboratory Practical Quantitation Limit.

J = Estimated value. The analyte was detected in the sample at a concentration less than the laboratory Practical Quantitation Limit (PQL), but above the Method Detection Limit (MDL).

B = Indicates the analyte was detected in the laboratory method blank analyzed concurrently with the sample.

NA = Not applicable/not available

TABLE 4

MONITORING WELL
ANALYTICAL RESULTS
TOP-1 AREA
NASB - TOPSHAM ANNEX
TOPSHAM, MAINE

Parameter	MW-01	MW-01 (RE)	MW-02	MW-02 (RE)	MW-03	MW-03 (RE)
	9/26/2006	9/26/2006	9/27/2006	9/27/2006	9/27/2006	9/27/2006
1,1,1,2-TETRACHLOROETHANE	5 U	NA	5 U	NA	5 U	NA
1,1,1-TRICHLOROETHANE	5 U	NA	5 U	NA	5 U	NA
1,1,2,2-TETRACHLOROETHANE	5 U	NA	5 U	NA	5 U	NA
1,1,2-TRICHLOROETHANE	5 U	NA	5 U	NA	5 U	NA
1,1-DICHLOROETHANE	5 U	NA	5 U	NA	5 U	NA
1,1-DICHLOROETHENE	5 U	NA	5 U	NA	5 U	NA
1,1-DICHLOROPROPENE	5 U	NA	5 U	NA	5 U	NA
1,2,3-TRICHLOROBENZENE	5 U	NA	5 U	NA	5 U	NA
1,2,3-TRICHLOROPROPANE	5 U	NA	5 U	NA	5 U	NA
1,2,4-TRICHLOROBENZENE	5 U	NA	5 U	NA	5 U	NA
1,2,4-TRIMETHYLBENZENE	5 U	NA	5 U	NA	5 U	NA
1,2-DIBROMO-3-CHLOROPROPANE	5 U	NA	5 U	NA	5 U	NA
1,2-DIBROMOETHANE	5 U	NA	5 U	NA	5 U	NA
1,2-DICHLOROBENZENE	5 U	NA	5 U	NA	5 U	NA
1,2-DICHLOROETHANE	5 U	NA	5 U	NA	5 U	NA
1,2-DICHLOROETHYLENE (TOTAL)	10 U	NA	10 U	NA	10 U	NA
1,2-DICHLOROPROPANE	5 U	NA	5 U	NA	5 U	NA
1,3,5-TRICHLOROBENZENE	5 U	NA	5 U	NA	5 U	NA
1,3,5-TRIMETHYLBENZENE	5 U	NA	5 U	NA	5 U	NA
1,3-DICHLOROBENZENE	5 U	NA	5 U	NA	5 U	NA
1,3-DICHLOROPROPANE	5 U	NA	5 U	NA	5 U	NA
1,4-DICHLOROBENZENE	5 U	NA	5 U	NA	5 U	NA
2,2-DICHLOROPROPANE	5 U	NA	5 U	NA	5 U	NA
2-BUTANONE	25 U	NA	25 U	NA	25 U	NA
2-CHLOROTOLUENE	5 U	NA	5 U	NA	5 U	NA
2-HEXANONE	25 U	NA	25 U	NA	25 U	NA
4-CHLOROTOLUENE	5 U	NA	5 U	NA	5 U	NA
4-METHYL-2-PENTANONE	25 U	NA	25 U	NA	25 U	NA
ACETONE	25 U	NA	25 U	NA	25 U	NA
BENZENE	5 U	NA	5 U	NA	5 U	NA
BROMOBENZENE	5 U	NA	5 U	NA	5 U	NA
BROMOCHLOROMETHANE	5 U	NA	5 U	NA	5 U	NA
BROMODICHLOROMETHANE	5 U	NA	5 U	NA	5 U	NA
BROMOFORM	5 U	NA	5 U	NA	5 U	NA
BROMOMETHANE	10 U	NA	10 U	NA	10 U	NA
CARBON DISULFIDE	5 U	NA	5 U	NA	5 U	NA
CARBON TETRACHLORIDE	5 U	NA	5 U	NA	5 U	NA
CHLOROBENZENE	5 U	NA	5 U	NA	5 U	NA
CHLOROETHANE	10 U	NA	10 U	NA	10 U	NA
CHLOROFORM	5 U	NA	5 U	NA	5 U	NA
CHLOROMETHANE	10 U	NA	10 U	NA	10 U	NA
CIS-1,2-DICHLOROETHENE	5 U	NA	5 U	NA	5 U	NA
CIS-1,3-DICHLOROPROPENE	5 U	NA	5 U	NA	5 U	NA
DIBROMOCHLOROMETHANE	5 U	NA	5 U	NA	5 U	NA
DIBROMOMETHANE	5 U	NA	5 U	NA	5 U	NA
DICHLORODIFLUOROMETHANE	10 U	NA	10 U	NA	10 U	NA
DIETHYL ETHER	5 U	NA	5 U	NA	5 U	NA
ETHYLBENZENE	5 U	NA	5 U	NA	5 U	NA
HEXACHLOROBUTADIENE	5 U	NA	5 U	NA	5 U	NA
ISOPROPYLBENZENE	5 U	NA	5 U	NA	5 U	NA

TABLE 4

MONITORING WELL
ANALYTICAL RESULTS
TOP-1 AREA
NASB - TOPSHAM ANNEX
TOPSHAM, MAINE

Parameter	MW-01	MW-01 (RE)	MW-02	MW-02 (RE)	MW-03	MW-03 (RE)
	9/26/2006	9/26/2006	9/27/2006	9/27/2006	9/27/2006	9/27/2006
M+P-XYLENES	10 U	NA	10 U	NA	10 U	NA
METHYL TERT-BUTYL ETHER	5 U	NA	5 U	NA	5 U	NA
METHYLENE CHLORIDE	5 U	NA	5 U	NA	5 U	NA
NAPHTHALENE	5 U	NA	5 U	NA	5 U	NA
N-BUTYLBENZENE	5 U	NA	5 U	NA	5 U	NA
N-PROPYLBENZENE	5 U	NA	5 U	NA	5 U	NA
O-XYLENE	5 U	NA	5 U	NA	5 U	NA
P-ISOPROPYLTOLUENE	5 U	NA	5 U	NA	5 U	NA
SEC-BUTYLBENZENE	5 U	NA	5 U	NA	5 U	NA
STYRENE	5 U	NA	5 U	NA	5 U	NA
TERT-BUTYLBENZENE	5 U	NA	5 U	NA	5 U	NA
TETRACHLOROETHENE	5 U	NA	5 U	NA	5 U	NA
TETRAHYDROFURAN	25 U	NA	25 U	NA	25 U	NA
TOLUENE	5 U	NA	5 U	NA	5 U	NA
TRANS-1,2-DICHLOROETHENE	5 U	NA	5 U	NA	5 U	NA
TRANS-1,3-DICHLOROPROPENE	5 U	NA	5 U	NA	5 U	NA
TRICHLOROETHENE	5 U	NA	5 U	NA	5 U	NA
TRICHLOROFLUOROMETHANE	10 U	NA	10 U	NA	10 U	NA
VINYL ACETATE	5 U	NA	5 U	NA	5 U	NA
VINYL CHLORIDE	10 U	NA	10 U	NA	10 U	NA
XYLENES (TOTAL)	15 U	NA	15 U	NA	15 U	NA
VINYL CHLORIDE	0.15 U	NA	0.15 U	NA	0.15 U	NA

TABLE 4

MONITORING WELL
ANALYTICAL RESULTS
TOP-1 AREA
NASB - TOPSHAM ANNEX
TOPSHAM, MAINE

Parameter	MW-01	MW-01 (RE)	MW-02	MW-02 (RE)	MW-03	MW-03 (RE)
	9/26/2006	9/26/2006	9/27/2006	9/27/2006	9/27/2006	9/27/2006
1,2,4-TRICHLOROBENZENE	10 U	10 U	10 U	10 U	10 U	10 U
1,2-DICHLOROBENZENE	10 U	10 U	10 U	10 U	10 U	10 U
1,3-DICHLOROBENZENE	10 U	10 U	10 U	10 U	10 U	10 U
1,4-DICHLOROBENZENE	10 U	10 U	10 U	10 U	10 U	10 U
2,2'-OXYBIS(1-CHLOROPROPANE)	10 U	10 U	10 U	10 U	10 U	10 U
2,4,5-TRICHLOROPHENOL	26 U	26 U	25 U	25 U	26 U	26 U
2,4,6-TRICHLOROPHENOL	10 U	10 U	10 U	10 U	10 U	10 U
2,4-DICHLOROPHENOL	10 U	10 U	10 U	10 U	10 U	10 U
2,4-DIMETHYLPHENOL	10 U	10 U	10 U	10 U	10 U	10 U
2,4-DINITROPHENOL	26 U	26 U	25 U	25 U	26 U	26 U
2,4-DINITROTOLUENE	10 U	10 U	10 U	10 U	10 U	10 U
2,6-DINITROTOLUENE	10 U	10 U	10 U	10 U	10 U	10 U
2-CHLORONAPHTHALENE	10 U	10 U	10 U	10 U	10 U	10 U
2-CHLOROPHENOL	10 U	10 U	10 U	10 U	10 U	10 U
2-METHYLNAPHTHALENE	10 U	10 U	10 U	10 U	10 U	10 U
2-METHYLPHENOL	10 U	10 U	10 U	10 U	10 U	10 U
2-NITROANILINE	26 U	26 U	25 U	25 U	26 U	26 U
2-NITROPHENOL	10 U	10 U	10 U	10 U	10 U	10 U
3&4-METHYLPHENOL	10 U	10 U	10 U	10 U	10 U	10 U
3,3'-DICHLOROBENZIDINE	10 U	10 U	10 U	10 U	10 U	10 U
3-NITROANILINE	26 U	26 U	25 U	25 U	26 U	26 U
4,6-DINITRO-2-METHYLPHENOL	26 U	26 U	25 U	25 U	26 U	26 U
4-BROMOPHENYL-PHENYLETHER	10 U	10 U	10 U	10 U	10 U	10 U
4-CHLORO-3-METHYLPHENOL	26 U	26 U	25 U	25 U	26 U	26 U
4-CHLOROANILINE	10 U	10 U	10 U	10 U	10 U	10 U
4-CHLOROPHENYL-PHENYLETHER	10 U	10 U	10 U	10 U	10 U	10 U
4-NITROANILINE	26 U	26 U	25 U	25 U	26 U	26 U
4-NITROPHENOL	10 U	10 U	10 U	10 U	10 U	10 U
ACENAPHTHENE	10 U	10 U	10 U	10 U	10 U	10 U
ACENAPHTHYLENE	10 U	10 U	10 U	10 U	10 U	10 U
ANTHRACENE	10 U	10 U	10 U	10 U	10 U	10 U
BENZO(A)ANTHRACENE	10 U	10 U	10 U	10 U	10 U	10 U
BENZO(A)PYRENE	10 U	10 U	10 U	10 U	10 U	10 U
BENZO(B)FLUORANTHENE	10 U	10 U	10 U	10 U	10 U	10 U
BENZO(G,H,I)PERYLENE	10 U	10 U	10 U	10 U	10 U	10 U
BENZO(K)FLUORANTHENE	10 U	10 U	10 U	10 U	10 U	10 U
BIS(2-CHLOROETHOXY)METHANE	10 U	10 U	10 U	10 U	10 U	10 U
BIS(2-CHLOROETHYL)ETHER	10 U	10 U	10 U	10 U	10 U	10 U
BIS(2-ETHYLHEXYL)PHTHALATE	10 U	10 U	10 U	10 U	10 U	10 U
BUTYLBENZYLPHTHALATE	10 U	10 U	10 U	10 U	10 U	10 U
CARBAZOLE	10 U	10 U	10 U	10 U	10 U	10 U
CHRYSENE	10 U	10 U	10 U	10 U	10 U	10 U
DIBENZO(A,H)ANTHRACENE	10 U	10 U	10 U	10 U	10 U	10 U
DIBENZOFURAN	10 U	10 U	10 U	10 U	10 U	10 U
DIETHYL PHTHALATE	10 U	10 U	10 U	10 U	10 U	10 U
DIMETHYL PHTHALATE	10 U	10 U	10 U	10 U	10 U	10 U
DI-N-BUTYL PHTHALATE	10 U	10 U	10 U	10 U	10 U	10 U

TABLE 4

MONITORING WELL
ANALYTICAL RESULTS
TOP-1 AREA
NASB - TOPSHAM ANNEX
TOPSHAM, MAINE

Parameter	MW-01 9/26/2006	MW-01 (RE) 9/26/2006	MW-02 9/27/2006	MW-02 (RE) 9/27/2006	MW-03 9/27/2006	MW-03 (RE) 9/27/2006
DI-N-OCTYL PHTHALATE	10 U	10 U	10 U	10 U	10 U	10 U
FLUORANTHENE	10 U	10 U	10 U	10 U	10 U	10 U
FLUORENE	10 U	10 U	10 U	10 U	10 U	10 U
HEXACHLOROBENZENE	10 U	10 U	10 U	10 U	10 U	10 U
HEXACHLOROBUTADIENE	10 U	10 U	10 U	10 U	10 U	10 U
HEXACHLOROCYCLOPENTADIENE	10 U	10 U	10 U	10 U	10 U	10 U
HEXACHLOROETHANE	10 U	10 U	10 U	10 U	10 U	10 U
INDENO(1,2,3-CD)PYRENE	10 U	10 U	10 U	10 U	10 U	10 U
ISOPHORONE	10 U	10 U	10 U	10 U	10 U	10 U
NAPHTHALENE	10 U	10 U	10 U	10 U	10 U	10 U
NITROBENZENE	10 U	10 U	10 U	10 U	10 U	10 U
N-NITROSO-DI-N-PROPYLAMINE	10 U	10 U	10 U	10 U	10 U	10 U
N-NITROSODIPHENYLAMINE	10 U	10 U	10 U	10 U	10 U	10 U
PENTACHLOROPHENOL	26 U	25 U	25 U	26 U	26 U	26 U
PHENANTHRENE	10 U	10 U	10 U	10 U	10 U	10 U
PHENOL	10 U	10 U	10 U	10 U	10 U	10 U
PYRENE	10 U	10 U	10 U	10 U	10 U	10 U
AROCLOR-1016	0.50 U	NA	0.53 U	NA	0.50 U	NA
AROCLOR-1221	0.50 U	NA	0.53 U	NA	0.50 U	NA
AROCLOR-1232	0.50 U	NA	0.53 U	NA	0.50 U	NA
AROCLOR-1242	0.50 U	NA	0.53 U	NA	0.50 U	NA
AROCLOR-1248	0.50 U	NA	0.53 U	NA	0.50 U	NA
AROCLOR-1254	0.50 U	NA	0.53 U	NA	0.50 U	NA
AROCLOR-1260	0.50 U	NA	0.53 U	NA	0.50 U	NA
ARSENIC, TOTAL	3.09 U	NA	6.1 B	NA	4.9 B	NA
BARIUM, TOTAL	**36.4**	NA	**38.0**	NA	**117**	NA
BERYLLIUM, TOTAL	0.84 B	NA	0.73 B	NA	0.96 B	NA
CADMIUM, TOTAL	0.50 U	NA	0.50 U	NA	0.50 U	NA
CHROMIUM, TOTAL	0.69 U	NA	10.0 B	NA	9.6 B	NA
LEAD, TOTAL	1.80 U	NA	**7.2**	NA	4.6 B	NA
MERCURY, TOTAL	0.01 U	NA	0.01 U	NA	0.01 U	NA
SELENIUM, TOTAL	4.04 U	NA	4.04 U	NA	6.1 B	NA
SILVER, TOTAL	1.21 U	NA	1.21 U	NA	1.21 U	NA
DRO	**44 J**	NA	**48**	NA	**75**	NA

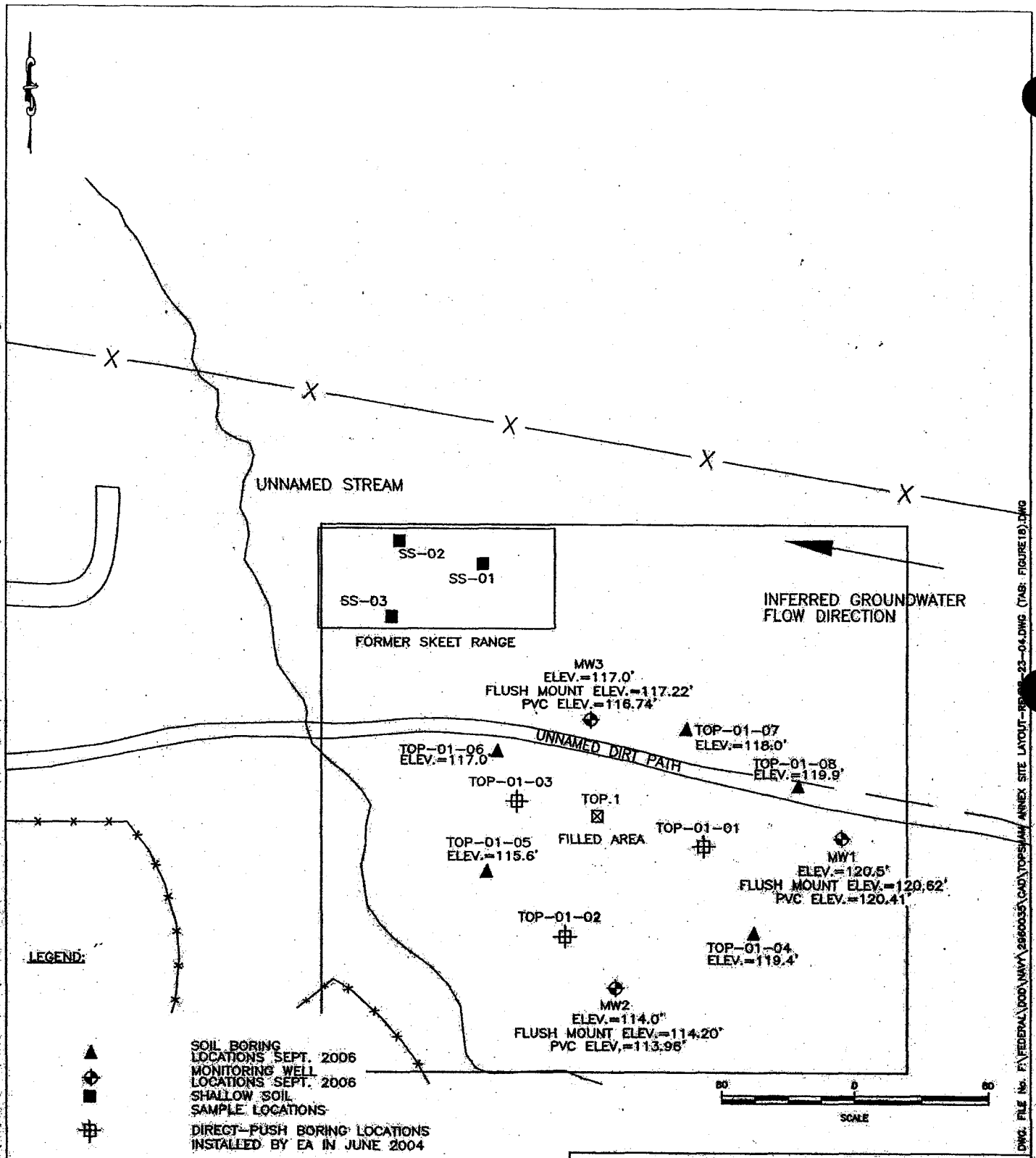
Notes:

U = Indicates the compound was analyzed for but not detected above the laboratory Practical Quantitation Limit.

J = Estimated value. The analyte was detected in the sample at a concentration less than the laboratory Practical Quantitation Limit (PQL), but above the Method Detection Limit (MDL).

B = Indicates the analyte was detected in the laboratory method blank analyzed concurrently with the sample.

NA = Not applicable/not available



LEGEND:

- SOIL BORING LOCATIONS SEPT. 2006
- MONITORING WELL LOCATIONS SEPT. 2006
- SHALLOW SOIL SAMPLE LOCATIONS
- DIRECT-PUSH BORING LOCATIONS INSTALLED BY EA IN JUNE 2004
- TOP.1 AREA
- CHAIN LINK FENCE
- INFERRED GROUNDWATER FLOW DIRECTION IS BASED ON REGIONAL GROUNWATER FLOW

NOTES:

1. THE LOCATION OF FORMER SKEET RANGE WAS TAKEN FROM AN OLD VICINITY PLAN AND IS APPROXIMATE.
2. FOSTER WHEELER ENVIRONMENTAL CORPORATION, FINAL REMOVAL ACTION TOPSHAM ANNEX PARCEL 2, TOPSHAM MAINE, MAY 2001.
3. INFERRED GROUNDWATER FLOW DIRECTION IS BASED ON REGIONAL GROUNDWATER FLOW.

FIGURE 1.0
TOP-01 AS BUILT
FORMER BUILDING 380 (SKEET RANGE)
TOPSHAM ANNEX
TOPSHAM, MAINE

DESIGN SAP	EA®	DATE 25 JAN 06
DRAWN SAP		SCALE AS SHOWN
CHECKED AE		PROJECT NO. 29600.35
PROJECT MANAGER AE		SHEET NO. --

P:\Navy RAC\BrunswickNAS\Topsham\Topsham\AsBuilt for Closeout Report\Topsham Annex Site Layout-REV11-17-2006.dwg

DWG. FILE No. F:\FEDERAL\DOD\NAVY\29600.35\CAD\TOPSHAM ANNEX SITE LAYOUT-REV06-23-04.DWG (TAB: FIGURE1B).DWG

ARDAMAN & ASSOCIATES, INC.
Geotechnical, Environmental and
Materials Consultants

BORING LOG

DEPTH FT.	BLOWS PER 6"	'N' VALUE	SAMPLE NO.	DESCRIPTION OF MATERIAL	% RECOVERY
				0-3.5' SANDY, FILL-LIKE MATERIAL NO ODOR; PID=0.0 ppm; 4 GAS= NO BLEEDING BRIDGES COLLECT TA-SB-04-03 FROM 2-3' INTERVAL	
				3.5-8' BROWN SILTY-CLAY	
5				NO-ODOR; PID=0.0 ppm; 4 GAS= N/R COLLECT TA-SB-04-08 FROM 7-8' INTERVAL	
				8'-15' GRAY/BLUE CLAY NO ODOR PID=0.0 ppm; 4 GAS= N/R	
10					
				15' END OF DIRECT PUSH	
20					
25					
30					

GENERAL DATA

FILE NO. TOP-01-04
CLIENT: NGDC/NMY
SITE: TOP-1
HOLE NO. 4 SHEET 1 OF 1
LOCATION OF BORING
DATE 9/5/06

CASING:
INSIDE DIAMETER n/a IN.
OUTSIDE DIAMETER n/a IN.
DEPTH OF CASING n/a FT.

SPOON:
INSIDE DIAMETER 2 IN.
OUTSIDE DIAMETER n/a IN.

HAMMER:
HAMMER WEIGHT n/a LB.
DROP OF HAMMER n/a IN.

ELEVATION:
GROUND SURFACE 119.4 FT.

GWL:
DATE 1st n/a
DATE 2nd n/a
ACTUAL ELEVATION n/a

DRILLING CREW:
GeoSearch

REMARKS:
SAMPLES
TA-SB-04-03
TA-SB-04-08

A0154A (1/92)

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BORING LOG

DEPTH FT.	BLOWS PER 6"	"N" VALUE	SAMPLE NO.	DESCRIPTION OF MATERIAL	% RECOVERY
				SANDY FILL-LIKE MATERIAL 0-5' NO ODOR ; PID=0.0ppm ; H-GAS - NO READINGS ABOVE BACKGROUND	
5				5'-8' - BROWN SILTY CLAY	
				8'-15' - GRAY-BLUE MARINE CLAY - MOIST/COHESIVE	
10				NO ODOR. PID=0.0 ppm H-GAS = NO READINGS ABOVE BACKGROUND COLLECT TA-SB-05-10 FROM 9-10' INTERVAL	
15				COLLECT TA-SB-05-15 FROM 14-15' INTERVAL	
				15' END OF DIRECT PUSH	
20					
25					
30					
35					

ADM44A (1/90)

GENERAL DATA

FILE NO. TOP-01-05
CLIENT: NEDEP/NAVY
SITE: TOBYHANNA - TOP-1
HOLE NO. 5 SHEET 1 OF 1
LOCATION OF BORING
DATE 9/5/06

CASING:
INSIDE DIAMETER N/A IN.
OUTSIDE DIAMETER N/A IN.
DEPTH OF CASING N/A FT.

SPOON:
INSIDE DIAMETER 2 IN.
OUTSIDE DIAMETER ~~N/A~~ IN.

HAMMER:
HAMMER WEIGHT N/A LB.
DROP OF HAMMER N/A IN.

ELEVATION:
GROUND SURFACE 115.6' FT.

GWL:
DATE 1st N/A
DATE 2nd N/A
ACTUAL ELEVATION N/A

DRILLING CREW:
GEOSEARCH
CHRIS + JOHN

REMARKS:
SAMPLES COLLECTED @:
9-10' INTERVAL
14-15' INTERVAL

ARDAMAN & ASSOCIATES, INC.
Geotechnical, Environmental and
Materials Consultants

BORING LOG

DEPTH FT.	BLOWS PER 6"	'N' VALUE	SAMPLE NO.	DESCRIPTION OF MATERIAL	% RECOVERY
				0-5' SANDY FILL-LIKE MATERIAL NO ODOR; PID=0.0 ppm; 4GAS=N/R	
5				COLLECT TA-SB-06-05 FROM 4-5' INTERVAL	
				5'-8' BROWN SILTY CLAY NO ODOR; PID=0.0 ppm; 4GAS=N/R	
				8'-15' COLLECT TA-SB-06-09 FROM 8-9' INTERVAL	
10				BLUE GRAY CLAY NO ODOR; PID=0.0 ppm; 4GAS=N/R MOIST; COHESIVE	
				15' END OF DIRECT PUSH	
20					
25					
30					
35					

A0154A (1/92)

GENERAL DATA

FILE NO. TOP-01-06
CLIENT: Moser/MMV
SITE: TOP-1
HOLE NO. 6 SHEET 1 OF 1
LOCATION OF BORING
DATE 9/5/06

CASING:
INSIDE DIAMETER N/A IN.
OUTSIDE DIAMETER N/A IN.
DEPTH OF CASING N/A FT.

SPOON:
INSIDE DIAMETER 2 IN.
OUTSIDE DIAMETER N/A IN.

HAMMER:
HAMMER WEIGHT N/A LB.
DROP OF HAMMER N/A IN.

ELEVATION:
GROUND SURFACE 117.0 FT.

GWL:
DATE 1st N/A
DATE 2nd N/A
ACTUAL ELEVATION N/A

DRILLING CREW:
GeoSearch

REMARKS:
SAMPLES
TA-SB-06-05
TA-SB-06-09

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Materials Consultants

BORING LOG

DEPTH FT.	BLOWS PER 6"	'N' VALUE	SAMPLE NO.	DESCRIPTION OF MATERIAL	% RECOVERY
				0-5' SANDY FILL-LIKE MATERIAL NO ODOR; PID=0.0 PPM; %GAS=N/R	
5				COLLECT TA-SB-07-05 FROM 4-5' INTERVAL	
				5'-7.5' BROWN-SILTY CLAY NO ODOR; PID=0.0 PPM; %GAS=N/R	
				COLLECT TA-SB-07-08 FROM 7-8' INTERVAL	
10				7.5-15' BLUE GRAY MARINE CLAY MOIST; COHESIVE NO ODOR; NO SHEEN; PID=0.0 PPM; %GAS=N/R	
15				15' END OF DIRECT PUSH	
20					
25					
30					
35					

AD154A (1/90)

GENERAL DATA

FILE NO. TOP-01-07
CLIENT: NEDES/NAVY
SITE: TOP-1
HOLE NO. 7 SHEET 1 OF 1
LOCATION OF BORING
DATE 9/5/06

CASING:
INSIDE DIAMETER N/A IN.
OUTSIDE DIAMETER N/A IN.
DEPTH OF CASING N/A FT.

SPOON:
INSIDE DIAMETER 2 IN.
OUTSIDE DIAMETER N/A IN.

HAMMER:
HAMMER WEIGHT N/A LB.
DROP OF HAMMER N/A IN.

ELEVATION:
GROUND SURFACE 118 FT.

GWL:
DATE 1st N/A
DATE 2nd N/A
ACTUAL ELEVATION N/A

DRILLING CREW:
GEOSEARCH

REMARKS:
SAMPLED
TA-SB-07-05
TA-SB-07-08

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BORING LOG

DEPTH FT.	BLOWS PER 6"	'N' VALUE	SAMPLE NO.	DESCRIPTION OF MATERIAL	% RECOVERY
				0-5' SANDY FILL-LIKE MATERIAL NO ODOR; PID=0.0PPM; %GAS=N/R	
5				COLLECT TA-SB-08-05 FROM 4-5' INTERVAL	
				5-7.5' BROWN SILTY CLAY NO ODOR; PID=0.0PPM; %GAS=N/R	
				COLLECT TA-SB-08-08 FROM 7-8' INTERVAL	
10				7.5-15' BLUE GRAY CLAY MOIST; COHESIVE NO ODOR; PID=0.0PPM; %GAS=N/R	
				15' END OF PUSH	
20					
25					
30					

AP184A (1/92)

GENERAL DATA

FILE NO. TOP01-08
CLIENT: MCDOR/NAVY
SITE: TOP-1
HOLE NO. 8 SHEET 1 OF 1
LOCATION OF BORING
DATE 9/5/06

CASING:
INSIDE DIAMETER N/A IN.
OUTSIDE DIAMETER N/A IN.
DEPTH OF CASING N/A FT.

SPOON:
INSIDE DIAMETER 2 IN.
OUTSIDE DIAMETER N/A IN.

HAMMER:
HAMMER WEIGHT N/A LB.
DROP OF HAMMER N/A IN.

ELEVATION:
GROUND SURFACE 119.9 FT.

GWL:
DATE 1st N/A
DATE 2nd N/A
ACTUAL ELEVATION N/A

DRILLING CREW:
Geo Sampell

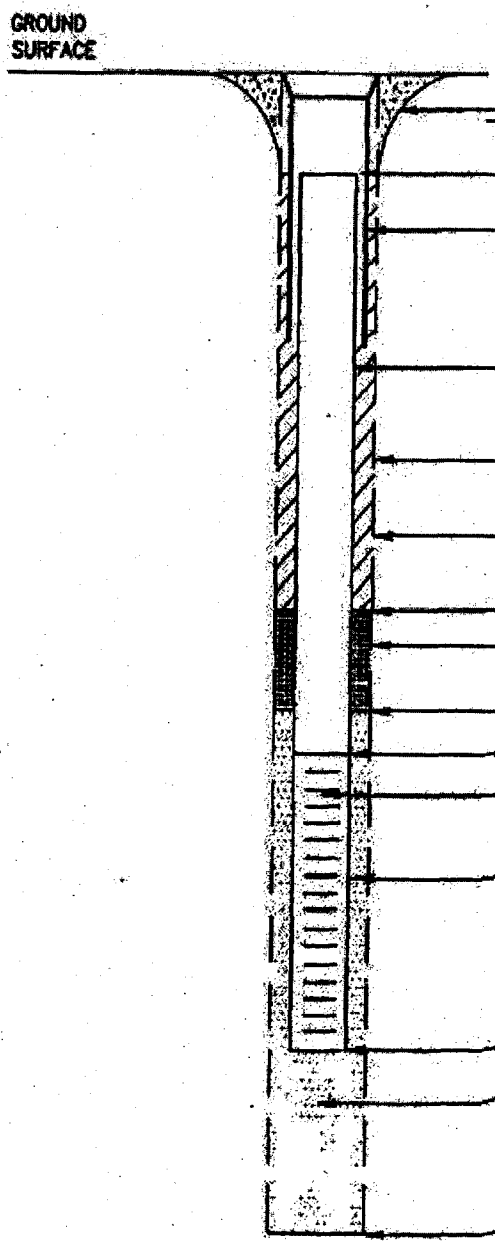
REMARKS:
SAMPLES
TA-SB-08-05
TA-SB-08-08

UNCONSOLIDATED
MONITORING WELL CONSTRUCTION DIAGRAM

WELL NO. MW-01

PROJECT: TOPSHAM
PROJECT NO.: TOP-1
DATE: 9/6/06 BORING NO.:
ELEVATION:
FIELD GEOLOGIST: STEVE LEDUNETTE

DRILLER: GEOSEARCH
DRILLING METHOD: SPLIT SPOON
DEVELOPMENT METHOD: PUMP SURGE



ELEVATION OF TOP OF SURFACE CASING: 120.62'
TYPE OF SURFACE SEAL: CONCRETE FLUSH MOUNT
GROUND SURFACE ELEVATION: 120.5'
ELEVATION OF TOP OF RISER: 120.41'

I.D. OF SURFACE CASING: 10"
TYPE OF SURFACE CASING: STEEL

RISER PIPE I.D.: 2"
TYPE OF RISER PIPE: PVC

BOREHOLE DIAMETER: N/A

TYPE OF BACKFILL: FILL

ELEVATION/DEPTH TOP OF SEAL: 3.25
TYPE OF SEAL: BENTONITE GROUT

ELEVATION/DEPTH TOP OF SAND PACK: 4.25
ELEVATION/DEPTH TOP OF SCREEN: 4.54

TYPE OF SCREEN: PVC
SLOT SIZE X LENGTH: 20 SLOT 5'
TYPE OF SAND PACK: FIL PAK #2

ELEVATION/DEPTH BOTTOM OF SCREEN: 9.54
ELEVATION/DEPTH BOTTOM OF SAND PACK: 9.54
TYPE OF BACKFILL BELOW OBSERVATION WELL: SAND

ELEVATION/DEPTH OF HOLE: 10.54

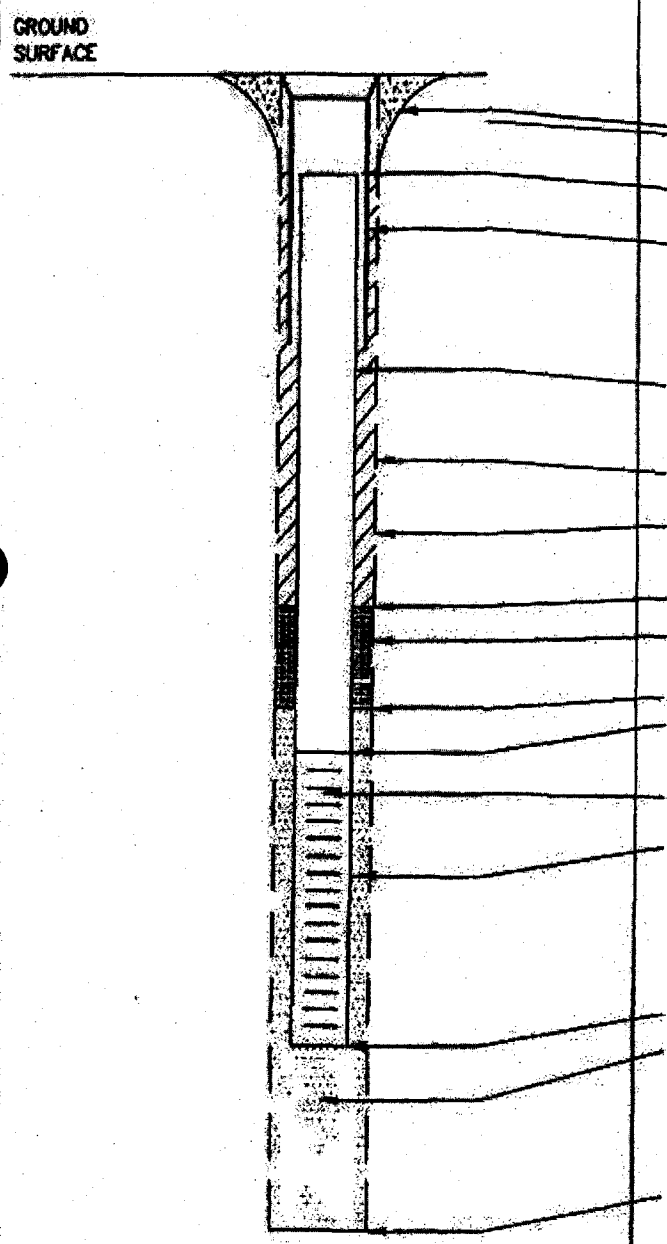
NOT TO SCALE

UNCONSOLIDATED
MONITORING WELL CONSTRUCTION DIAGRAM

WELL NO. MW-02

PROJECT: TOPSHAM
PROJECT NO.: TOP-1
DATE: 9/16/06 BORING NO.:
ELEVATION:
FIELD GEOLOGIST: STEVE LEONETTE

DRILLER: GEOSEARCH
DRILLING METHOD: SPLIT SPOON
DEVELOPMENT METHOD: PUMP SURGE



NOT TO SCALE

ELEVATION OF TOP OF SURFACE CASING: 114.20
TYPE OF SURFACE SEAL: CONCRETE FLUSH MOUNT
GROUND SURFACE ELEVATION: 114.0
ELEVATION OF TOP OF RISER: 113.96

I.D. OF SURFACE CASING: 10"
TYPE OF SURFACE CASING: STEEL

RISER PIPE I.D.: PVC - 2"
TYPE OF RISER PIPE: PVC

BOREHOLE DIAMETER: N/A

TYPE OF BACKFILL: FILL

ELEVATION/DEPTH TOP OF SEAL: 3.37
TYPE OF SEAL: BENTONITE

ELEVATION/DEPTH TOP OF SAND PACK: 4.37
ELEVATION/DEPTH TOP OF SCREEN: 4.87

TYPE OF SCREEN: PVC
SLOT SIZE X LENGTH: 20 SLOT 5'
TYPE OF SAND PACK: FIL PRO # 2

ELEVATION/DEPTH BOTTOM OF SCREEN: 9.87
ELEVATION/DEPTH BOTTOM OF SAND PACK: 9.87
TYPE OF BACKFILL BELOW OBSERVATION WELL: SAND

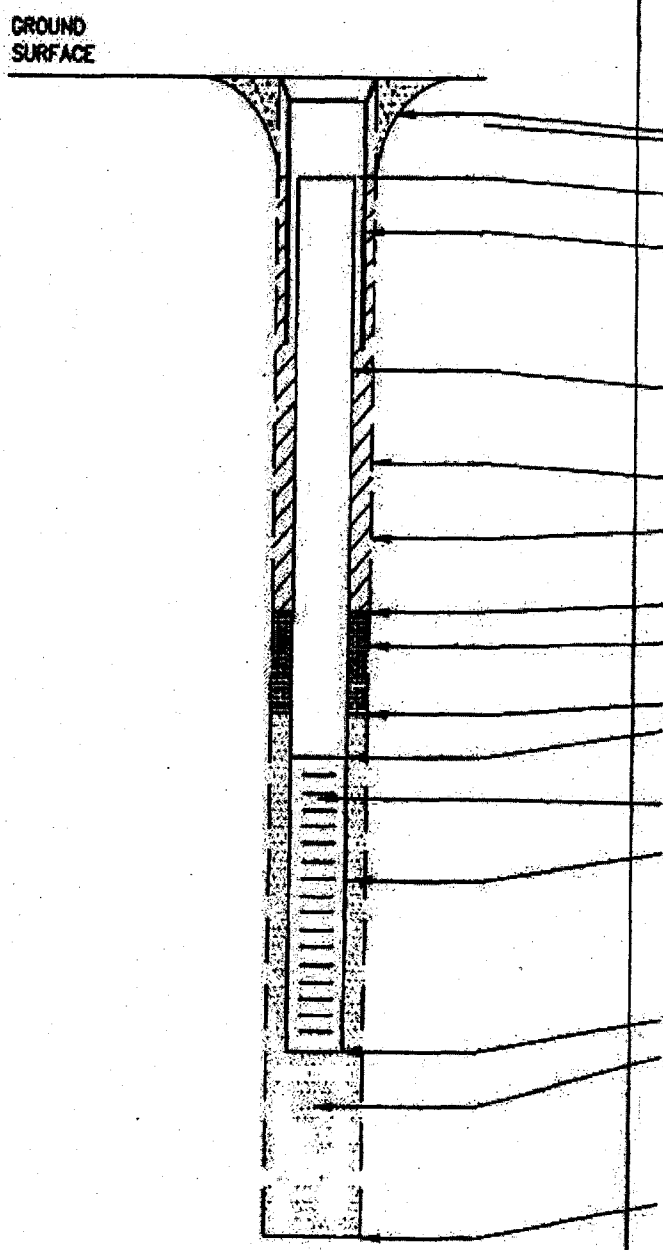
ELEVATION/DEPTH OF HOLE: 10.87

UNCONSOLIDATED
MONITORING WELL
CONSTRUCTION DIAGRAM

WELL NO. MW-03

PROJECT TOBYHANNA
PROJECT NO. TOP-1
DATE 9/16/06 BORING NO.:
ELEVATION
FIELD GEOLOGIST STEVE LOWERTE

DRILLER GEOSEARCH
DRILLING METHOD SPLIT SPOON
DEVELOPMENT METHOD SURGE PUMP



NOT TO SCALE

ELEVATION OF TOP OF SURFACE CASING: 117.22'
TYPE OF SURFACE SEAL: CONCRETE FLUSH MOUNT
GROUND SURFACE ELEVATION: 117.0'
ELEVATION OF TOP OF RISER: 116.74'

I.D. OF SURFACE CASING: 10"
TYPE OF SURFACE CASING: STEEL

RISER PIPE I.D. PVC 2"
TYPE OF RISER PIPE: PVC

BOREHOLE DIAMETER: N/A

TYPE OF BACKFILL: FILL

ELEVATION/DEPTH TOP OF SEAL: 7.29
TYPE OF SEAL: BENTONITE

ELEVATION/DEPTH TOP OF SAND PACK: 8.29
ELEVATION/DEPTH TOP OF SCREEN: 8.79

TYPE OF SCREEN: PVC
SLOT SIZE X LENGTH: 20-SLOT 5'
TYPE OF SAND PACK: FIL PRO #2

ELEVATION/DEPTH BOTTOM OF SCREEN: 13.79
ELEVATION/DEPTH BOTTOM OF SAND PACK: 13.79
TYPE OF BACKFILL BELOW OBSERVATION WELL: SAND

ELEVATION/DEPTH OF HOLE: 14.79

WELL DEVELOPMENT DATA SHEET

Well I.D. MW-01
Date 9/26/2006

Project Name: TOBYHANNA
Project No.: TOP-1
Samplers E. HINKLE

Screened Interval (ft): 5 ft
Depth of Pump (ft below TIC): 7 ft
Total Depth (ft below TIC): 9.54 ft
Static Water Level (ft below TIC): 2.87 ft
Volume of Flow Through Cell: L

2" PVC
5' 20 SLOT PVC SCREEN
1 VOL = 1.09 GAL

Frequency of Parameter reading Calculation

Volume of Flow Through Cell = V (L)
Pump Discharge Flow Rate = Q (L/m)
Frequency of Readings = F (m)

F = V/Q

Purge Method

LOW FLOW PERISTALTIC W/ TEFLON LINED TUBING

Field Parameters

TIME	FLOW RATE (L/m)	DTW (ft TIC)	pH (SU)	SPEC. COND. (S/m)	TURB (NTU)	DO (mg/l)	TEMP (°C)	ORP (mV)	APPEARANCE
1000	0.5	2.87	5.86	0.190	543.0	3.22	15.88	132	
1005	0.25	3.05	5.18	0.188	150.0	1.59	16.02	208	
1010	0.25	3.04	5.20	0.190	139.0	1.55	16.01	209	
1015	0.25	3.09	5.20	0.190	134.0	1.44	15.96	216	
1020	0.25	3.10	5.18	0.184	64.5	1.24	15.98	226	
1025	0.25	3.15	5.21	0.179	41.4	0.62	15.96	229	
1030	0.25	3.16	5.22	0.177	40.3	0.59	15.98	229	
1035	0.25	3.17	5.21	0.178	40.2	0.59	15.97	229	
1040	0.25	3.16	5.20	0.179	39.9	0.60	15.97	228	
1045	0.25	3.20	5.21	0.179	39.7	0.58	15.98	229	
1050	0.25	3.23	5.20	0.179	39.8	0.57	15.96	229	
1055	0.25	3.27	5.20	0.178	39.8	0.58	15.97	229	

Total Volume Purged: ≈16.25 L

Notes/Observations: 1100 COLLECT MW-01 & MS/MSD
DTW = 3.35'

WELL DEVELOPMENT DATA SHEET

Well I.D. MW-02
Date 9/27/2006

Project Name: TOBYHANNA
Project No.: TOP-1
Samplers E. HINKLE

Screened Interval (ft): 5 ft
Depth of Pump (ft below TIC): 7.37 ft
Total Depth (ft below TIC): 9.87 ft
Static Water Level (ft below TIC): 5.22 ft
Volume of Flow Through Cell: L

2" PVC
5' 20-SLOT PVC SCREEN
1 VOL = 0.76 GAL

Frequency of Parameter reading Calculation

Volume of Flow Through Cell = V (L)
Pump Discharge Flow Rate = Q (L/m)
Frequency of Readings = F (m)

F = V/Q

Purge Method

LOW FLOW PERISTALTIC W/ TEFLON LINED TUBING

Field Parameters

TIME	FLOW RATE (L/m)	DTW (ft TIC)	pH (SU)	SPEC. COND. (S/m)	TURB (NTU)	DO (mg/l)	TEMP (°C)	ORP (mV)	APPEARANCE
0940	0.25	5.22	7.08	0.253	56.3	3.71	13.25	35	
0945	0.26	5.80	6.46	0.265	51.6	0.00	13.09	23	
0950	0.1	5.83	6.29	0.260	49.6	0.00	13.07	24	
0955	0.1	5.87	6.24	0.256	42.1	0.00	13.05	23	
0980 1000	0.1	5.90	6.22	0.256	37.7	0.00	13.04	24	
1005	0.1	5.95	6.22	0.256	41.4	0.00	13.04	23	
1010	0.1	5.98	6.22	0.255	41.1	0.00	13.04	23	
1015	0.1	6.01	6.22	0.254	41.3	0.00	13.03	23	
1020	0.1	6.04	6.23	0.253	41.7	0.00	13.00	23	
1025	0.1	6.05	6.23	0.253	41.5	0.00	13.00	23	

Total Volume Purged: ≈6.5L

Notes/Observations: 1030 COLLECT MW-02 & MW-DUP-01
FINAL DTW = 6.05'

WELL DEVELOPMENT DATA SHEET

Well I.D. MW-03
Date 9/27/2006

Project Name: TOBYHAN
Project No.: TOP-1
Samplers E. HINKLE

Screened Interval (ft):	5	ft
Depth of Pump (ft below TIC):	11.29	ft
Total Depth (ft below TIC):	13.79	ft
Static Water Level (ft below TIC):	5.21	ft
Volume of Flow Through Cell:		L

2" PVC
5' 20 SLOT PVC SCREEN
1 VOL = 1.4 GAL

Frequency of Parameter reading Calculation

Volume of Flow Through Cell = V (L)
Pump Discharge Flow Rate = Q (L/m)
Frequency of Readings = F (m)

F = V/Q

Purge Method

LOW FLOW PERISTALTIC W/ TEFLON LINED TUBING

Field Parameters

TIME	FLOW RATE (L/m)	DTW (ft TIC)	pH (SU)	SPEC. COND. (S/m)	TURB (NTU)	DO (mg/l)	TEMP (°C)	ORP (mV)	APPEARANCE
0755	0.2	6.87	6.47	0.465	73.8	6.08	11.70	96	
0800	0.1	7.52	6.48	0.463	66.2	6.10	11.63	98	
0805	0.1	8.45	6.52	0.461	66.5	5.85	11.73	100	
0810	0.1	9.10	6.63	0.462	66.4	5.52	11.81	95	
0815	0.1	9.19	6.73	0.477	47.5	3.91	11.75	98	
0820	0.1	9.35	6.87	0.479	47.1	3.42	11.69	98	
0825	0.1	9.34	6.90	0.489	40.1	3.10	11.69	93	
0830	0.1	9.34	6.89	0.487	39.2	2.94	11.68	93	
0835	0.1	9.35	6.89	0.489	38.2	2.25	11.68	92	
0840	0.1	9.35	6.88	0.489	38.1	2.24	11.68	91	
0845	0.1	9.35	6.89	0.490	38.2	2.25	11.69	92	

Total Volume Purged: ≈ 55 L

Notes/Observations: 0850 COLLECT MW-03
FINAL DTW = 9.35'

WELL AND TEST PIT LOCATIONS FOR TETRA TECH EC, INC.- NAS BRUNSWICK/TOPSHAM ANNEX

PERFORMED BY OEST ASSOCIATES, INC. ON OCTOBER 3, 2006

HORIZONTAL DATUM IS MAINE STATE GRID COORDINATE SYSTEM NAD83 WEST ZONE

VERTICAL DATUM IS NATIONAL GEODETIC VERTICAL DATUM OF 1929 MSL = 0.00

ALL UNITS ARE EXPRESSED IN U.S. SURVEY FEET

DESCRIPTION	NORTHING	EASTING	TOP OF PVC INSERT ELEVATION	FLUSH MOUNT WELL CAP ELEVATION	GROUND ELEVATION
TP01 11	405504.08	3006487.05			128.7
TP01 12	405509.49	3006471.11			128.6
TP01 13	405510.76	3006460.09			128.5
TP01 14	405497.27	3006455.18			129.6
TP01 15	405495.71	3006466.44			129.5
TP01 16	405491.07	3006484.03			129.6
TP01 PIPE 01	405510.34	3006467.35			128.6
TP01 PIPE 02	405496.29	3006462.55			129.6
TP02 17	405457.40	3006528.92			131.0
TP02 20	405454.47	3006541.88			130.9
TP03 22	405445.27	3006457.07			131.7
TP03 23	405447.07	3006452.91			131.7
TP03 21 & 24	405465.31	3006453.75			131.1
MW 1	406575.72	3006670.17	120.41	120.62	120.5
MW 2	406557.70	3006458.15	113.96	114.20	114.0
MW 3	406648.17	3006520.41	116.74	117.22	117.0
TOP 01 04	406520.98	3006617.16			119.4
TOP 01 05	406486.79	3006535.07			115.6
TOP 01 06	406629.96	3006463.81			117.0
TOP 01 07	406644.08	3006578.06			118.0
TOP 01 08	406608.88	3006644.71			119.9

TOPSHAM ANNEX SKEET RANGE
(WESTERN & CENTRAL)
2006 AERIAL
PROVIDED BY TOWN OF TOPSHAM

TOPSHAM ANNEX SKEET RANGE
(EASTERN)
2006 AERIAL
PROVIDED BY TOWN OF TOPSHAM

APPENDIX D

RESPONSES TO STAKEHOLDER COMMENTS

D-1 FEBRUARY 2008 ADDENDUM UPDATE TO STAKEHOLDER FOLLOW-UP COMMENTS

ADDENDUM TO RESPONSE TO COMMENTS
DRAFT FINAL SITE INSPECTION WORK PLAN
NAVAL AIR STATION BRUNSWICK, MAINE

Based on the October 2007 kickoff meeting/site visit of MRP sites with the Stakeholders and Stakeholder disagreement with Navy (Malcolm Pirnie) responses to Stakeholder follow-up comments, and newly gathered information, numerous revisions to Malcolm Pirnie February 2007 Draft Final Site Inspection Work Plan and associated comment responses are warranted. The revisions are detailed as follows and have been incorporated into the Tetra Tech February 2007 Revised Draft Final version of the document.

In order to respond effectively, only the follow-up comments warranting Tetra Tech updated responses are included. The Stakeholder follow-up comment, the Malcolm Pirnie response, and the Tetra Tech updated response (February 2008) associated with this addendum are provided to document the rationale for the changes. Malcolm Pirnie and Tetra Tech's responses are displayed in ***bold italics.***

Draft Final Site Investigation Work Plan (February 2007) – Comments from Claudia Sait (MEDEP) also addressing Comments from Christine Williams (USEPA – Region 1):

MEDEP Follow-Up General Comment 1:

A number of the RTCs indicate that new text or information will be provided (i.e., 6, 7, 16, 17, 18, 20 and 30. Before MEDEP can accept the workplan the requested information must be provided for stakeholder review.

Navy's follow-up RTC:
The requested information is presented below:

Comment 6: The sample location maps for the SI Work Plan have been attached to this Response to Comments document.

Comment 7: Malcolm Pirnie has been working closely with Tetra Tech, who is responsible for the QAPP. The Tetra Tech QAPP will be provided as an appendix to the Malcolm Pirnie SI Work Plan.

Comment 16: The draft MEC Work Plan and HASP are anticipated for submittal in October 2007. Additionally, a site visit is scheduled for October 23-24, 2007.

Comment 17: The draft MEC Work Plan and HASP are anticipated for submittal in October 2007. Additionally, a site visit is scheduled for October 23-24, 2007.

Comment 18: The draft MEC Work Plan and HASP are anticipated for submittal in October 2007. Additionally, a site visit is scheduled for October 23-24, 2007.

Comment 20: Malcolm Pirnie has been working closely with Tetra Tech, who is responsible for the QAPP. The Tetra Tech QAPP will be provided as an appendix to the Malcolm Pirnie SI Work Plan.

Comment 30: The draft MEC Work Plan and HASP are anticipated for submittal in October 2007. Additionally, a site visit is scheduled for October 23-24, 2007.

Updated Navy Response (February 2008):
Comment 6: The sample location maps have been significantly revised based on the October 2007 kickoff meeting/site visits among the Stakeholders and new information gathered. The updated figures are included in the main body of the Work Plan.

Follow-Up to MEDEP Specific Comment 3:

3. For the sites with lead (i.e., Naval Air Station skeet range, Topsham Annex skeet range, and the Machine Gun Bore Sight Range) as a potential contaminant MEDEP suggests screening the areas with an X-Ray Florescence (XRF) or metal detector prior to sampling to determine the boundaries and maximum shot fallout zones. It would be extremely helpful in determining if the Machine Bore Sight Range berm was graded in place rather than removed. By the Navy own documents (Preliminary Assessment February 2006) it estimates that 1,641,600 rounds of lead was discharged at this site. The XRF or metal detector would also be valuable for determining if the original NAS skeet range which was oriented to the north still has pellets on the embankment since the stream was dredged in the 1990's to create impoundment ponds.

Navy's RTC: ***The purpose of the SI is determine presence or absence of MEC and MC, not to define the extent of the MEC or MC contamination. Conventional soil sampling techniques will be utilized during the SI phase and locations will be biased to source areas where the maximum concentrations would be expected. If warranted, an XRF will be utilized during the RI phase.***

MEDEP's follow up comment: Conventional soil sampling techniques are designed for homogeneous contamination not heterogeneous particulate matter such as found on a range. Also, the source areas (firing point and target area) appear to have been disturbed at the Machine Gun Bore Sight Range and the Naval Air Station (NAS) Skeet range. Since this investigation determines whether or not there is a remedial investigation, it is imperative that this investigation is well thought out and executed. It is unclear to MEDEP how the Navy plans to ensure that the biased sampling is focused taking into account the disturbance of the target area and fall out zone at these sites.

Navy's follow-up RTC (September 2007): The Navy will collect the majority of soil samples in the shotfall zones and firing point of the skeet ranges and in the area from the firing line to and including the berm at the Machine Gun Boresight Range. Of note, the Machine Gun Boresight Range berm appears to have been graded in place since mounding is evident at the former location; several depth intervals are planned for sample collection considering that the berm has been disturbed. Of note for the NAS Skeet Range, the sampling is planned to encompass both the older and more recent range orientations. For the two skeet ranges, contamination, if present, would primarily be expected from 0 to 6 inches, so the proposed 0 to 1 foot sample interval allows for site disturbance; in addition, several subsurface soil samples are includes to further assess vertical contamination/disturbance, if any. The sample location maps for the SI Work Plan have been attached to this Response to Comments document. It should be noted that sampling locations can be refined during the pre-field activities site visit to be conducted in October.

Updated Navy Response (February 2008):
XRF sampling will be employed for the Machine Gun Boresight Range and the Topsham Skeet Range.

X-Ray Fluorescence (XRF) investigations and additional site mapping will be conducted at two

sites – Machine Gun Boresight Range and Topsham Skeet Range. Discrepancies and or lack of information on these two sites have led to uncertainty on the site boundaries. Therefore, XRF investigations may aid in establishing the locations for MC sampling. At each of the two sites, a grid will be established over the site area and samples will be analyzed in the field for lead by XRF in the field, beginning with the grid squares expected to have the maximum concentrations and working outward. The USEPA will conduct the XRF investigation at the Machine Gun Boresight Range. TtNUS will conduct the XRF investigation at the Topsham Skeet Range.

At the Machine Gun Boresight Range, the disposition of the former berm is unknown and a dirt pile on site may or may not be the remnants of the former berm. The existing dirt pile boundary will be mapped in the field using a hand-held surveying Global Positioning System (GPS) device. The distance to the dirt pile has been measured but the entire outline of the dirt pile should be mapped. Additionally, any areas with concrete debris should be mapped as they may represent the historical flared concrete end walls of the backstop berm. Maximum concentrations are expected at the existing dirt pile and/or the former berm location based on historical aerial photographs. Up to 90 soil samples are planned for collection via XRF, with a minimum of 30 soil samples. The minimum 30 samples may be sufficient if it can be established that maximum lead concentrations present exceed the residential standard of 400 mg/kg and the source area locations (i.e., former berm location, existing dirt pile, and area in between) are consistent with those of the current-day conceptual site model, because the decision to move forward to an RI would be evident at this point and the data collected could be built upon during the RI. Otherwise, up to 90 samples will be collected via XRF to meet the objective. During field analysis, the XRF lead data will be reviewed on a ½ day basis (approximately every 15 samples) to support the continuing effort of determining sample locations.

At Topsham Skeet Range, very little information is available on the exact location of the range and aerial photographs do not resolve this issue. Also, additional effort is warranted because most of the site lies outside of the NAS Brunswick boundary and so the Navy will not be able to place land use controls on the property. Any remaining historical features, such as remaining wooden posts, etc., will be mapped via a hand-held surveying GPS device. Maximum concentrations are expected within the shotfall zone, working radially outward from the center. Similar to the Machine Gun Boresight Range, up to 90 surface soil samples are planned for collection via XRF, with a minimum of 30 surface soil samples. The minimum 30 samples may be sufficient if it can be established that maximum lead concentrations present exceed the residential standard of 400 mg/kg and the source area location (i.e., shotfall zone) is consistent with that of the current-day conceptual site model, because the decision to move forward to an RI would be evident at this point and the data collected could be built upon during the RI. Otherwise, up to 90 samples will be collected via XRF to meet the objective. During field analysis, the XRF lead data will be reviewed on a ½ day basis (approximately every 15 samples) to support the continuing effort of determining sample locations.

The XRF results will aid in locating the samples for MC sampling, which will be conducted at the locations of highest concentrations established by the XRF investigation. The XRF results will be used for field screening purposes only and so no confirmation sampling for the XRF samples are planned prior to collecting the MC soil samples for fixed-based laboratory analysis. Planned soil sampling locations shown on the figures may be adjusted based on XRF results.

Follow-Up to MEDEP Specific Comment 21 and USEPA General Comment 3:

21. Table 3-2:

a.) At this time MEDEP can not concur with the number of surface soil samples proposed in this table.

b.) Munitions Bunker Area West: 15 composite surface soil samples are proposed for a 29 acre site - roughly one composited sample per two acres. Please provide the rationale for this number of samples, the compositing scheme and what criteria will be used to determine where the samples will be taken.

c.) Machine Gun Boresight Range: 5 composite surface soil samples are proposed for a 0.03 acres site (firing line to berm). This may be adequate if it can be determined whether the berm was removed or just graded out in the vicinity, however prior screening prior with a metal detector or an XRF will be needed before the number of soil samples can be determined. No groundwater samples are proposed, yet in the Preliminary Assessment it was estimated that 1.6 million rounds (30 and 50 caliber bullets) were discharged into the berm. Groundwater sampling must be performed unless it can be determined that the berm was removed and that expended rounds were collected and disposed of offsite.

d.) NAS Skeet Range: 20 composite surface soil samples are proposed for a 73 acres site. Without prior screening to determine where to sample, this number is inadequate. The majority of the shot fallout zone appears to be over the streams and their floodplain wetland. Additional sediment sampling is necessary and due to the amount of run off and sediment from the storm drains it may be necessary to do some sediment sampling at depth.

e.) Site 12 EOD: 25 composite surface samples may be adequate if only the bermed area (1.5 acres) is being characterized but it is not adequate for the entire 112.7 acres. The 3 groundwater samples may be adequate, however the site is shallow to bedrock and direct push may be necessary to just determine the amount of soil coverage. Ultimately it may be necessary to install bedrock wells.

f.) Topsham Annex Skeet Range: 20 composite surface soil samples are proposed for a 29 acres site. If the preliminary screening with an XRF or metal detector is performed to direct the sampling locations to the maximum shot fallout zone this may be adequate. MEDEP agrees with EPA regarding the necessity to sample groundwater at this site.

g.) State criteria for soil, groundwater, and surface water, if lower than that of Region 9, must also be used for comparison regarding for whether to continue with a Remedial Investigation. The sediment criteria agreed upon for the other IR sites should also be considered where appropriate.

Navy's RTC: The number of soil samples was based on the premise that the SI is not intended to characterize the sites but to determine the presence or absence of MEC and MC. Sampling locations will be biased to source areas where maximum concentrations would be expected.

MEDEP's follow up comment: The Navy response did not address the questions. Also the stated approach may work for undisturbed ranges where the firing point and target area are clear however that approach appears questionable for ranges that have been disturbed and area with no defined source areas.

Navy's follow-up RTC (September 2007): The sampling program is based on a biased sampling approach designed to focus on locations of anticipated maximum concentrations (e.g., groundwater just beyond the shotfall area, soil within a berm). Furthermore, for MEC sites, surface sweeps and geophysical surveying will be used to identify suspicious locations for sampling (e.g., sample collection near MEC surface debris or subsurface anomaly). Additionally, groundwater sampling is planned at ALL of the six sites.

No surface water/sediment samples are planned for the NAS Skeet Range since the pond is actually a PAH treatment area and we would not be able to determine if the NAS Skeet Range was the source.

State criteria will also be used in determining the path forward for each of the sites.

Section 3.3.2 has been revised as follows:

3.3.2 MC Field Activities

Table 3-3-2 provides a summary of decisions made to address MC.

Former Munitions Bunker West Area	Collect 15 composite surface soil samples and analyze for target analyte list (TAL) metals and explosives, polyaromatic hydrocarbons (PAHs), and propellants. Collect 10 discrete subsurface soil samples and analyze for TAL metals and explosives, PAHs, and propellants. Collect five groundwater samples and analyze for TAL metals, explosives, PAHs, and propellants.	CTC and site prioritization. Support MC NFA or RI determination. NFA determination is made if sample analytical data for site are below analyte-specific USEPA Region 9 Preliminary Remediation Goals (PRGs) for soil and USEPA Federal Drinking Water Standards for groundwater.

Machine Gun Boresight Range	Collect discrete soil samples from 12 locations: six locations surface only, six locations surface and two subsurface depth intervals. Total of 12 surface soil samples and 12 subsurface soil samples to analyze for TAL metals and propellants. Collect five groundwater samples and analyze for TAL metals and propellants.	CTC and site prioritization. Support MC NFA or RI determination. NFA determination is made if sample analytical data for site are below analyte-specific USEPA Region 9 PRGs for soil and USEPA Federal Drinking Water Standards for groundwater.
Skeet Range	Collect 20 discrete surface soil samples and analyze for TAL metals and PAHs. Collect 8 discrete subsurface soil samples and analyze for TAL metals and PAHs. Collect eight groundwater samples and analyze for TAL metals and PAHs.	CTC and site prioritization. Support MC NFA or RI determination. NFA determination is made if sample analytical data for site are below analyte-specific USEPA Region 9 PRGs for soil, and USEPA Federal Drinking Water Standards for groundwater.
Site 12 EOD	Collect 25 composite surface soil samples and analyze for TAL metals and explosives. Collect 10 discrete subsurface soil samples and analyze for TAL metals and explosives. Collect five sediment samples and analyze for TAL metals, explosives, and TOC. Collect five surface water samples and analyze for TAL metals, perchlorate, and explosives. Collect six groundwater samples and analyze for TAL metals, perchlorate, and explosives.	CTC and site prioritization. Support MC NFA or RI determination. NFA determination is made if sample analytical data for site are below analyte-specific USEPA Region 9 PRGs for soil and sediment, and USEPA Federal Drinking Water Standards for surface water and groundwater.

Quarry	Collect 10 composite surface soil samples and analyze for explosives. Collect 15 discrete subsurface soil samples and analyze for explosives. Collect five sediment samples and analyze for explosives and TOC. Collect five groundwater samples and analyze for explosives and perchlorate. Collect five surface water samples and analyze for explosives and perchlorate.	CTC and site prioritization. Support MC NFA or RI determination. NFA determination is made if sample analytical data for site are below analyte-specific USEPA Region 9 PRGs for soil and sediment, and USEPA Federal Drinking Water Standards for surface water and groundwater.
Topsham Annex Skeet Range	Collect 14 discrete surface soil samples and analyze for TAL metals and PAHs. Collect five discrete subsurface soil samples and analyze for TAL metals and PAHs. Collect two sediment samples and analyze for TAL metals, PAHs, and TOC. Collect five groundwater samples and analyze for TAL metals and PAHs. Collect two surface water samples and analyze for TAL metals and PAHs.	CTC and site prioritization. Support MC NFA or RI determination. NFA determination is made if sample analytical data for site are below analyte specific USEPA Region 9 PRGs for soil and sediment, and USEPA Federal Drinking Water Standards for surface water and groundwater.

The suggested method for Site 12 EOD Area and the Quarry surface soils is the Cold Regions Research and Engineering Laboratory (CRREL) seven sample wheel approach, as described in the CRREL Special Report 95-15, because these two sites were potentially associated with the detonation of explosives. Collection of discrete grab samples is proposed for surface soil samples at other sites and all sites requiring subsurface soil samples. Sediment and surface water samples will be obtained as discrete grab samples.

The goal of the field sampling activities for MC is to determine if the site has been impacted by MC. Anomaly avoidance techniques will be utilized during the MC field sampling activities, where necessary. Analytical data exceeding background levels and appropriate regulatory limits will be used as justification to move the site into the RI phase. The SI field sampling activities are not intended to determine the nature and extent of all contaminants.

All fieldwork will be of sufficient quality to meet the DQOs for the project as dictated in the QAPP. The details of the planned MC field sampling activities are provided in the SAP.

Investigative activities will be conducted based on information obtained during the PA process and data review. Complete sampling and analysis of relevant media will be conducted to assess the risk/hazard posed by any MC found at the site. The UXOSS will use visual observations and a magnetometer to detect the presence of MEC as a precaution will escort the team during fieldwork at MEC-suspected sites.

The following text is presented in Section 4.3 of the Sampling and Analysis Plan (Appendix A to the SI Work Plan):

Former Munitions Bunker West Area

MEC Activities: A detector-aided, non-intrusive site survey will be conducted over the walking path to sample locations in order to avoid MEC items.

MC Activities: Fifteen biased surface soil samples will be collected from an interval of zero to one foot. Ten discrete subsurface samples will be collected from an interval of one to three feet in depth. The locations of the soil samples will be determined in the field based on TtNUS surface sweep and MEC field assessments (conducted under a different work plan). One groundwater sample will be collected from each of five temporary wells that will be installed during the field work effort. The location of the wells will be determined based on the MEC field assessment conducted by TtNUS. Groundwater and soil samples will be analyzed for TAL metals, PAHs, propellants, and explosives using USEPA Methods 6010B, 8270C SIM, 8332/lab SOP, and 8330, respectively.

Machine Gun Boresight Range

MEC Activities: MEC presence is not anticipated; and so, activities associated with MEC are not applicable.

MC Activities: Twenty-four biased soil samples will be collected at this site based on visual observations and locations of the former range features. Six surface soil locations will be sampled in the zone between the firing point and the berm. Six locations at the berm will be sampled from the surface and two subsurface intervals since the berm was disturbed and turned over. Soil samples will be analyzed for TAL metals and propellants using USEPA Method 6010B and 8332/lab SOP, respectively. One groundwater sample will be collected from 5 different temporary wells that will be installed during the field work effort. The location of the wells will be one just down-gradient of the firing point, one in between the firing point and target area, and three just down-gradient of the berm.

Skeet Range

MEC Activities: MEC presence is not anticipated; and so, activities associated with MEC are not applicable.

MC Activities: Twenty biased surface soil, 8 subsurface soil, and eight groundwater samples will be collected at this site based on visual observations and locations of the former range features. All samples will be analyzed for TAL metals using USEPA Method 6010B and for PAHs using USEPA Method 8270C SIM. One groundwater sample will be collected from each of the seven temporary wells that will be installed at the Skeet Range during field activities. In addition, one groundwater sample will be collected from the existing well MW-NASB-078 for the firing point well. These eight groundwater samples will be analyzed for TAL metals and PAHs. Five of the temporary wells will be installed in the

post-1950 Skeet Range Area: one just down-gradient of the firing point; one in between the firing point and the target area; and three surrounding the target area. The remaining two temporary wells will be installed in the pre-1950 Skeet Range Area within the likely shotfall zone.

Site 12 EOD Area

MEC Activities: A detector-aided, non-intrusive site survey will be conducted over the walking path to sample locations in order to avoid MEC items.

MC Activities: Twenty-five biased surface soil, ten subsurface soil, five sediment samples, five surface water, and six groundwater samples will be collected at this site. The locations of the soil samples will be determined in the field based on TtNUS surface sweep and MEC field assessments (conducted under a different work plan). All samples will be analyzed for TAL metals using USEPA Method 6010B and for explosives using USEPA Method 8330. All sediment samples will be analyzed for TOC using method SW846 9060. Additionally, surface water and groundwater samples will be analyzed for perchlorate using USEPA Method 6850 (with filtration in the lab). Six temporary wells will be installed during the field work effort. One well will be placed just down-gradient of the existing bermed area and the remaining five well locations will be determined in the field based on TtNUS surface sweep and MEC field assessments. Surface water and sediment samples were determined based on site conditions and runoff topography. These locations can be found on Map 2-5 of the Work Plan.

Quarry

MEC Activities: A detector-aided, non-intrusive site survey will be conducted over the walking path to sample locations in order to avoid MEC items.

MC Activities: Ten biased surface soil, 15 subsurface soil, five sediment, five surface water, and five groundwater samples will be collected at this MRP site. Subsurface sample locations will be determined in the field based on TtNUS surface sweep and MEC field assessments (conducted under a different work plan). Subsurface soil samples will be collected from depth intervals of one to five feet. Five groundwater samples will be collected from five temporary wells to be installed during the field work effort. Groundwater sample locations will be determined in the field based on TtNUS surface sweep and MEC field assessments. Surface water and sediment sample locations were determined based on site conditions and runoff topography, these locations can be found on Map 2-6 of the Work Plan. All samples will be analyzed for explosives by USEPA Method 8330. Additionally surface water and groundwater samples will be analyzed for perchlorate using USEPA Method 6850 (with filtration done in the lab). Because of recent findings of debris at the site, the work scope is being expanded to include VOC, SVOC, pesticide, and PCB analyses for all media. This scope expansion will be addressed via a Work Plan Addendum to be submitted by TtNUS.

Topsham Annex Skeet Range

MEC Activities: MEC presence is not anticipated; therefore, activities associated with MEC will be for avoidance purposes only.

MC Activities: Fourteen biased surface soil, five subsurface soil, two sediment, two surface water, and five groundwater samples will be collected at this site. Surface soil samples will be collected based on visual observations and locations of the former range features. All samples will be analyzed for TAL

metals using USEPA Method 6010B and for PAHs using USEPA Method 8270C SIM. The samples will be collected from five wells to be installed during the SI field effort. One well will be installed down-gradient from the firing point, one in between the firing point and the target area, and three surrounding the target area.

Updated Navy Response (February 2008): ***The sampling program and sample location maps have been significantly revised based on the October 2007 kickoff meeting/site visits among the Stakeholders and new information gathered. The updated figures are included in the main body of the Work Plan. A summary of revisions to the sampling program and work plan figures is detailed as follows:***

Former Munitions Bunker West –

- ***Added 2 co-located surface water/sediment samples from the drainage ditch running through the site as a possible migration pathway via runoff.***
- ***Added perchlorate analysis to aqueous samples (surface water and groundwater); perchlorate was not initially included since it was not deemed a primary contaminant of concern. However perchlorate was in use historically, albeit in small quantities, and so was added to the sampling program.***

Machine Gun Boresight Range -

- ***Revised site figure to reflect 1959 aerial photograph and field measurements concerning the former berm location and the existing dirt pile.***
- ***Added XRF sampling to aid in refining MC sampling locations.***
- ***Added GPS mapping of existing dirt pile to work plan scope. The distance to the dirt pile has been measured but the entire outline of the dirt pile should be mapped.***
- ***Added a background surface soil sample to aid in comparing soil results at the Machine Gun Boresight Range and the adjacent Skeet Range to natural conditions.***

Skeet Range –

- ***Added 2 co-located surface water/sediment samples for lead analysis from the impoundments. The impoundments are PAH treatment areas for runoff from the runways. Analysis was not originally planned because it would be difficult to ascertain whether any contamination was site related. Ultimately, the Stakeholders agreed to forego PAH analysis but include lead analysis.***
- ***Replaced a planned temporary well inside the shotfall zone with an existing monitoring well associated with adjacent Site 9 that can serve the purpose.***
- ***Relocated planned soil samples beyond the shotfall zone to inside the shotfall zone to maximize the number of samples expected to have contamination.***

Site 12 –

- ***Shifted the 3 predetermined temporary well locations to allow for more triangulation for interpretation of potentiometric surface for interpreting groundwater flow direction.***
- ***Shifted the 2 surface water/sediment locations based on historical aerials.***
- ***Recognized that the planned shallow temporary wells may not yield groundwater based on anticipated shallow depth to bedrock. However, even if temporary wells cannot be installed, bedrock wells will not be installed during the SI but will be deferred to the RI. The Stakeholders recognize that an interim measure to remove MEC from the site will likely occur following the SI, in which case, any wells in the area would interfere with the removal effort. It was agreed that bedrock wells would be best installed during the RI phase, after clearance of munitions from the site.***

Quarry –

- *Based on new information from NAS Brunswick at a May 2007 site visit by Tetra Tech, garbage and debris dumping are suspected. Therefore, the addition of TCL VOCs, SVOCs, pesticides, and PCBs, and TAL Metals to the sampling suite is warranted. The additional analyses will be integrated into the Revised Draft Final Work Plan.*
- *More detailed information on the sources of contaminated soil treated at the landfarming area was obtained from MEDEP.*

Topsham Skeet Range –

- *Added XRF sampling to aid in refining MC sampling locations since the historical site boundaries are uncertain and additional effort is warranted because most of the site lies outside of the NAS Brunswick boundary and so the Navy will not be able to place land use controls on the property.*
- *Relocated planned soil samples beyond the shotfall zone to inside the shotfall zone to maximize the number of samples expected to have contamination.*
- *Included 3 existing monitoring wells (MW01, MW02, and MW03)from a nearby investigation of September 2006 for water-level measurements to enhance the evaluation of groundwater flow.*
- *Deleted surface water/sediment sample at high school detention pond since this location would not be reflective of runoff from the Topsham Skeet Range.*
- *Added 2 co-located surface water/sediment samples from the brook running through the site with one located at the brook and the other within the shotfall zone as these locations are potentially impacted by runoff of contamination from the site.*

For all the sites, the criteria for evaluation was corrected. The surface water/sediment screening criteria will be those values agreed upon by Stakeholders for other IR sites. For soil, Region 9 PRGs will be employed. For groundwater, Region 9 tap water PRGs will be employed or other State criteria (e.g., MEGs) if more stringent.

<u>Follow-Up to MEDEP Specific Comment 22:</u>

22. Section 3.3.2.2, Surface Water/Groundwater Sampling, para 2:

a.) The Navy is proposing two groundwater samples for the NAS Skeet Range to be taken from an existing site well. Please clarify if the groundwater samples are to be taken from the two existing wells rather than one well. Please provide the monitoring well number(s) and location(s). There are two existing wells (MW-NASB-077 and MW-NASB-078), which are located more in the vicinity of the firing point rather than the vicinity of the maximum shot fallout zone. Since there does not appear to be an existing monitoring well in the maximum shot fall out zone, please explain how the selected sampling point(s) will provide a representative groundwater sample for the skeet range.

Navy's RTC: The Navy is proposing 7 temporary wells at the NASB Skeet Range. Five of these wells will be in the post-1950 Skeet Range Area:...

MEDEP's follow up comment: This may be adequate. MEDEP would like to see the locations marked on a figure before accepting this response.

Navy's follow-up RTC (September 2007): The sample location maps for the SI Work Plan have

been attached to this Response to Comments document. It should be noted that sampling locations can be refined during the pre-field activities site visit to be conducted in October.

Updated Navy Response (February 2008): Refer to the follow-up to MEDEP Specific Comment 21 for additional rationale for use of two existing monitoring wells from Site 9 to aid the Skeet Range SI.

b.) The Navy is proposing three direct push wells for Site 12 EOD Area. This site appears to be shallow to bedrock. Direct push may be necessary to determine soil coverage at the site, however to obtain a representative groundwater sample it may be necessary to install bedrock wells. The 1990 Remedial Investigation information for Site 12 is limited to three test pits. What is the rationale for the three direct push locations? Depending upon the bedrock topography, shallow groundwater flow may be to the east, south or southwest, while migration in the bedrock will depend upon the dominant fractures, foliations, and other features. Additional information is required to determine the best location for sampling groundwater at the site.

Navy's RTC: The Navy proposes collection of surface soil samples from the surface (0-12 inches). The subsurface soil sample locations and depths will be established following the geophysical survey in consultation with the Navy and regulatory agencies.

The Navy is proposing installation of 6 temporary wells. One well will be positioned just downgradient of the bermed area. The remaining 5 temporary well positions will be determined in the field based on geophysical survey anomaly identification. Bedrock wells will not be installed during the SI phase.

MEDEP's follow up comment: (The first portion of the response is acceptable.) Regarding the temporary wells, if the soil depth is thin or on bedrock in the area of the EOD sites within Site 12 it will be impossible to evaluate impacts to groundwater without bedrock wells.

Navy's follow-up RTC (September 2007): In the event that bedrock is shallow in the area of investigation, the Navy will install bedrock wells and investigate the groundwater beneath the site in the future during the RI phase of the project. Installation of bedrock wells is beyond the scope of an SI.

Updated Navy Response (February 2008): Refer to the follow-up to MEDEP Specific Comment 21 for additional rationale for excluding bedrock wells during the SI phase of work.

Follow-Up to MEDEP Comment on Attachment 2 (SOPs):

Attachment 2 (SOPs)

- Under "Temporary Well Abandonment" it must comply with Maine Chap 405 Sec 5 (H).

Comment noted.

Updated Navy Response (February 2008): The requirement will be added to the Appendix A Sampling and Analysis Plan, Section 4.8.4.

D-2 SEPTEMBER 2007 RESPONSE TO STAKEHOLDER FOLLOW-UP COMMENTS

DEPARTMENT OF THE NAVY
BASE REALIGNMENT AND CLOSURE
PROGRAM MANAGEMENT OFFICE, NORTHEAST
4911 SOUTH BROAD STREET
PHILADELPHIA, PA 19112-1303

5090
BPMO NE/LM
Ser 07-129
September 13, 2007

Ms. Christine A.P. Williams
Remedial Project Manager
Federal Facilities Superfund Section
United States Environmental Protection Agency (EPA)
1 Congress Street, Suite 1100 (HBT)
Boston, MA 02114-2023

Ms. Claudia Sait
Remedial Project Manager
Maine Department of Environmental Protection (MEDEP)
Bureau of Remediation and Waste Management
17 State House Station
Augusta, ME 04333-0017

Dear Ms. Williams and Ms. Sait:

SUBJECT: RESPONSE TO COMMENTS ON DRAFT FINAL SITE INVESTIGATION WORK PLAN AND DRAFT FINAL PRELIMINARY ASSESSMENT ADDENDUM FOR THE NAVAL AIR STATION BRUNSWICK, MAINE

Enclosed you will find the Navy's response to comments on the Draft Final Site Investigation Work Plan and the Draft Final Preliminary Assessment Addendum for the Naval Air Station Brunswick (NASB) Maine.

If you have any questions or comments, please contact the Navy's RPM, Lonnie Monaco, at (215) 897-4911, or Dawn Kincaid at (215) 897-4915.

Sincerely,

David Drozd
Director

Enclosure:
Responses to Comments

Copy to:
MEDEP (C. Evans)
Gannett-Fleming (P. Golanka, D. McTigue)
NASB (L. Joy, D. Mosher)
Lepage Environmental (C. Lepage)
NAVFAC Mid-LANT (L. Monaco, D. Barclift)
NAVFAC Atlantic (D. Waddill, J. Wright, B. Capito – Admin Record)
ECC (A. Easterday, G. Calderone, C. Guido)
TtNUS (L. Klink)
NOSSA (D. Murray)

Copy to: (w/o encl)
BLRA (C. Warren)
RAB Community Co-chair (T. Fusco)
RAB Navy Co-Chair (CAPT Womack)
BASCE (E. Benedikt)
RAB Harpswell rep (D. Chipman)
BRAC PMO (distribution)

Malcolm Pirnie Response to Comments on the
January 2007 Draft Final Preliminary Assessment Addendum
Naval Air Station Brunswick, Maine

Malcolm Pirnie is pleased to provide this Response to Comments for comments received on the January 2007 Draft Final Preliminary Assessment Addendum for Munitions Response Program Sites at Naval Air Station Brunswick. In order to respond effectively, the original comments have been reproduced below. Malcolm Pirnie's responses are displayed in ***bold italics.***

Draft Final Preliminary Assessment Addendum (January 2007) – Response to USEPA (Christine Williams) Follow-up Comments dated 06/21/07 on the Navy's Comment Response Letter dated 05/22/07:

General Comments

2. The original Comment noted the potential for impacts from Site 12 to downgradient water-supply wells. The Response provides additional information on the regional groundwater setting, which is welcome. The Response also notes that site groundwater will be analyzed for perchlorate, and that downgradient wells may be sampled if perchlorate is detected in site groundwater. Please note that perchlorate is relatively mobile in groundwater, and may be "flushed" from site soil and groundwater, while it is still present downgradient. Some assurance that downgradient drinking-water wells are not contaminated may be required.

Response

Agree. The Site Investigation (SI) Work Plan provides for perchlorate sampling on site as well as establishment of groundwater flow direction. Upon evaluation of the SI results, the path forward will be established for the Remedial Investigation (RI) in concurrence with the Stakeholders.

Specific Comments

10. The original Comment asked for further information about the regional groundwater flow setting. The Response notes that groundwater in the vicinity of the Quarry flows to the southwest, and that there are private drinking-water wells downgradient of the site. Please see follow-up comment with respect to potential impacts from Site 12 (GC2).

Response

Agree. Please see the above response.

Updated Response

The Navy recognizes that our response to this comment is the same as that for the Site 12 comment. The Site Investigation (SI) Work Plan provides for establishment of groundwater flow direction at the Quarry. Upon evaluation of the SI results, the path forward will be established for the Remedial Investigation (RI) in concurrence with the Stakeholders.

Malcolm Pirnie Response to Comments on the
February 2007 Draft Final Site Investigation Work Plan
Naval Air Station Brunswick, Maine

Malcolm Pirnie is pleased to provide this Response to Comments for follow-up comments received on the January 2007 Draft Final Site Investigation Work Plan for Munitions Response Program Sites at Naval Air Station Brunswick. In order to respond effectively, the original comments have been reproduced below. Malcolm Pirnie's responses are displayed in ***bold italics.***

Draft Final Site Investigation Work Plan (February 2007) – Response to USEPA (Christine Williams) Follow-up Comments dated 06/21/07 on the Navy's Comment Response Letter dated 05/22/07

General Comments

2. Expansion of the soil analytes to the full Target Analyte List (TAL) is a welcome response to the concern for metals in addition to lead. This will result in far more complete characterization.

The original Comment also requested the rationale for neglect of propellant compounds at the skeet ranges. The Response states that black powder is "usually" the propellant for shotguns. While this may be the case, modern shotgun shells are often made with "double-base" (smokeless) powder. For this reason, it is recommended that at least a few soil samples immediately downrange of the firing points be analyzed for nitroglycerin. Alternatively, if further research can demonstrate that the advent of double-base shotgun shells post-dates the use of the ranges, this might be reconsidered.

According to the reference document "Characterization and Remediation of Soils at Closed Small Arms Firing Ranges" (Interstate Technology and Regulatory Council (ITRC, January 2003), jointly developed by the Department of Energy (DOE), Department of Defense (DoD), and USEPA:

> ***"Small arms firing ranges may contain lead, antimony, copper, zinc, arsenic, and polycyclic aromatic hydrocarbons (PAHs) from nonexploding (nonenergetic) bullets and fragments, bullet jackets, and related sporting material (e.g., clay targets); however, lead is the primary risk driver and is thereby the focus of this guidance."***

The Navy has included in the SI Work Plan not only the primary contaminant lead but also other metals and PAHs. Moreover, both soil and groundwater are being investigated even though PAHs are not anticipated in groundwater because of their low mobility. The SI focuses on the expected contaminants of concern based on the Preliminary Assessment (PA) findings. Upon evaluation of the SI results, the path forward will be established in concurrence with the Stakeholders. If primary analytes are not present at concentrations of concern at the planned biased locations (e.g., firing point, target area), the Navy will propose a No Further Action (NFA) decision. Otherwise, the investigation will proceed to an RI and the Navy will consider expanding the analytical suite.

3. The original Comment raised a concern for the lack of groundwater characterization for most sites considered, particularly for Former Munitions Bunker West Area and the Former Machine Gun Boresight Range. The Response commits to groundwater characterization at these sites via five temporary

monitoring wells. This Response is welcome. The Response is not specific with respect to proposed analytes. It is recommended that groundwater at these two sites be analyzed for propellants, nitroglycerin, perchlorate, VOC, SVOC, pesticides, and TAL metals.

Each of the two sites is addressed individually as follows:

Former Munitions Bunker West

Groundwater analysis is planned for explosives, propellants, PAHs (a subset of semivolatile organic compounds), and Target Analyte List (TAL) metals. However, the Navy disagrees that perchlorates, volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs) (all instead of only PAHs), and pesticides should be added to the analytical suite at this time based on the site history provided in the PA. The explosive compound RDX is the prevalent compound found in military munitions. HMX, TNT, and tetryl are also frequently found. Nitroglycerine is a common component of propellants. All these primary contaminants, and others, are included in the planned explosives and propellants list. Note that PAHs are included in the analytical suite to confirm the site history. VOCs, SVOCs, and pesticides are not components of military munitions.

Upon evaluation of the SI results, the path forward will be established in concurrence with the Stakeholders. If primary analytes are not present at concentrations of concern at the planned biased locations (e.g., at anomaly), the Navy will propose a No Further Action (NFA) decision. Otherwise, the investigation will proceed to an RI and the Navy will consider expanding the analytical suite to include perchlorates if explosives are encountered.

Machine Gun Boresight Range

Groundwater analysis is planned for propellants and TAL metals. Note that propellants are included in the analytical suite to confirm the site history. Perchlorates, VOCs, SVOCs, and pesticides haven't been added to the analytical suite at this time based on the site history provided in the PA. VOCs, SVOCs, and pesticides are not components of military munitions. Perchlorates are a common component of rocket motors fuels and are not expected to be present based on the site history. Upon evaluation of the SI results, the path forward will be established in concurrence with the Stakeholders.

Comment received via e-mail on July 30, 2007: Please indicate whether or not the Navy will be determining groundwater flow at the Quarry and sampling groundwater for MC.

The SI Work Plan provides for establishment of groundwater flow direction at the Quarry. Additionally, the SI Work Plan details the following sampling activities:

MC Activities: Ten biased surface soil, 15 subsurface soil, five sediment, five surface water, and five groundwater samples will be collected at this MRP site. Subsurface sample locations will be determined in the field based on TtNUS surface sweep and MEC field assessments (conducted under the MEC Work Plan in preparation by TtNUS. Subsurface soil samples will be collected from depth intervals of one to five feet. Five groundwater samples will be collected from five temporary wells to be installed during the field work effort. Groundwater sample locations will be determined in the field based on TtNUS surface sweep and MEC field assessments. Surface water and sediment sample locations were determined based on site conditions and runoff topography, these locations can be found on Map 2-6 of the Work Plan. All samples will be analyzed for explosives by USEPA Method 8330. Additionally surface water and groundwater samples will be analyzed for perchlorate using USEPA Method 6850 (with filtration done in the lab).

Malcolm Pirnie Response to Comments on the
February 2007 Draft Final Site Investigation Work Plan
Naval Air Station Brunswick, Maine

Malcolm Pirnie is pleased to provide this Response to Comments for follow-up comments received on the January 2007 Draft Final Site Investigation Work Plan for Munitions Response Program Sites at Naval Air Station Brunswick. In order to respond effectively, the original comments have been reproduced below. Malcolm Pirnie's responses are displayed in ***bold italics.***

Draft Final Site Investigation Work Plan (February 2007) – Follow-up Comments from Claudia Sait (MEDEP) dated 07/26/07:

General Comments:

1. A number of the RTCs indicate that new text or information will be provided (i.e., 6, 7, 16, 17, 18, 20 and 30. Before MEDEP can accept the workplan the requested information must be provided for stakeholder review.

   The requested information is presented below:

   Comment 6: The sample location maps for the SI Work Plan have been attached to this Response to Comments document.

   Comment 7: Malcolm Pirnie has been working closely with Tetra Tech, who is responsible for the QAPP. The Tetra Tech QAPP will be provided as an appendix to the Malcolm Pirnie SI Work Plan.

   Comment 16: The draft MEC Work Plan and HASP are anticipated for submittal in October 2007. Additionally, a site visit is scheduled for October 23-24, 2007.

   Comment 17: The draft MEC Work Plan and HASP are anticipated for submittal in October 2007. Additionally, a site visit is scheduled for October 23-24, 2007.

   Comment 18: The draft MEC Work Plan and HASP are anticipated for submittal in October 2007. Additionally, a site visit is scheduled for October 23-24, 2007.

   Comment 20: Malcolm Pirnie has been working closely with Tetra Tech, who is responsible for the QAPP. The Tetra Tech QAPP will be provided as an appendix to the Malcolm Pirnie SI Work Plan.

   Comment 30: The draft MEC Work Plan and HASP are anticipated for submittal in October 2007. Additionally, a site visit is scheduled for October 23-24, 2007.

2. MEDEP is very disappointed with the Data Quality Objectives (DQO) text. The DQOs appear to be biased toward no further action goal where they should neutral in nature. For example, MEDEP would rewrite the problem statement: "No data is available at the six MMRP sites to determine if environmental impacts have occurred". While MEDEP would prefer to advance to the site investigation it may be necessary for stakeholders to be involved in developing the DQOs for this project.

The SI Work Plan targets the expected contaminants of concern using information from the Preliminary Assessment (PA) as a basis. Upon evaluation of the SI results, the path forward will be established in concurrence with the Stakeholders. The Navy has no preconceptions concerning what the analytical results will indicate or what the path forward will be. The scope of work proposed by the Navy is to provide additional support to aid the Stakeholders in determining the path forward.

3. It has come to MEDEP's attention that if a workplan is too narrowly focused then it is not in contractor's scope to report other environmental issues that are found at the site. Please be sure that the contractor is instructed to report any evidence they find of environmental concern, such as buried drums, petroleum odors, etc.

   Comment noted. All personnel working on the SI will be instructed to report evidence of environmental concern to the Navy, State, and USEPA.

Follow Up Comments:

3. For the sites with lead (i.e., Naval Air Station skeet range, Topsham Annex skeet range, and the Machine Gun Bore Sight Range) as a potential contaminant MEDEP suggests screening the areas with an X-Ray Florescence (XRF) or metal detector prior to sampling to determine the boundaries and maximum shot fallout zones. It would be extremely helpful in determining if the Machine Bore Sight Range berm was graded in place rather than removed. By the Navy own documents (Preliminary Assessment February 2006) it estimates that 1,641,600 rounds of lead was discharged at this site. The XRF or metal detector would also be valuable for determining if the original NAS skeet range which was oriented to the north still has pellets on the embankment since the stream was dredged in the 1990's to create impoundment ponds.

   Navy's RTC: The purpose of the SI is determine presence or absence of MEC and MC, not to define the extent of the MEC or MC contamination. Conventional soil sampling techniques will be utilized during the SI phase and locations will be biased to source areas where the maximum concentrations would be expected. If warranted, an XRF will be utilized during the RI phase.

   MEDEP's follow up comment: Conventional soil sampling techniques are designed for homogeneous contamination not heterogeneous particulate matter such as found on a range. Also, the source areas (firing point and target area) appear to have been disturbed at the Machine Gun Bore Sight Range and the Naval Air Station (NAS) Skeet range. Since this investigation determines whether or not there is a remedial investigation, it is imperative that this investigation is well thought out and executed. It is unclear to MEDEP how the Navy plans to ensure that the biased sampling is focused taking into account the disturbance of the target area and fall out zone at these sites.

   The Navy will collect the majority of soil samples in the shotfall zones and firing point of the skeet ranges and in the area from the firing line to and including the berm at the Machine Gun Boresight Range. Of note, the Machine Gun Boresight Range berm appears to have been graded in place since mounding is evident at the former location; several depth intervals are planned for sample collection considering that the berm has been disturbed. Of note for the NAS Skeet Range, the sampling is planned to encompass

both the older and more recent range orientations. For the two skeet ranges, contamination, if present, would primarily be expected from 0 to 6 inches, so the proposed 0 to 1 foot sample interval allows for site disturbance; in addition, several subsurface soil samples are includes to further assess vertical contamination/disturbance, if any. The sample location maps for the SI Work Plan have been attached to this Response to Comments document. It should be noted that sampling locations can be refined during the pre-field activities site visit to be conducted in October.

5. If the Site Investigation (SI) field activities are not intended to determine the nature and extent then the proposed sampling must be sufficient to adequately determine presence or absence of MEC and/or MC.

   Navy's RTC: Comment noted. The purpose of the SI is to determine presence or absence of MEC and MC, not to define the extent of MEC or MC contamination.

   MEDEP's follow up comment: MEDEP stands by its comment. Since the presence or absence of MEC and MC determine if an RI is needed then sampling is critical. The Navy needs provide enough information so that MEDEP is satisfied that the number and location of the samples is adequate to determine presence or absence. To that end please show on the figures the different components of the ranges and discuss how the target (berm) area and fall out zones, if disturbed, will be sampled.

   The sample location maps for the SI Work Plan have been attached to this Response to Comments document. It should be noted that sampling locations can be refined during the pre-field activities site visit to be conducted in October. Of note, the sampling locations are biased to where maximum concentrations are expected. Additionally, for sites involving MEC, some of the locations will be determined only AFTER anomalies (if any) are identified by the geophysical surveys to ensure that suspicious areas are included in the SI effort. For example, a temporary monitoring well would likely be placed near an anomaly to determine whether groundwater has been impacted. Of note, the Stakeholders will be provided with geophysical survey results and will be involved with determining sampling locations based on these results.

17. Table 3-1, Summary of MEC SI Activities, Munitions Bunker West Area, Activity: "Conduct a 100%, detector-aid, nonintrusive site survey..."

    It is unclear what is meant by 100% site survey. Does it mean that the 100% of the 29 acres will be visually surveyed and transects will be completed with a magnetometer or other detector? Please clarify the proposed procedure and how it will differ from the hand-held detector used for anomaly avoidance.

    Navy's RTC: TtNUS will conduct a surface sweep of the site and flag MEC, if any. TtNUS is preparing a separate Work Plan and HASP to address the MEC (including geophysical surveys) portion of the investigation.

    MEDEP's follow up comment: The Navy did not address what is meant by 100% site survey. Also will the separate work plan be part of this investigation or another phase?

    The text referencing the "100% site survey" has been removed from the work plan. The MEC Work Plan prepared by TtNUS will be submitted under separate cover prior to the

SI field activities and will detail the proposed work scope. The draft MEC Work Plan is anticipated for submittal in October 2007.

18. Table 3-1, Summary of MEC SI Activities, Site 12 EOD Area: Conduct a 100%, detector-aid, nonintrusive site survey..."

    It is unclear what is meant by 100% (100% of the 112.7 acres?); please clarify. (As stated in MEDEP comments on the Preliminary Assessment-Addendum, Navy personnel stated that the demolition area may have moved around within Site 12 and is not just the currently bermed area.)

    Navy's RTC: TtNUS will conduct a surface sweep of the site and will detonate any MEC encountered. TtNUS is preparing a separate Work Plan and HASP to address the MEC (including geophysical surveys) portion of the investigation.

    MEDEP's follow up comment: The Navy did not address what is meant by 100% site survey. Also will the separate work plan be part of this investigation or another phase?

    The text referencing the "100% site survey" has been removed from the work plan. The MEC Work Plan prepared by TtNUS will be submitted under separate cover prior to the SI field activities and will detail the proposed work scope. The draft MEC Work Plan is anticipated for submittal in October 2007.

21. Table 3-2:

    a.) At this time MEDEP can not concur with the number of surface soil samples proposed in this table.

    b.) Munitions Bunker Area West: 15 composite surface soil samples are proposed for a 29 acre site - roughly one composited sample per two acres. Please provide the rationale for this number of samples, the compositing scheme and what criteria will be used to determine where the samples will be taken.

    c.) Machine Gun Boresight Range: 5 composite surface soil samples are proposed for a 0.03 acres site (firing line to berm). This may be adequate if it can be determined whether the berm was removed or just graded out in the vicinity, however prior screening prior with a metal detector or an XRF will be needed before the number of soil samples can be determined. No groundwater samples are proposed, yet in the Preliminary Assessment it was estimated that 1.6 million rounds (30 and 50 caliber bullets) were discharged into the berm. Groundwater sampling must be performed unless it can be determined that the berm was removed and that expended rounds were collected and disposed of offsite.

    d.) NAS Skeet Range: 20 composite surface soil samples are proposed for a 73 acres site. Without prior screening to determine where to sample, this number is inadequate. The majority of the shot fallout zone appears to be over the streams and their floodplain wetland. Additional sediment sampling is necessary and due to the amount of run off and sediment from the storm drains it may be necessary to do some sediment sampling at depth.

    e.) Site 12 EOD: 25 composite surface samples may be adequate if only the bermed area (1.5 acres) is being characterized but it is not adequate for the entire 112.7 acres. The 3

groundwater samples may be adequate, however the site is shallow to bedrock and direct push may be necessary to just determine the amount of soil coverage. Ultimately it may be necessary to install bedrock wells.

f.) Topsham Annex Skeet Range: 20 composite surface soil samples are proposed for a 29 acres site. If the preliminary screening with an XRF or metal detector is performed to direct the sampling locations to the maximum shot fallout zone this may be adequate. MEDEP agrees with EPA regarding the necessity to sample groundwater at this site.

g.) State criteria for soil, groundwater, and surface water, if lower than that of Region 9, must also be used for comparison regarding for whether to continue with a Remedial Investigation. The sediment criteria agreed upon for the other IR sites should also be considered where appropriate.

Navy's RTC: The number of soil samples was based on the premise that the SI is not intended to characterize the sites but to determine the presence or absence of MEC and MC. Sampling locations will be biased to source areas where maximum concentrations would be expected.

MEDEP's follow up comment: The Navy response did not address the questions. Also the stated approach may work for undisturbed ranges where the firing point and target area are clear however that approach appears questionable for ranges that have been disturbed and area with no defined source areas.

The sampling program is based on a biased sampling approach designed to focus on locations of anticipated maximum concentrations (e.g., groundwater just beyond the shotfall area, soil within a berm). Furthermore, for MEC sites, surface sweeps and geophysical surveying will be used to identify suspicious locations for sampling (e.g., sample collection near MEC surface debris or subsurface anomaly). Additionally, groundwater sampling is planned at ALL of the six sites.

No surface water/sediment samples are planned for the NAS Skeet Range since the pond is actually a PAH treatment area and we would not be able to determine if the NAS Skeet Range was the source.

State criteria will also be used in determining the path forward for each of the sites.

Section 3.3.2 has been revised as follows:

3.3.2 MC Field Activities

Table 3-3-2 provides a summary of decisions made to address MC.

Table 3-2: Summary of MC SI Activities		
MRP sites	MC SI Activities	
	Activity	Purpose
Former Munitions Bunker West Area	Collect 15 composite surface soil samples and analyze for target analyte list (TAL) metals and explosives, polyaromatic hydrocarbons (PAHs), and propellants. Collect 10 discrete subsurface soil samples and analyze for TAL metals and explosives, PAHs, and propellants. Collect five groundwater samples and analyze for TAL metals, explosives, PAHs, and propellants.	CTC and site prioritization. Support MC NFA or RI determination. NFA determination is made if sample analytical data for site are below analyte-specific USEPA Region 9 Preliminary Remediation Goals (PRGs) for soil and USEPA Federal Drinking Water Standards for groundwater.
Machine Gun Boresight Range	Collect discrete soil samples from 12 locations: six locations surface only, six locations surface and two subsurface depth intervals. Total of 12 surface soil samples and 12 subsurface soil samples to analyze for TAL metals and propellants. Collect five groundwater samples and analyze for TAL metals and propellants.	CTC and site prioritization. Support MC NFA or RI determination. NFA determination is made if sample analytical data for site are below analyte-specific USEPA Region 9 PRGs for soil and USEPA Federal Drinking Water Standards for groundwater.
Skeet Range	Collect 20 discrete surface soil samples and analyze for TAL metals and PAHs. Collect 8 discrete subsurface soil samples and analyze for TAL metals and PAHs. Collect eight groundwater samples and analyze for TAL metals and PAHs.	CTC and site prioritization. Support MC NFA or RI determination. NFA determination is made if sample analytical data for site are below analyte-specific USEPA Region 9 PRGs for soil, and USEPA Federal Drinking Water Standards for groundwater.

Site 12 EOD	Collect 25 composite surface soil samples and analyze for TAL metals and explosives. Collect 10 discrete subsurface soil samples and analyze for TAL metals and explosives. Collect five sediment samples and analyze for TAL metals, explosives, and TOC. Collect five surface water samples and analyze for TAL metals, perchlorate, and explosives. Collect six groundwater samples and analyze for TAL metals, perchlorate, and explosives.	CTC and site prioritization. Support MC NFA or RI determination. NFA determination is made if sample analytical data for site are below analyte-specific USEPA Region 9 PRGs for soil and sediment, and USEPA Federal Drinking Water Standards for surface water and groundwater.
Quarry	Collect 10 composite surface soil samples and analyze for explosives. Collect 15 discrete subsurface soil samples and analyze for explosives. Collect five sediment samples and analyze for explosives and TOC. Collect five groundwater samples and analyze for explosives and perchlorate. Collect five surface water samples and analyze for explosives and perchlorate.	CTC and site prioritization. Support MC NFA or RI determination. NFA determination is made if sample analytical data for site are below analyte-specific USEPA Region 9 PRGs for soil and sediment, and USEPA Federal Drinking Water Standards for surface water and groundwater.

Topsham Annex Skeet Range	Collect 14 discrete surface soil samples and analyze for TAL metals and PAHs. Collect five discrete subsurface soil samples and analyze for TAL metals and PAHs. Collect two sediment samples and analyze for TAL metals, PAHs, and TOC. Collect five groundwater samples and analyze for TAL metals and PAHs. Collect two surface water samples and analyze for TAL metals and PAHs.	CTC and site prioritization. Support MC NFA or RI determination. NFA determination is made if sample analytical data for site are below analyte specific USEPA Region 9 PRGs for soil and sediment, and USEPA Federal Drinking Water Standards for surface water and groundwater.

The suggested method for Site 12 EOD Area and the Quarry surface soils is the Cold Regions Research and Engineering Laboratory (CRREL) seven sample wheel approach, as described in the CRREL Special Report 95-15, because these two sites were potentially associated with the detonation of explosives. Collection of discrete grab samples is proposed for surface soil samples at other sites and all sites requiring subsurface soil samples. Sediment and surface water samples will be obtained as discrete grab samples.

The goal of the field sampling activities for MC is to determine if the site has been impacted by MC. Anomaly avoidance techniques will be utilized during the MC field sampling activities, where necessary. Analytical data exceeding background levels and appropriate regulatory limits will be used as justification to move the site into the RI phase. The SI field sampling activities are not intended to determine the nature and extent of all contaminants.

All fieldwork will be of sufficient quality to meet the DQOs for the project as dictated in the QAPP. The details of the planned MC field sampling activities are provided in the SAP.

Investigative activities will be conducted based on information obtained during the PA process and data review. Complete sampling and analysis of relevant media will be conducted to assess the risk/hazard posed by any MC found at the site. The UXOSS will use visual observations and a magnetometer to detect the presence of MEC as a precaution will escort the team during fieldwork at MEC-suspected sites.

The following text is presented in Section 4.3 of the Sampling and Analysis Plan (Appendix A to the SI Work Plan):

Former Munitions Bunker West Area

MEC Activities: A detector-aided, non-intrusive site survey will be conducted over the walking path to sample locations in order to avoid MEC items.

MC Activities: Fifteen biased surface soil samples will be collected from an interval of zero to one foot. Ten discrete subsurface samples will be collected from an interval of one to three feet in depth. The locations of the soil samples will be determined in the field based on TtNUS surface sweep and MEC field assessments (conducted under a different work plan). One groundwater sample will be collected from each of five temporary wells that will be installed during the field work effort. The location of the wells will be determined based on the MEC field assessment conducted by TtNUS. Groundwater and soil samples will be analyzed for TAL metals, PAHs, propellants, and explosives using USEPA Methods 6010B, 8270C SIM, 8332/lab SOP, and 8330, respectively.

Machine Gun Boresight Range

MEC Activities: MEC presence is not anticipated; and so, activities associated with MEC are not applicable.

MC Activities: Twenty-four biased soil samples will be collected at this site based on visual observations and locations of the former range features. Six surface soil locations will be sampled in the zone between the firing point and the berm. Six locations at the berm will be sampled from the surface and two subsurface intervals since the berm was disturbed and turned over. Soil samples will be analyzed for TAL metals and propellants using USEPA Method 6010B and 8332/lab SOP, respectively. One groundwater sample will be collected from 5 different temporary wells that will be installed during the field work effort. The location of the wells will be one just down-gradient of the firing point, one in between the firing point and target area, and three just down-gradient of the berm.

Skeet Range

MEC Activities: MEC presence is not anticipated; and so, activities associated with MEC are not applicable.

MC Activities: Twenty biased surface soil, 8 subsurface soil, and eight groundwater samples will be collected at this site based on visual observations and locations of the former range features. All samples will be analyzed for TAL metals using USEPA Method 6010B and for PAHs using USEPA Method 8270C SIM. One groundwater sample will be collected from each of the seven temporary wells that will be installed at the Skeet Range during field activities. In addition, one groundwater sample will be collected from the existing well MW-NASB-078 for the firing point well. These eight groundwater samples will be analyzed for TAL metals and PAHs. Five of the temporary wells will be installed in the post-1950 Skeet Range Area: one just down-gradient of the firing point; one in between the firing point and the target area; and three surrounding the target area. The remaining two temporary wells will be installed in the pre-1950 Skeet Range Area within the likely shotfall zone.

Site 12 EOD Area

MEC Activities: A detector-aided, non-intrusive site survey will be conducted over the walking path to sample locations in order to avoid MEC items.

MC Activities: Twenty-five biased surface soil, ten subsurface soil, five sediment samples, five surface water, and six groundwater samples will be collected at this site. The locations of the soil

samples will be determined in the field based on TtNUS surface sweep and MEC field assessments (conducted under a different work plan). All samples will be analyzed for TAL metals using USEPA Method 6010B and for explosives using USEPA Method 8330. All sediment samples will be analyzed for TOC using method SW846 9060. Additionally, surface water and groundwater samples will be analyzed for perchlorate using USEPA Method 6850 (with filtration in the lab). Six temporary wells will be installed during the field work effort. One well will be placed just down-gradient of the existing bermed area and the remaining five well locations will be determined in the field based on TtNUS surface sweep and MEC field assessments. Surface water and sediment samples were determined based on site conditions and runoff topography. These locations can be found on Map 2-5 of the Work Plan.

Quarry

MEC Activities: A detector-aided, non-intrusive site survey will be conducted over the walking path to sample locations in order to avoid MEC items.

MC Activities: Ten biased surface soil, 15 subsurface soil, five sediment, five surface water, and five groundwater samples will be collected at this MRP site. Subsurface sample locations will be determined in the field based on TtNUS surface sweep and MEC field assessments (conducted under a different work plan). Subsurface soil samples will be collected from depth intervals of one to five feet. Five groundwater samples will be collected from five temporary wells to be installed during the field work effort. Groundwater sample locations will be determined in the field based on TtNUS surface sweep and MEC field assessments. Surface water and sediment sample locations were determined based on site conditions and runoff topography, these locations can be found on Map 2-6 of the Work Plan. All samples will be analyzed for explosives by USEPA Method 8330. Additionally surface water and groundwater samples will be analyzed for perchlorate using USEPA Method 6850 (with filtration done in the lab). Because of recent findings of debris at the site, the work scope is being expanded to include VOC, SVOC, pesticide, and PCB analyses for all media. This scope expansion will be addressed via a Work Plan Addendum to be submitted by TtNUS.

Topsham Annex Skeet Range

MEC Activities: MEC presence is not anticipated; therefore, activities associated with MEC will be for avoidance purposes only.

MC Activities: Fourteen biased surface soil, five subsurface soil, two sediment, two surface water, and five groundwater samples will be collected at this site. Surface soil samples will be collected based on visual observations and locations of the former range features. All samples will be analyzed for TAL metals using USEPA Method 6010B and for PAHs using USEPA Method 8270C SIM. The samples will be collected from five wells to be installed during the SI field effort. One well will be installed down-gradient from the firing point, one in between the firing point and the target area, and three surrounding the target area.

22. Section 3.3.2.2, Surface Water/Groundwater Sampling, para 2:

    a.) The Navy is proposing two groundwater samples for the NAS Skeet Range to be taken from an existing site well. Please clarify if the groundwater samples are to be taken from the two existing wells rather than one well. Please provide the monitoring well number(s) and

location(s). There are two existing wells (MW-NASB-077 and MW-NASB-078), which are located more in the vicinity of the firing point rather than the vicinity of the maximum shot fallout zone. Since there does not appear to be an existing monitoring well in the maximum shot fall out zone, please explain how the selected sampling point(s) will provide a representative groundwater sample for the skeet range.

Navy's RTC: The Navy is proposing 7 temporary wells at the NASB Skeet Range. Five of these wells will be in the post-1950 Skeet Range Area:...

MEDEP's follow up comment: This may be adequate. MEDEP would like to see the locations marked on a figure before accepting this response.

The sample location maps for the SI Work Plan have been attached to this Response to Comments document. It should be noted that sampling locations can be refined during the pre-field activities site visit to be conducted in October.

b.) The Navy is proposing three direct push wells for Site 12 EOD Area. This site appears to be shallow to bedrock. Direct push may be necessary to determine soil coverage at the site, however to obtain a representative groundwater sample it may be necessary to install bedrock wells. The 1990 Remedial Investigation information for Site 12 is limited to three test pits. What is the rationale for the three direct push locations? Depending upon the bedrock topography, shallow groundwater flow may be to the east, south or southwest, while migration in the bedrock will depend upon the dominant fractures, foliations, and other features. Additional information is required to determine the best location for sampling groundwater at the site.

Navy's RTC: The Navy proposes collection of surface soil samples from the surface (0-12 inches). The subsurface soil sample locations and depths will be established following the geophysical survey in consultation with the Navy and regulatory agencies.

The Navy is proposing installation of 6 temporary wells. One well will be positioned just downgradient of the bermed area. The remaining 5 temporary well positions will be determined in the field based on geophysical survey anomaly identification. Bedrock wells will not be installed during the SI phase.

MEDEP's follow up comment: (The first portion of the response is acceptable.) Regarding the temporary wells, if the soil depth is thin or on bedrock in the area of the EOD sites within Site 12 it will be impossible to evaluate impacts to groundwater without bedrock wells.

In the event that bedrock is shallow in the area of investigation, the Navy will install bedrock wells and investigate the groundwater beneath the site in the future during the RI phase of the project. Installation of bedrock wells is beyond the scope of an SI.

26. See general comment # 2 above.

See response to general comment #2 above.

27. See follow up comment #5 above.

The sample location maps for the SI Work Plan have been attached to this Response to Comments document. It should be noted that sampling locations can be refined during the pre-field activities site visit to be conducted in October.

28. See follow up comment #22.a above.

The sample location maps for the SI Work Plan have been attached to this Response to Comments document. It should be noted that sampling locations can be refined during the pre-field activities site visit to be conducted in October.

Attachment 2 (SOPs)

- MEDEP does not have copies of the following Standard Operating Procedures (SOPs) listed, so it cannot review S-5, S-11, and S-12.

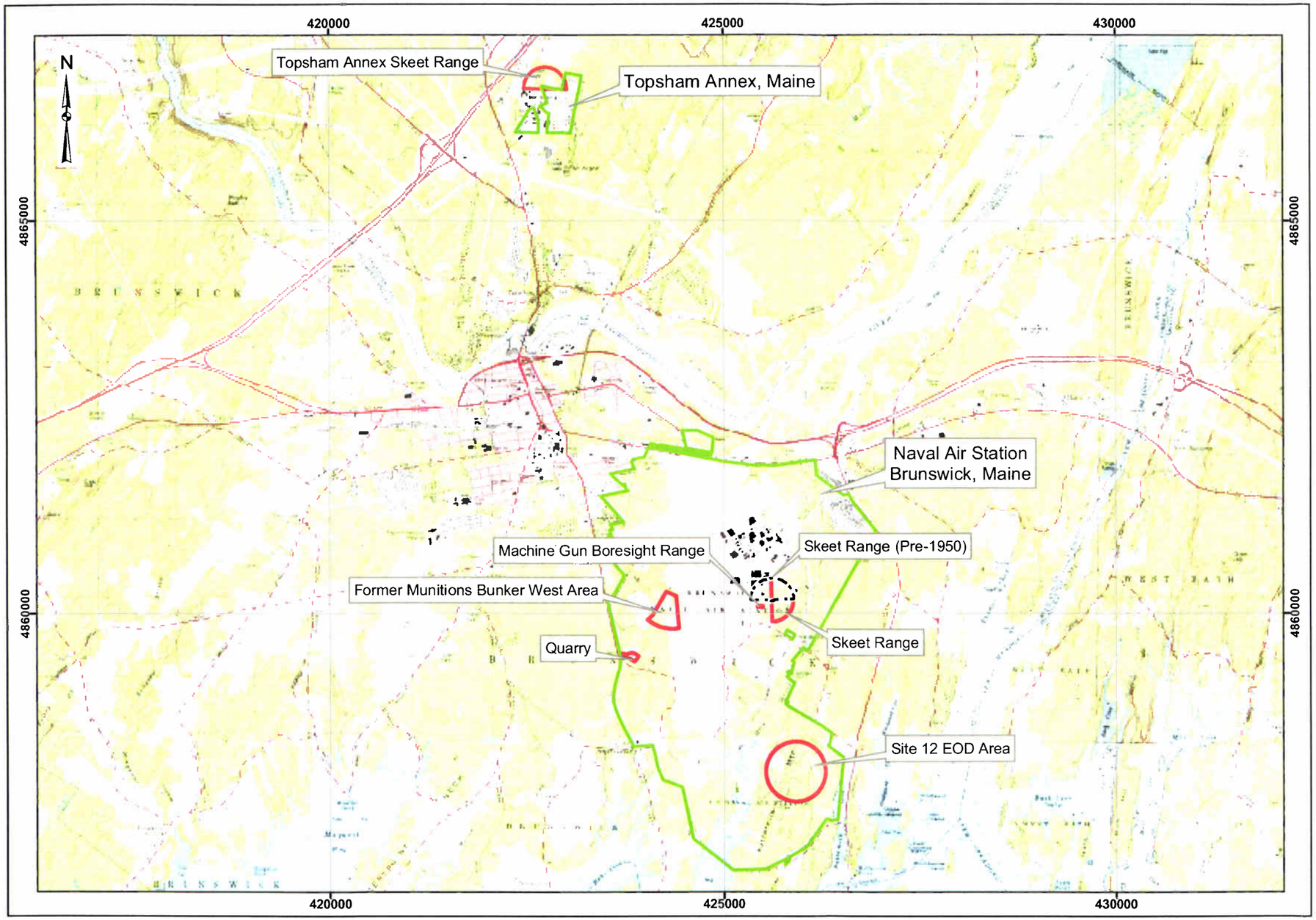
Malcolm Pirnie has been working closely with Tetra Tech, who is responsible for the QAPP. The Tetra Tech QAPP will be provided as an appendix to the Malcolm Pirnie SI Work Plan.

- Under "Temporary Well Abandonment" it must comply with Maine Chap 405 Sec 5 (H).

Comment noted.

- Under soil sampling for VOCs (S3/SA-1.3) MEDEP prefers whenever possible to use the freezing method. If it can be employed please include it in the SOP.

Analytical procedures are not included in the SOP for soil sampling SOP (S3/SA-1.3) but will be addressed in the QAPP worksheets by Tetra Tech. Volatile organic soil samples will be collected using the EnCore sampler (3 aliquots per sample location) and shipped to the laboratory for preservation. If the soil samples are determined to be reactive with sodium bisulfate (low level preservative), then the laboratory should preserve the soil samples by placing 5 grams of soil (contents of EnCore Sampler) in 5 mL of organic-free reagent grade water in a standard VOA vial. The vial should then be capped, placed in a Ziplock bag and placed in the freezer at -10°C for storage until analysis. Preservation must occur within 48-hours of sample collection and analysis must occur within 14 days of sample collection.



Site Inspection Work Plan
Naval Air Station Brunswick, Maine

MALCOLM PIRNIE

Map 2-1
Location Map

Legend

- Installation Boundary
- Ranges
- Skeet Range (Pre-1950)

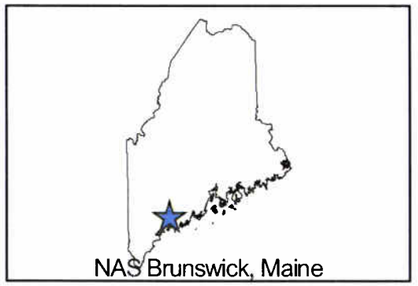
DRAFT

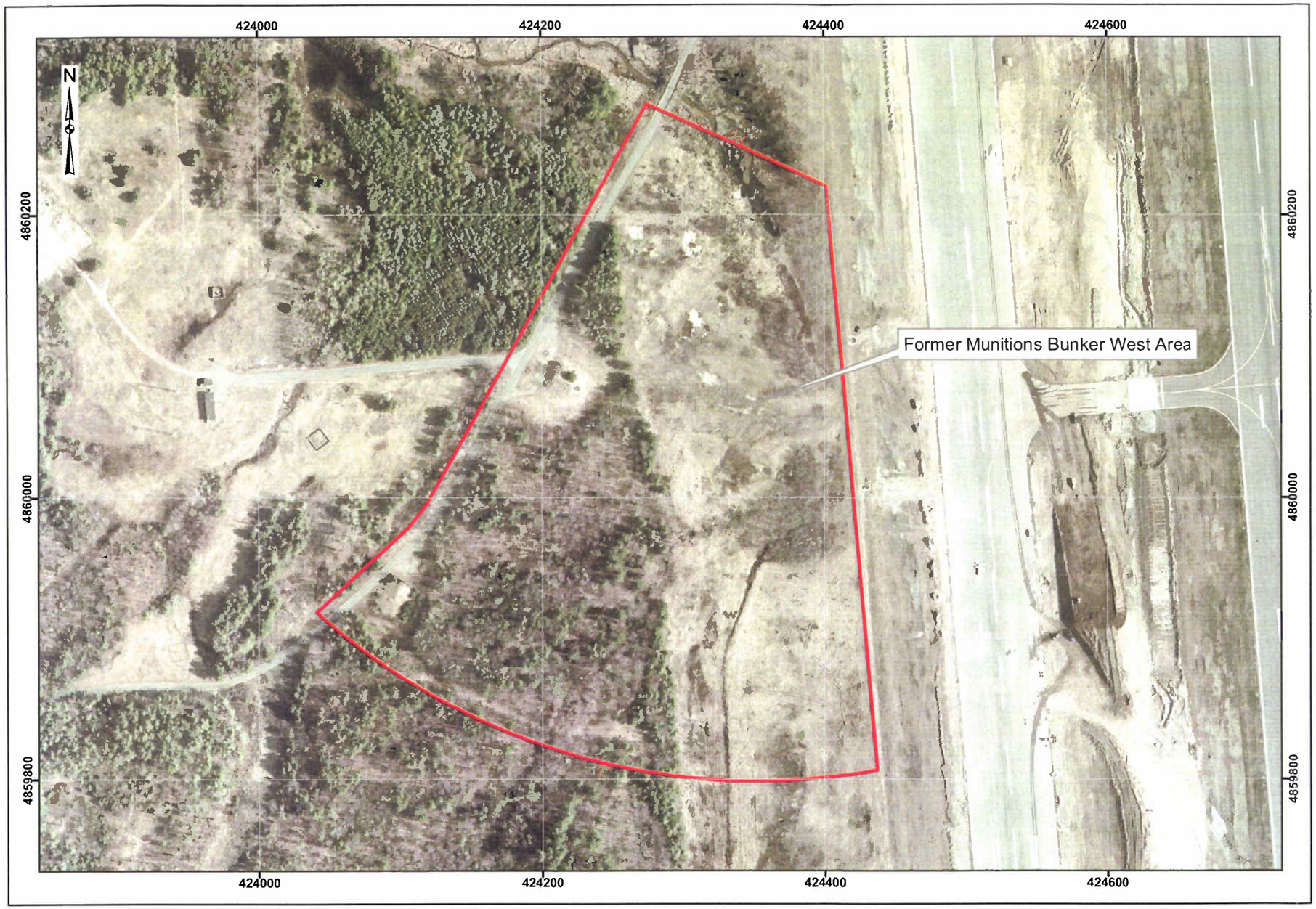
0 1,000 2,000 3,000 Meters

Data Source: USGS. 7.5 Minute Series
Topographic Survey - Bathymetric Map
Brunswick, ME, 1980
Orrs Island, ME, 1978
Lisbon Falls South, ME, 1990
Freeport, ME, 1970
Phippsburg, ME, 1997
Bath, ME, 1985

Coordinate System: UTM Zone 19N
Datum: NAD 83
Units: Meters

Contract: N62472-02-D-1300
Edition: Final Site Inspection Work Plan
Date: June 2007





Site Inspection Work Plan
Naval Air Station Brunswick, Maine

MALCOLM PIRNIE

Map 2-2
Former Munitions Bunker West Area

Legend

- Installation Boundary
- Former Munitions Bunker West Area

DRAFT

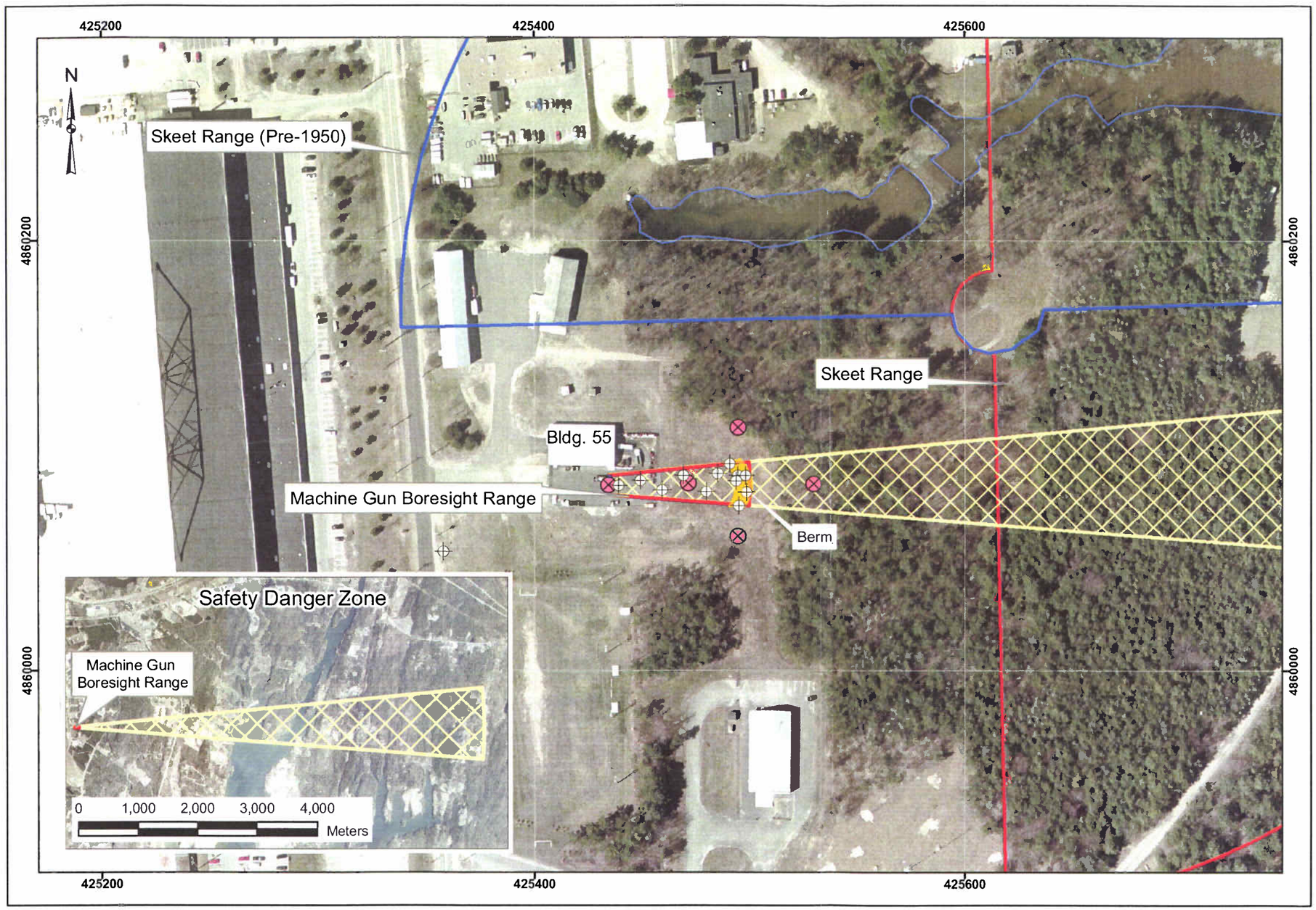
Note: Soil.and ground water well locations will be determined after the TtNUS MEC field assessment. There will be 15 surface soil samples, 10 subsurface soil samples, and 5 temporary ground water sampling wells.

0 50 100 150 200 Meters

Data Source:
Maine Office of GIS, ORTHO_HF, 2003

Coordinate System: UTM Zone 19N
Datum: NAD 83
Units: Meters

Contract: N62472-02-D-1300
Edition: Final Site Inspection Work Plan
Date: June 2007



Site Inspection Work Plan
Naval Air Station Brunswick, Maine

MALCOLM PIRNIE

Map 2-3
Machine Gun Boresight Range

Legend

- Installation Boundary
- Ranges
- Surface Danger Zone
- Skeet Range (Pre-1950)
- Berm
- Firing Line
- Water Body
- Proposed Monitoring Groundwater Wells
- Soil Sample Locations*

DRAFT

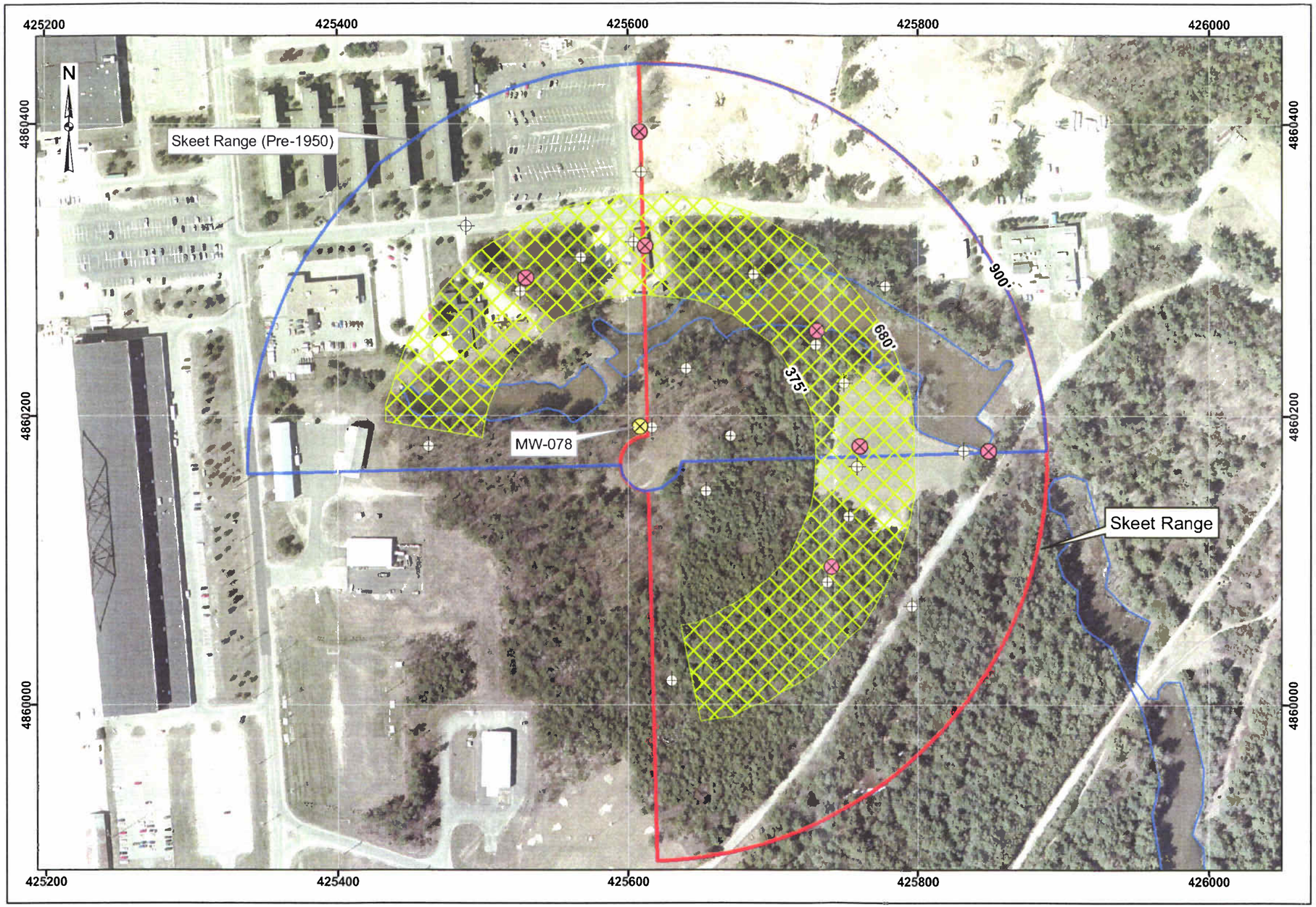
*There will be 12 soil sample locations and a total of 24 soil samples collected. Six locations in the zone between the firing point and the berm - surface interval only. Six locations at the berm - surface interval and two subsurface depth intervals.

0 25 50 75 100 Meters

Data Source:
Maine Office of GIS, ORTHO_HF, 2003

Coordinate System: UTM Zone 19N
Datum: NAD 83
Units: Meters

Contract: N62472-02-D-1300
Edition: Final Site Inspection Work Plan
Date: July 2007



Site Inspection Work Plan
Naval Air Station Brunswick, Maine

MALCOLM PIRNIE

Map 2-4
Skeet Range

Legend

- Skeet Range
- Skeet Range (Pre-1950)
- Likely Shotfall Zone
- Water Body
- Surface Soil Sample Locations

Ground Water Wells

- Current Monitoring Well
- Proposed Additional Monitoring Well

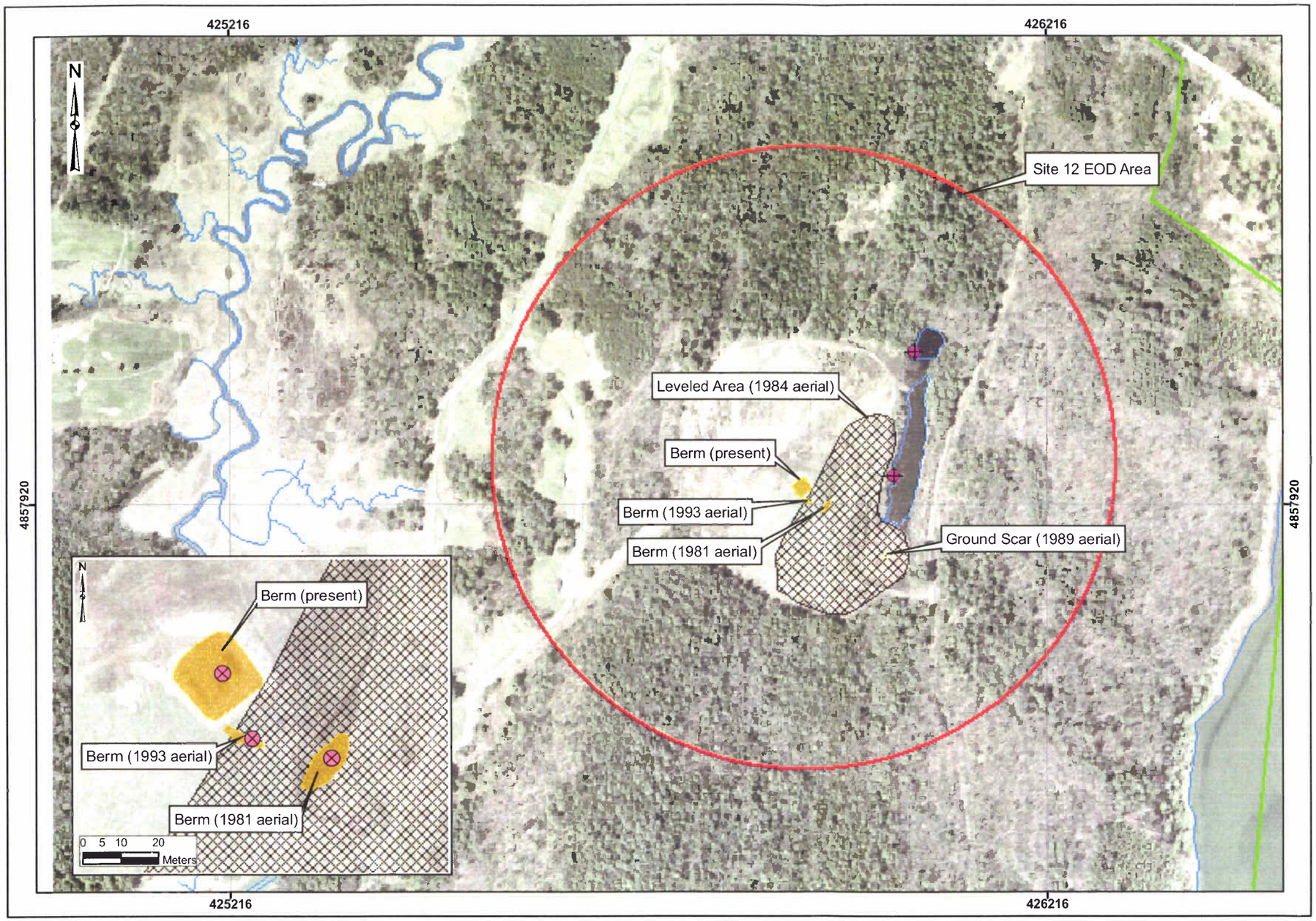
DRAFT

0 25 50 75 100 Meters

Data Source:
Maine Office of GIS, ORTHO_HF, 2003

Coordinate System: UTM Zone 19N
Datum: NAD 83
Units: Meters

Contract: N62472-02-D-1300
Edition: Final Site Inspeciton Work Plan
Date: June 2007



Site Inspection Work Plan
Naval Air Station Brunswick, Maine

MALCOLM PIRNIE

Map 2-5
Site 12 EOD Area

Legend

Site Features

- Leveled Area
- Other Feature
- Berm
- Installation Boundary
- Site 12 EOD Area
- Water Body
- Surface Water and Sediment Sample Locations
- Proposed Monitoring Ground Water Wells

DRAFT

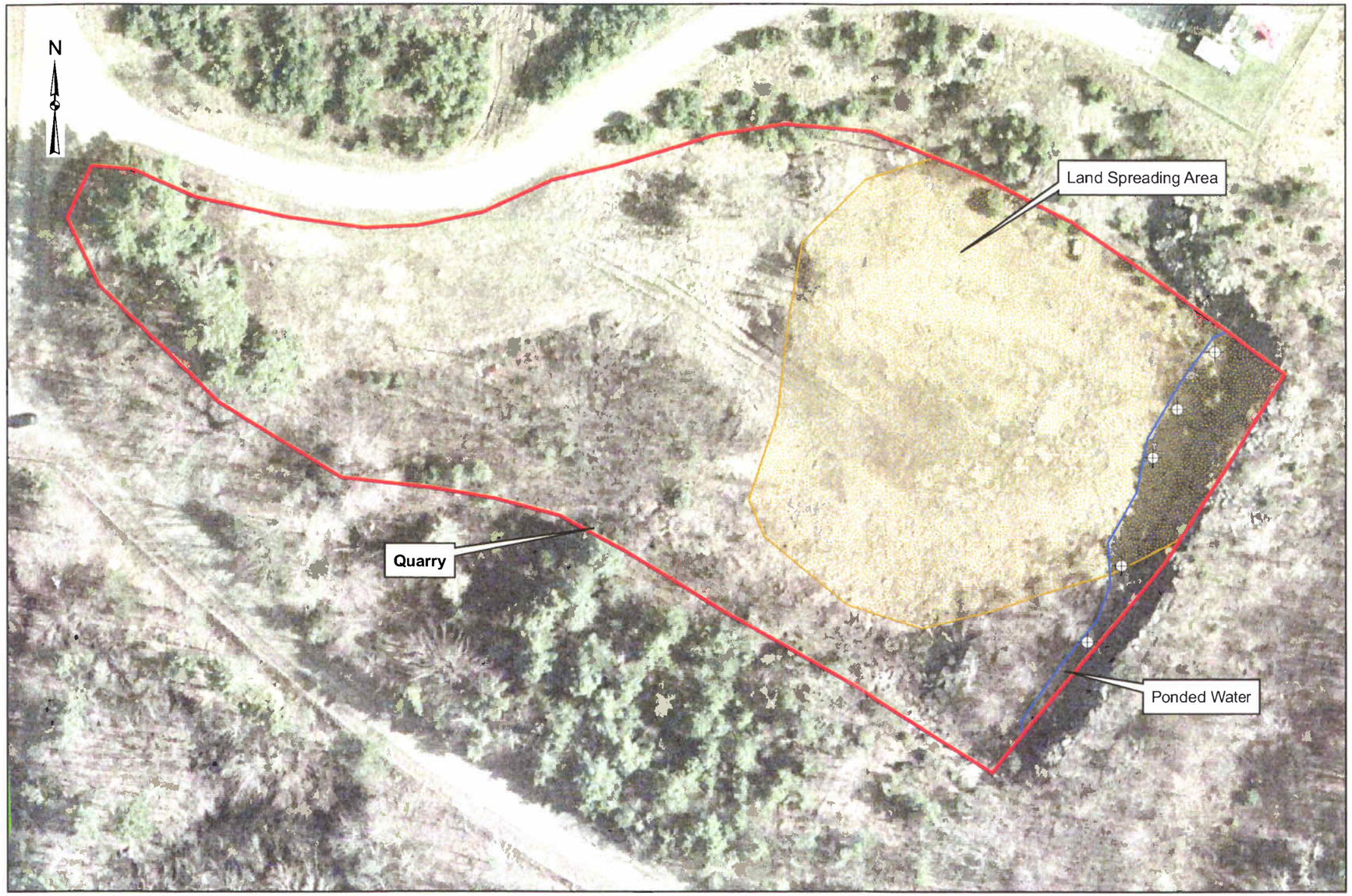
Note: Three additional ground water well locations and 25 soil samples will be determined after TtNUS MEC study.

0 75 150 225 300 Meters

Data Source:
Maine Office of GIS, ORTHO_HF, 2003

Coordinate System: UTM Zone19N
Datum: NAD83
Units: Meters

Contract: N62472-02-D-1300
Edition: Final Site Inspection Work Plan
Date: June 2007



Site Inspection Work Plan
Naval Air Station Brunswick, Maine

MALCOLM PIRNIE

Map 2-6
Quarry

Legend

- Installation Boundary
- Quarry
- Land Spreading Area
- Water
- Sediment & Surface Water Sample Locations

DRAFT

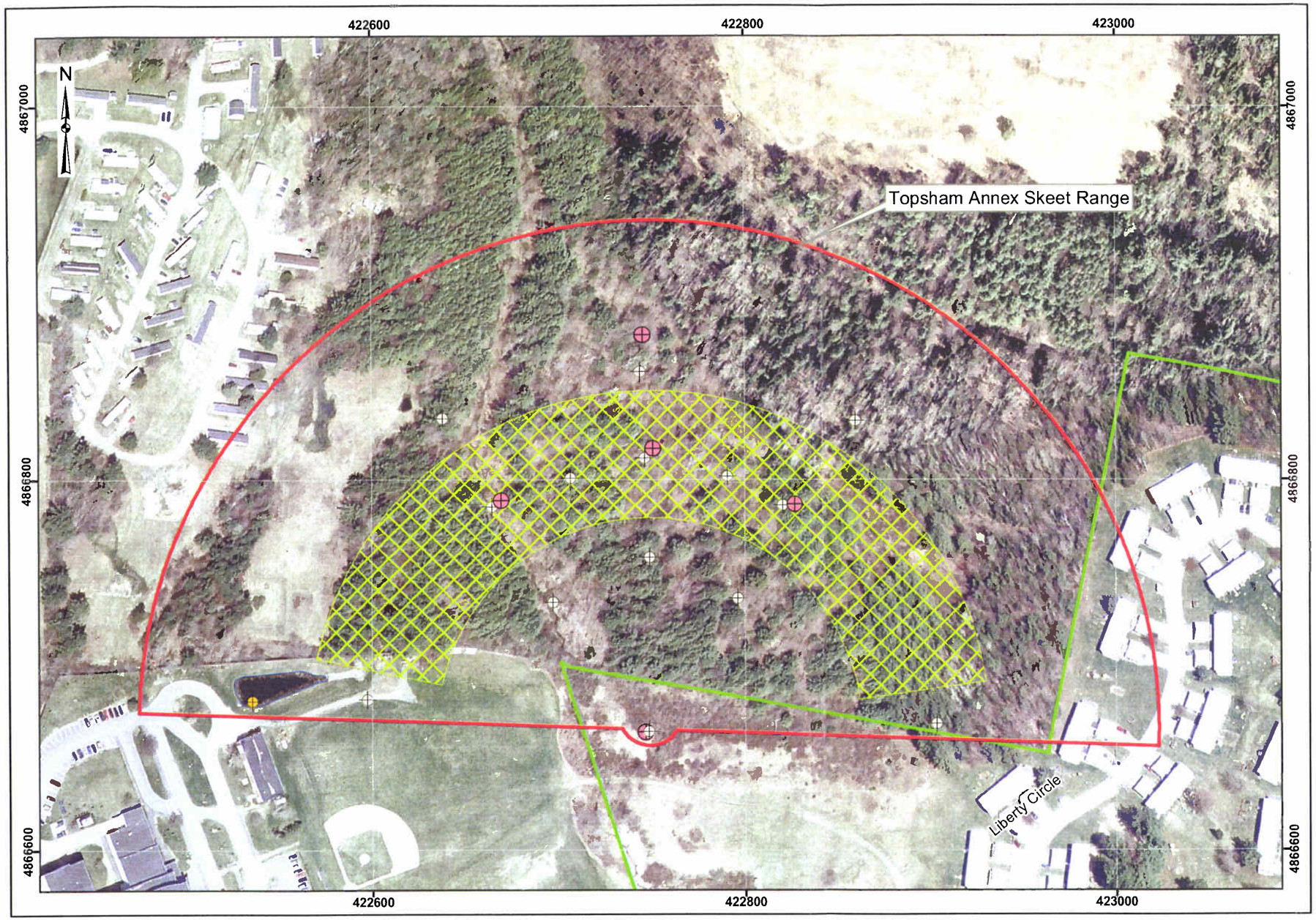
Note: The 25 soil samples and five groundwater monitoring well locations will be determined after the TtNUS MEC study.

0 10 20 30 40 Meters

Data Source:
Maine Office of GIS, ORTHO_HF, 2003

Coordinate System: UTM Zone19N
Datum: NAD83
Units: Meters

Contract: N62472-02-D-1300
Edition: Final Site Inspection Work Plan
Date: June 2007



Site Inspection Work Plan
Topsham Annex, Maine

MALCOLM PIRNIE

Map 2-7
Topsham Annex Skeet Range

Legend

- Installation Boundary
- Topsham Annex Skeet Range
- Water Body
- Likely Shotfall Zone
- Proposed Additional Monitoring Well
- Soil Sample Location
- Surface Water and Sediment Sample Location

DRAFT

0 40 80 120 160 Meters

Data Source:
Maine Office of GIS, ORTHO_HF, 2003

Coordinate System: UTM Zone 19N
Datum: NAD 83
Units: Meters

Contract: N62472-02-D-1300
Edition: Final Site Inspection Work Plan
Date: June 2007

D-3 MAY 2007 RESPONSE TO STAKEHOLDER COMMENTS

Klink, Linda

From: Akbas, Terri [TAkbas@PIRNIE.COM]
Sent: Tuesday, May 22, 2007 10:36 AM
To: williams.christine@epamail.epa.gov; Claudia.B.Sait@maine.gov; calepage@adelphia.net
Cc: Monaco, Orlando J CIV NAVFAC MIDLANT; Joy, Lisa M CIV NAS Brunswick, N45; Mosher, Dale CIV NAS BRUNSWICK, Environmental; Waddill, Dan W CIV NAVFAC Lant; Wright, Jennifer H CIV NAVFAC Lant; Klink, Linda; douglas.murray1@navy.mil; Smith, David Gregory
Subject: Response to Comments Documents - NASB PA Addendum and SI Work Plan

Attachments: RTC Brunswick PA Addendum.pdf; RTC Brunswick SI WP.pdf

RTC Brunswick PA Addendum.pdf ... RTC Brunswick SI WP.pdf (327 K...

Good Morning All,

Attached are the RTC documents for the PA Addendum and the SI Work Plan for NASB. Malcolm Pirnie has worked together with the Navy and TtNUS to prepare the responses to these comments. Hardcopy versions of these documents will be sent via regular mail.

Upon approval of these responses, Malcolm Pirnie will issue the Final versions of these documents.

Regards,
Terri

Terri L. Akbas, QEP
Project Scientist
Malcolm Pirnie, Inc.
104 Corporate Park Drive
White Plains, NY 10602
(914) 641-2414 (voice)
(914) 641-2455 (fax)

Malcolm Pirnie Response to Comments on the
February 2007 Draft Final Site Investigation Work Plan
Naval Air Station Brunswick, Maine

Malcolm Pirnie is pleased to provide this Response to Comments for comments received on the January 2007 Draft Final Site Investigation Work Plan for Munitions Response Program Sites at Naval Air Station Brunswick. In order to respond effectively, the original comments have been reproduced below. Malcolm Pirnie's responses are displayed in ***bold italics.***

Draft Final Site Investigation Work Plan (February 2007) – Comments from Christine Williams (USEPA – Region 1):

General Comments:

1) The SI Work Plan proposes a level of effort that is generally appropriate for the stated purpose, and should reveal MEC/MC contamination, if present in the areas evaluated. However, while the work plan gives a general description of the investigation it does not contain specific details on how the investigation is to be performed. The QAPP needs to be completed before EPA can agree to the work.

TtNUS will be completing the QAPP.

2) The Work Plan does not propose to analyze soil samples from the skeet ranges for propellant compounds near the firing points. Please provide the rationale. Similarly, the Work Plan proposes to analyze only for lead. Please provide the rationale for neglecting other metals, particularly antimony, which is sometimes present in shot.

Usually black powder is the propellant for shotguns. Black powder is a mixture of finely pulverized potassium nitrate or sodium nitrate, sulfur, and charcoal. Potassium and sodium nitrates are used in pesticides and could likely appear in the soil due to use of pesticides on the land surrounding the skeet ranges. Sulfur is a ubiquitous element.

Lead is the primary soil contaminant of concern at the skeet range and will be the best indicator of an environmental issue. Antimony, arsenic and nickel also contribute to soil contamination. Soil samples will be analyzed for the entire Target Analyte List of metals.

3) Groundwater sampling is proposed only for Site 12, where the need is quite compelling, and for the NASB Skeet Range, where there is an existing well. Please provide the rationale for concluding a priori that groundwater impacts are not expected, particularly for the Former Munitions Bunker West Area and the Machine Gun Boresight Range. At these two sites, the proposed soil sampling recognizes the potential for contamination by propellant compounds. Given that some of these compounds (e.g., nitroglycerine) can be fairly mobile, there may be potential for groundwater impacts.

Groundwater impacts will be investigated further. Five temporary shallow groundwater wells will be installed at the former Munitions Bunker West Area in order to analyze groundwater for perchlorate that was deposited on the site through the use of pyrotechnics. These well locations will be determined in the field based on the results of the geophysical survey conducted by TtNUS. Five temporary groundwater wells will be installed at the former Machine Gun Boresight Range at the following locations: one just downgradient of the firing pint, one between the firing point and the target area

(berm), and three just downgradient of the berm.

4) The likelihood of MEC and MC is dismissed for the Quarry site, based on the argument that EOD activity there is "undocumented," and on a visual inspection. However, such activity has often been carried out without formal record keeping. In addition, given that the Quarry site was used for land farming in the 1990s, and, presumably, was graded repeatedly, the visual inspection is not conclusive. Some minimal sampling and analysis at the Quarry should be carried out in order to support the case for NFA. It is recommended that subsurface soils, groundwater (if above the bedrock surface), and surface water from the on-site pond be sampled and analyzed for explosives compounds and perchlorate.

An SI will be conducted at the Quarry. Analyses will be conducted for explosives compounds for all media (groundwater, soil, surface water, and sediment). Additionally, groundwater analyses for perchlorate will be conducted.

5) The importance of metals in this investigation (analyses of at least Pb, and, for several sites the TAL, are proposed) raises the question of whether or not adequate "background" values have been established for NASB for soil, groundwater, and surface water. Has a "background" data set been assembled for past NASB environmental investigations, or is such an effort planned in conjunction with forthcoming investigations?

A Basewide Background Study is going to be conducted at NASB. ECC prepared the Work Plan for this investigation. TtNUS will be implementing the investigation. If this background data is available at the time of the SI report, analytical results will be compared to background data.

Specific Comments:

6) p. 1-1, sec. 1: The text notes that deviations from the Work Plan will be brought to the attention of the Navy RPM. Please verify that such deviations will also be brought to the attention of state (MEDEP) and federal (EPA) regulators.

Concur. The text will be revised to reflect that MEDEP and EPA will be notified by the Navy of deviations from the WP.

7) p. 2-3, sec. 2.3: It is agreed that lead is a primary concern at the Machine Gun Boresight Range, particularly given the possibility that the backstop berm simply may have been graded out in place. It should be noted that other metals are also of concern, including antimony, which, although present in typical projectiles at lower mass fraction than lead, also has lower cleanup standards because of its greater mobility. (For example, the EPA Region IX PRG for residential soil for Sb is 31 mg/kg; that for Pb is 400 mg/kg). Other alloying or jacketing elements, such as As and Cu are also of interest.

Concur. Full TAL metals will be analyzed.

8) p. 2-4, sec 2.5 Site 12 Explosive Ordnance Disposal (EOD) Area: The second paragraph states "based on quantities involved, low level contamination of unburned explosive residues and elevated concentration of lead, . . . and perchlorate may be expected in the soils". However, Table 3-3: Sample

Summary Table, does not list any soil samples being analyzed for perchlorate. Please include or provide a rationale for not including.

The text will be revised as follows: "Perchlorate may have been present at the site." Perchlorate will be added to the list of analytes for surface water and groundwater for this site.

9) p. 2-5, sec. 2.6: The description of the Quarry site states, "It is assumed that there are no MC associated with the Quarry since there is no evidence of munitions use at the Quarry." The lack of evidence in the visual survey performed for the Preliminary Assessment is somewhat equivocal, because the land farming carried out at the Quarry may have obscured visual evidence for historical EOD activities. It should be verified that there are no MC through sampling and analysis, rather than assuming the result from limited observations.

An SI will be conducted at the Quarry.

10) p. 3-1, sec. 3: *typo:* Please change, "The Final SI WP will *incorporates* decisions ..." to, "The Final SI WP will *incorporate* decisions ... ."

Concur. The suggested editorial change was made.

11) p. 3-2, sec. 3.3: *typo:* Please change, "The *goal* of the planned field activities are ..." to, "The *goals* of the planned field activities are ... ."

Concur. The suggested editorial change was made.

12) p. 3-3, sec. 3.3.1, Table 3-1: The table states that, if the surface survey does not identify any MEC, then NFA with respect "MEC at the surface" will be recommended. What assurance is there that the area has not been graded since it was used for training, such that MEC formerly at the surface is now buried? What assurance is there that no disposal activities (e.g., burn pits) were carried out in the area? The magnetometer survey is described as "for avoidance purposes only." What is the anticipated sensitivity of the magnetometer (e.g., depth and size of anomalies that might be detected), and what is the proposed line density?

TtNUS is preparing a separate Work Plan and HASP to address the geophysical portion of the investigation for MEC. For the MC investigation, a TtNUS UXO specialist will serve as an escort for avoidance purposes during sample collection.

13) p. 3-5, sec. 3.3.2, Table 3-2: The table shows that all proposed soil sampling will entail compositing. Please provide further details on the proposed compositing scheme. How many subsamples will be composited for each location? Over what length scale will the subsamples be collected?

The suggested method for Site 12 and the Quarry surface soils is the CRREL seven-sample wheel approach as described in the CRREL Special Report 96-15 because these two sites were potentially associated with the detonation of explosives. Reference to this method has been added to the text. Collection of discrete grab samples is proposed for the subsurface soils (and surface water/sediment) at

Site 12 and the Quarry, as well as for the surface and subsurface soils and surface water/sediment at the other sites.

14) p. 3-5, sec. 3.3.2, Table 3-2: The "Activity" entry for the Former Munitions Bunker West Area shows that soil samples will be collected for each location "from two different depths averaging 4 feet." What specific depths are proposed? Please clarify.

Fifteen surface soil samples will be collected from the 0-1 foot interval. Ten discrete subsurface soil samples will be collected from 1-3 foot interval. The locations of the subsurface soil samples will be determined in the field based on the results of the TtNUS surface sweep and MEC field assessment.

15) p. 3-5, sec. 3.3.2, Table 3-2: The "Activity" entry for the Machine Gun Boresight Range shows that soil samples will be analyzed for lead. This should be expanded to the full TAL metals. In particular, it is not known in advance that the machine gun projectiles did not contain other metals, such as As, Sb, and Cu, or that tracers were not fired.

Concur. Full TAL metals will be analyzed.

16) p. 3-5, sec. 3.3.2, Table 3-2: The "Activity" entry for the Machine Gun Boresight Range indicates no investigation of groundwater. If propellants in soil are of interest, it would seem that there may be groundwater impacts from leaching. Please provide the rationale for neglecting groundwater.

Groundwater impacts will be investigated further. Five temporary groundwater wells will be installed at the former Machine Gun Boresight Range at the following locations: one just downgradient of the firing pint, one between the firing point and the target area (berm), and three just downgradient of the berm.

17) p. 3-6, sec. 3.3.2, Table 3-2: The "Activity" entry for the Quarry indicates that there is no evidence of munitions use at the Quarry, and therefore no MC characterization is proposed. The lack of evidence in the visual survey performed for the Preliminary Assessment is somewhat equivocal, because the land farming carried out at the Quarry may have obscured visual evidence for historical EOD activities. It should be verified that there are no MC through sampling and analysis, rather than assuming the result from limited observations. It is recommended that subsurface soil samples be collected and analyzed, groundwater (if found above the bedrock surface), and surface water in the pond.

An SI will be conducted at the Quarry.

18) p. 3-6, sec. 3.3.2, Table 3-2: The "Activity" entry for the Topsham Annex Skeet Range indicates no investigation of groundwater. This appears to be inconsistent with the proposed groundwater sampling for the skeet range at NASB. Please provide the rationale for neglecting groundwater.

Five temporary wells will be installed at each skeet range: one downgradient from the firing point, one in between the firing point and the target area, and 3 just downgradient of the target area. The existing well at the NASB skeet range will be used as the firing point well.

19) p. 3-7, sec 3.3.2.3 Laboratory Analysis: Change "Method 8330" to "Method 8330B". Change "Method 314.0" to "Method 314.1". If soils are to be analyzed for perchlorates, use Method 6850. Note these methods appear in other parts of this document and those parts will need to be changed to the new methods.

Based on the limited availability of commercial labs offering method 8330B as well as concerns that have been raised regarding the preparation step in this new method, the Navy feels that Method 8330 is more technically defensible for explosives analysis. The Navy also recommends using Method 331 (with filtration at the laboratory) instead of Method 314.1 for perchlorate analysis. The text will be revised to reflect these analytical methods.

SAMPLING & ANALYSIS PLAN:

20) p. 4-1 -4-2, 4 Field Activities: This Section discusses collecting composite surface samples, but does not explain how the compositing will be performed. For compositing explosive residue, see Section A.4.0 Secondary Explosive and Propellant Residues (Method 8330B Appendix A) for information on compositing samples. Add the procedure for collecting composite samples to the Sampling and Analysis Plan.

The suggested method for Site 12 and the Quarry surface soils is the CRREL seven-sample wheel approach as described in the CRREL Special Report 96-15. Reference to this method has been added to the text. Collection of discrete grab samples is proposed for the subsurface soils at Site 12 and the Quarry, as well as for the surface and subsurface soils at the other sites.

21) p. 4-2, sec.4.9 Sampling: The first paragraph states "soil/sediment will be collected as composite samples and water samples will be obtained as discrete samples based on USEPA standard operating procedures". Identify the USEPA standard operating procedures.

Sediment will be collected as discrete grab samples. The USEPA standard operating procedures have been identified in the text.

22) p. 4-5, sec 4.15 Laboratory Analysis: The section states "the method detection limits for these methods are included in the QAPP". The detection limits are missing from the QAPP. Please provide.

TtNUS will be producing the QAPP.

QUALITY ASSURANCE PROJECT PLAN (QAPP):

23) The QAPP is incomplete (major information is missing). Most of the worksheets are marked as "TBD". The QAPP needs to be completed before it can be reviewed.

TtNUS will be producing the QAPP.

Malcolm Pirnie Response to Comments on the
February 2007 Draft Final Site Investigation Work Plan
Naval Air Station Brunswick, Maine

Draft Final Site Investigation Work Plan (February 2007) – Comments from Richard Willey, Hydrogeologist (USEPA – Region 1):

In general the subject document appears to be well organized and reasonably thorough. However, some comments are warranted.

General Comments:

1) While the acronyms MEC and MC are defined, the reader (public) should be provided with a more specific understanding of what munitions and explosives of concern, and/or munitions constituents will be sampled and analyzed for.

Concur. A discussion of MEC and MC will be added to the text.

2) Suggest adding a table showing a "master list" of contaminants of concern (COCs) and the six sites under this investigation. Check marks could be used to indicate which analytes would be sampled for at each site. The list of COCs might also be compared to information from MMR or other DOD sites that had similar activities.

The COCs are identified by site and are summarized in the Site Description provided in the WP. A table will be added to Section 2 listing the COCs for each site. Those that will be analyzed are identified in the Field Activities section (see Table 3-3).

3) The work plan should provide some justification for the blanket exclusion of certain types of contaminants (for example, MECs) at a specific site.

The Site Description section of the Work Plan discusses the contaminants of concern for each site as well as the reason(s) that types of contaminants are not considered for the area.

4) While there appears to be little need for concern regarding contamination at the quarry site, explosives were no doubt used during quarrying operations and there is no indication of what materials may have been present in the "land Spreading" materials. Prudence and sensitivity to possible community concerns, suggests that some level of confirmatory data are advisable, and in order to reach "closure" on the subject.

An SI will be conducted at the Quarry. Text has been added to the Work Plan to discuss the SI activities at the Quarry.

5) Procedurally, it would be worthwhile to specify times when oversight personnel might meet with project representatives to track progress and to identify potential data gaps and course corrections (in a timely manner) from a regulatory perspective.

The SI MEC Work Plan for Site 12, the Quarry, and Munitions Bunker West will entail surface sweeps and/or geophysical surveys and MEC field assessment before establishing some or all of the SI MC sampling locations. At this point in the project, TtNUS will consult with the Navy and regulatory agencies to discuss results and establish remaining sampling locations. Another point for consultation with the Navy and regulatory agencies will be the determination of whether temporary wells should be abandoned or converted to permanent wells. Additionally, overall progress will be discussed during routine conference calls and technical meetings. The SI Report will identify any data gaps, if present, for a given site and make recommendations to address any data gaps during a subsequent RI.

Malcolm Pirnie Response to Comments on the
February 2007 Draft Final Site Investigation Work Plan
Naval Air Station Brunswick, Maine

Draft Final Site Investigation Work Plan (February 2007) – Comments from Claudia Sait (MEDEP):

General Comments:

1. Until MEDEP's comments on the draft Preliminary Assessment Addendum are satisfactorily addressed MEDEP cannot agree with eliminating the Quarry from the Site Inspection.

An SI will be conducted at the Quarry. Text has been added to the Work Plan to discuss the SI activities at the Quarry.

2. MEDEP anticipates that the former munitions bunkers west of the runway which were used to store high explosives and incendiary devices will be handled under the Resource Conservation and Recovery Act (RCRA) program as part of the base closure process.

Comment noted.

3. For the sites with lead (i.e., Naval Air Station skeet range, Topsham Annex skeet range, and the Machine Gun Bore Sight Range) as a potential contaminant MEDEP suggests screening the areas with an X-Ray Florescence (XRF) or metal detector prior to sampling to determine the boundaries and maximum shot fallout zones. It would be extremely helpful in determining if the Machine Bore Sight Range berm was graded in place rather than removed. By the Navy own documents (Preliminary Assessment February 2006) it estimates that 1,641,600 rounds of lead was discharged at this site. The XRF or metal detector would also be valuable for determining if the original NAS skeet range which was oriented to the north still has pellets on the embankment since the stream was dredged in the 1990s to create impoundment ponds.

The purpose of the SI is to determine presence or absence of MEC and MC, not to define the extent of MEC or MC contamination. Conventional soil sampling techniques will be utilized during the SI phase and locations will be biased to source areas where maximum concentrations would be expected. If warranted, an XRF will be utilized during the RI phase.

4. Please provide the list of the potential propellants and explosives for each site.

Concur. This information will be added to Section 2 (Site Description) for each site.

5. If the Site Investigation (SI) field activities are not intended to determine the nature and extent then the proposed sampling must be sufficient to adequately determine presence or absence of MEC and/or MC.

Comment noted. The purpose of the SI is to determine presence or absence of MEC and MC, not to define the extent of MEC or MC contamination.

6. Without a layout of where the sampling points are to be located it is impossible to determine the adequacy of the proposal. The SI overall lacks detail in the form of proposed sample, boring and well

locations, descriptions of compositing procedures, and detail of transect or grid size for the anomaly surveys. The text also indicates that locations will be based on features detected in the initial surveys and on site specific observations or former features. This suggests a two phase process is needed so that the final locations can be determined after the initial surveys are completed.

Sample locations will be determined in the field based on the results of the geophysical survey and MEC field assessment conducted by TtNUS for Site 12, Munitions Bunker West Area, and the Quarry in consultation with the Navy and regulatory agencies. Sample locations for the Machine Gun Boresight Range and the two skeet ranges have been added to the maps in the work plan.

7. A Quality Assurance Project Plan must be provided as part of the workplan. The QAPP cannot be approved or reviewed with so many fields listed as "TBD".

The QAPP will be produced by TtNUS.

Specific Comments:

8. Section 1, para 3: Any necessary deviations from the plan will be brought to the attention of the NAVFAC, MidAtlantic ..."

Concur. The text will be revised to reflect that MEDEP and EPA will be notified by the Navy of deviations from the WP.

9. Section 2.2, para 3: In addition, the potential exists for MC in the surface soil. Contaminants potentially present at the site include..."

Please add perchlorate as a potential contaminant in groundwater.

Concur. Perchlorate will be added as a potential contaminant in groundwater.

10. Section 2.3, para 4: "Today, Building 55 sits on the approximate area of the former range. Approximately 0.5 acres of the former range SDZ area are paved over for the parking lot and storage area associated with Building 55."

Building 55 and the associated parking lot appear to sit on the approximate area of the compass rose and part of the range floor; however the former location of the backstop for the range is not currently covered. Please restate.

Concur. The text will be revised as suggested.

11. Section 2.4, para 2: "The former Skeet Range was used for the training of the military personnel during ...defined the SDZ of a skeet range as a 900-foot radius from the shooting field giving it an area of 58 acres."

While accurate it does not take into account that the configuration of the skeet range changed, therefore

the acreage for the skeet range using a 900-foot radius is 78 acres according the Preliminary Assessment (Feb 2006). Please revise.

Concur. The text will be revised as suggested.

12. Section 2.6: It is unfortunate that there is no documentation regarding who provided the information that the Old Quarry was used for ordnance disposal. MEDEP called the consulting firm that developed the Remedial Investigation report and the ultimately called the consultant to whom the comment was made. His recollection was that it was by an unknown person during a tour of the Base; however it was not rebutted by the Navy during their review of the original Remedial Investigation. Therefore it cannot be discounted without further review or investigation.

In addition, there is clear documentation of disposal of munitions and ordnance at Site 12 from 1981 – 2004 and according to "Water Area Munitions Study, Naval Air Station, Brunswick (April 2005), the Navy used the Deep Sea Munitions Disposal Area off Cape Elizabeth, Maine, during the 1940s and early 1950s. That leaves the time period from between the early 1950s and the early 1980s unaccounted for as to where the Base disposed of it munitions and ordnance. It is possible that the land spreading in the 1990 covered the physical evidence.

MEDEP also reviewed historic aerial photos from the 1940-1980s. From that review it appears that Site 12 was not used until after 1970s, which is consistent with the information provided in Preliminary Assessment Addendum. In contrast, there was evidence of activity at the Old Quarry during that time period. The Quarry was also located near the bunkers on the west side of the runway. If possible, a more detailed examination of aerial photos needs to be performed. Also the Navy needs to perform interviews with former Navy personnel who were stationed at the Base in the 1950s through the 1970s. Unfortunately the interviews performed for this addendum include personnel who have only been at Brunswick Naval Air Station since 1990.

Without more convincing evidence that the Quarry was not used for destroying ordnance, it will have to be investigated.

An SI will be conducted at the Quarry. Text has been added to the Work Plan to discuss the SI activities at the Quarry.

13. Section 2.7, Topsham Annex, para 1: The majority (27 acres) of the SDZ extends off-base onto private property transferred to the MSAD No. 75.

MEDEP is concerned that the SDZ is primarily on private property, therefore the Navy must characterize and remediate, as necessary, the entire range.

The Navy will begin the process to secure right of entry for the private property so that field activities can be conducted, including soil and groundwater sampling.

14. Map 2-1: Please add the pre-1950 configuration for the Naval Air Station skeet range to the figure.

Concur. This information will be added to the map.

15. Map 2-4: Figure 2-4 needs to be revised to show surface danger zone for both configurations of the skeet range.

Concur. This information will be added to the map.

16. Section 3.3., SI Fieldwork, para 1: Please explain what instrument is being used for the instrument assisted visual field work if the magnetometer is being used for anomaly avoidance only as stated in Table 3-1. This also conflicts with is written in Appendix A, sections 4.3 and 4.6, which states "A detector-aided, nonintrusive site survey will be conducted over the … acres of the MRS in order to identify surface anomalies and MEC items." Please resolve these contradictions.

TtNUS is preparing a separate Work Plan and HASP to address the MEC (including geophysical surveys) portion of the investigation.

17. Table 3-1, Summary of MEC SI Activities, Munitions Bunker West Area, Activity: "Conduct a 100%, detector-aid, nonintrusive site survey…"

It is unclear what is meant by 100% site survey. Does it mean that the 100% of the 29 acres will be visually surveyed and transects will be completed with a magnetometer or other detector? Please clarify the proposed procedure and how it will differ from the hand-held detector used for anomaly avoidance.

TtNUS will conduct a surface sweep of the site and will flag MEC, if any. TtNUS is preparing a separate Work Plan and HASP to address the MEC (including geophysical surveys) portion of the investigation.

18. Table 3-1, Summary of MEC SI Activities, Site 12 EOD Area: Conduct a 100%, detector-aid, nonintrusive site survey…"

It is unclear what is meant by 100% (100% of the 112.7 acres?); please clarify. (As stated in MEDEP comments on the Preliminary Assessment-Addendum, Navy personnel stated that the demolition area may have moved around within Site 12 and is not just the currently bermed area.)

TtNUS will conduct a sweep of the site and will detonate any MEC encountered. TtNUS is preparing a separate Work Plan and HASP to address the MEC (including geophysical surveys) portion of the investigation.

19. Table 3-1, Summary of MEC SI Activities, Quarry: "No MEC activities."

MEDEP strongly disagrees. See comment 12 above.

An SI will be conducted at the Quarry, including an MEC investigation (including a geophysical survey). Any MEC encountered will be flagged. Text has been added to the Work Plan to discuss the

SI activities at the Quarry.

20. Section 3.3.1.2, Para. 1: Data Analysis:

The SAP and the QAPP are missing much of the information including DQOs, calibration requirements, and proposed data validation.

TtNUS will be producing the QAPP. The missing information will be added to the SAP.

21. Table 3-2:

a.) At this time MEDEP can not concur with the number of surface soil samples proposed in this table.

b.) Munitions Bunker Area West: 15 composite surface soil samples are proposed for a 29 acre site - roughly one composited sample per two acres. Please provide the rationale for this number of samples, the compositing scheme and what criteria will be used to determine where the samples will be taken.

c.) Machine Gun Boresight Range: 5 composite surface soil samples are proposed for a 0.03 acres site (firing line to berm). This may be adequate if it can be determined whether the berm was removed or just graded out in the vicinity, however prior screening prior with a metal detector or an XRF will be needed before the number of soil samples can be determined. No groundwater samples are proposed, yet in the Preliminary Assessment it was estimated that 1.6 million rounds (30 and 50 caliber bullets) were discharged into the berm. Groundwater sampling must be performed unless it can be determined that the berm was removed and that expended rounds were collected and disposed of offsite.

d.) NAS Skeet Range: 20 composite surface soil samples are proposed for a 73 acres site. Without prior screening to determine where to sample, this number is inadequate. The majority of the shot fallout zone appears to be over the streams and their floodplain wetland. Additional sediment sampling is necessary and due to the amount of run off and sediment from the storm drains it may be necessary to do some sediment sampling at depth.

e.) Site 12 EOD: 25 composite surface samples may be adequate if only the bermed area (1.5 acres) is being characterized but it is not adequate for the entire 112.7 acres. The 3 groundwater samples may be adequate, however the site is shallow to bedrock and direct push may be necessary to just determine the amount of soil coverage. Ultimately it may be necessary to install bedrock wells..

f.) Topsham Annex Skeet Range: 20 composite surface soil samples are proposed for a 29 acres site. If the preliminary screening with an XRF or metal detector is performed to direct the sampling locations to the maximum shot fallout zone this may be adequate. MEDEP agrees with EPA regarding the necessity to sample groundwater at this site.

g.) State criteria for soil, groundwater, and surface water, if lower than that of Region 9, must also be used for comparison regarding for whether to continue with a Remedial Investigation. The sediment criteria agreed upon for the other IR sites should also be considered where appropriate.

The number of soil samples was based on the premise that the SI is not intended to characterize the sites but to determine the presence or absence of MEC and MC. Sampling locations will be biased to source areas where maximum concentrations would be expected.

22. Section 3.3.2.2, Surface Water/Groundwater Sampling, para 2:

a.) The Navy is proposing two groundwater samples for the NAS Skeet Range to be taken from an existing site well. Please clarify if the groundwater samples are to be taken from the two existing wells rather than one well. Please provide the monitoring well number(s) and location(s). There are two existing wells (MW-NASB-077 and MW-NASB-078), which are located more in the vicinity of the firing point rather than the vicinity of the maximum shot fallout zone. Since there does not appear to be an existing monitoring well in the maximum shot fall out zone, please explain how the selected sampling point(s) will provide a representative groundwater sample for the skeet range.

The Navy is proposing 7 temporary wells at the NASB Skeet Range. Five of these wells will be in the post-1950 Skeet Range Area: 1 just downgradient of firing point, 1 in between firing point and target area, and 3 just downgradient of target area. Existing well MW-NASB-078 will be sampled for the firing point well. Two temporary wells will be installed in the pre-1950 Skeet Range Area within the likely shotfall zone.

b.) The Navy is proposing three direct push wells for Site 12 EOD Area. This site appears to be shallow to bedrock. Direct push may be necessary to determine soil coverage at the site, however to obtain a representative groundwater sample it may be necessary to install bedrock wells. The 1990 Remedial Investigation information for Site 12 is limited to three test pits. What is the rationale for the three direct push locations? Depending upon the bedrock topography, shallow groundwater flow may be to the east, south or southwest, while migration in the bedrock will depend upon the dominant fractures, foliations, and other features. Additional information is required to determine the best location for sampling groundwater at the site.

The Navy proposes collection of surface soil samples from the surface (0-12 inches). The subsurface soil sample locations and depths will be established following the geophysical survey in consultation with the Navy and regulatory agencies.

The Navy is proposing installation of 6 temporary wells. One well will be positioned just downgradient of the bermed area. The remaining 5 temporary well positions will be determined in the field based on geophysical survey anomaly identification. Bedrock wells will not be installed during the SI phase.

23. Section 3.3.2.4, Chemistry Analysis:

a.) The Navy must provide a list of the analytes of concern for each of the sites.

Concur. A list of the analytes of concern has been added to Section 2 for each site.

b.) Para. 6: Please define "a comprehensive data level of QA/QC", i.e., Tier III or other.

Tier III data validation will be specified. Data validation reports for all parameters will be generated according to the requirements described in Attachment B of the USEPA – New England Data Validation Functional Guidelines for Evaluating Environmental Analyses.

24. Section 6, para 1, bullet 4: As stated in section 1.1, the objective of this work plan is to determine the presence or absence of munitions and explosives of concern and munitions constituents, therefore, as designed, there are not enough sample points to refine the MRS boundaries. Please delete this bullet.

Concur. This change will be made to the text.

Appendix A:

25. Section 3, Project Scope and Objectives, Para. 3: "Metals concentrations will be compared to the Anticipated Typical Concentration Levels…"

Please provide a reference for these criteria. Metals concentrations must also be compared to EPA and MEDEP residential soil criteria.

The text has been revised as follows: "Metals concentrations will be compared to USEPA and MEDEP residential soil criteria." Reference to "Anticipated Typical Concentration Levels" has been removed from the text.

26. Section 3.1.1, Data Quality Objectives:

This section outlines the process in general, but does not indicate the rationale used, the conditions considered appropriate, or the tolerable limits for the project.

The text for this section will be revised. Please see Attachment 1 to this Response to Comments document for the detailed text.

27. Section 4.4, Machine Gun Boresight Range:

a.) The Navy proposes to base the soil samples based on visual observation. It would be better combine visual observation with pre-screening using a metal detector or an XRF. If the material from the berm was bulldozed then it is possible the MC could be a few inches beneath the surface. The best instrument for the situation should be used.

Sample locations will be biased toward where the berm was located and along the range floor. Samples will be collected at 0-1 foot, 1-3 foot, and 3-5 foot intervals since contamination could be present at depth when the berm was bulldozed. If warranted, an XRF will be utilized during the RI phase to further delineate the extent of lead presence.

b.) Para 2: This paragraph states the 5 composite soil samples will be analyzed for lead and propellants.

The propellants and the lead will not necessarily be co-located. By the time the bullet hit the berm the propellants on the bullet should be minimal. Therefore additional samples will be necessary ensure that the propellants were not discharged along the range floor.

Sample locations will be biased toward where the berm was located and along the range floor. Samples will be collected at 0-1 foot, 1-3 foot, and 3-5 foot intervals since contamination could be present at depth.

28. Section 4.5, Skeet Range: Please see comments 21.d and 22.a above.

The number of soil samples was based on the premise that the SI is not intended to characterize the sites but to determine the presence or absence of MEC and MC.

The Navy is proposing 7 temporary wells at the NASB Skeet Range. Five of these wells will be in the post-1950 Skeet Range Area: 1 just downgradient of firing point, 1 in between firing point and target area, and 3 just downgradient of target area. Existing well MW-NASB-078 will be sampled for the firing point well. Two temporary wells will be installed in the pre-1950 Skeet Range Area within the likely shotfall zone.

29. Section 4.9, Sampling:
Additional detail or SOPs are needed to evaluate the collection methods, at least for the groundwater sampling. For example what field parameters will be collected?

See Attachment 2 to this Response to Comments document for additional detail that will be included in the Work Plan.

30. Section 4.14, Field Equipment:
The table should include the detector or instrumentation to be utilized for the 100% coverage of appropriate areas for MEC.

TtNUS is preparing a separate Work Plan and HASP to address the geophysical portion of the MEC investigation.

Malcolm Pirnie Response to Comments on the
February 2007 Draft Final Site Investigation Work Plan
Naval Air Station Brunswick, Maine

Draft Final Site Investigation Work Plan (February 2007) – Comments from Brunswick Area Citizens for a Safe Environment (BACSE):

1. General Comment. BACSE concurs with comments submitted previously by the Maine Department of Environmental Protection (MEDEP) and the U.S. Environmental Protection Agency (EPA) regarding the two documents, and will not repeat the agencies' comments except where additional emphasis is desired. MEDEP's comment letters on the two documents are dated March 5 and March 16, 2007, and EPA's comment letters are dated February 28 and March 8, 2007.

Comment Number 6 in the MEDEP letter dated March 16, 2007 is indicative that a major revision of the SI Work Plan must be submitted to the stakeholders for review and comment before the final document can be prepared for release. BACSE anticipates reviewing this next draft of SI Work Plan. In the meantime, since comments regarding the PA Addendum have yet to be addressed to the satisfaction of the MEDEP, BACSE will focus this comment letter on the PA Addendum, and data gaps that need to be addressed in that document, as well as in the SI Work Plan.

A Revised Draft Final submittal is not anticipated. Comments will be responded to via this Response to Comments document. Malcolm Pirnie will submit the Final PA Addendum and Final SI Work Plan only after receiving concurrence from all stakeholders on the responses.

2. Munitions Storage and Handling. To date, five sites or areas of concern have been identified in two Preliminary Assessment reports under the Military Munitions Response Program (MMRP). The February 2006 Preliminary Assessment report covered the Munitions Bunker Area West, the former Machine Gun Bore Sight Range, and the former Skeet Range. The January 2007 Addendum addresses the Site 12 Explosives Ordnance Disposal (EOD) Area and the Quarry. Please clarify if MMRP will cover all munitions-related activities, including storage and handling that ever occurred at NAS Brunswick. If MMRP does not address storage and handling of munitions, what program(s) will? How will the possibility that nuclear weapons were handled or stored be addressed? When will potential environmental impacts associated with the Weapons Compound be addressed?

The MRP addresses MEC and MC at other than operational military ranges and other sites. MRP does not cover storage and handling of munitions. MEDEP anticipates that the former munitions bunkers west of the runway which were used to store high explosives and incendiary devices will be handled under the Resource Conservation and Recovery Act (RCRA) program as part of the base closure process. The Navy is not aware of any nuclear weapon storage or handling at NASB.

3. Bedrock Geology. The description of bedrock geology in Section 3.3 and elsewhere in the Preliminary Assessment Addendum is inadequate as a basis for designing the investigation in the Work Plan. An understanding of the geologic information currently available for NAS Brunswick (NASB) is crucial for developing a work plan that will adequately characterize the MMRP areas of interest, and properly evaluate potential contaminant migration pathways, especially in fractured bedrock. Two MMRP areas where the fractured bedrock pathways are of particular interest are Site 12, where there is only a thin layer of overburden covering rock, and the Quarry, where the rock exposures were presumably blasted in the past.

Existing sources of geologic information must be utilized to develop the Work Plan. The investigation of bedrock geology conducted under the Remedial Investigation (RI) [E.C. Jordan, August 1990, Draft Final *Remedial Investigation Report*; E.C. Jordan, August 1991, Draft Final *Supplemental Remedial Investigation Report*] was very limited, and focused on bedrock "highs" that were more resistant to glacial action and erosion. Presumably the resistant bedrock was less fractured than adjacent bedrock slopes or low areas. The 1991 RI report also describes the site-specific geology, based on a limited investigation, at Site 12. The variation of the overburden deposits over a relatively short horizontal distance should be factored into the MMRP investigation.

Additional information regarding fractured bedrock at NAS Brunswick is included in the March 2005 *Groundwater Monitoring Report, Site 11 – Bedrock Well*, prepared by ECC, and the January 2004 *Geophysical Investigation of Bedrock Fracture Zones at Site 11 and Areas Downgradient*, prepared by Hagar GeoScience for Gannett Fleming, Inc., for the U.S. Environmental Protection Agency. The 2005 report provides evidence of the fractured nature of bedrock beneath parts of NAS Brunswick, and that contamination has been found in bedrock on base. The January 2004 report, along with the August 14, 2003, Draft Final *Workplan for Geophysical Investigation of Bedrock Fracture Zones at Site 11 and Areas Downgradient*, prepared for the EPA, provide a framework and approach for investigating potential contaminant pathways in fractured bedrock at NASB. Of particular interest are the linear features identified on aerial photographs and their correlation with bedrock fracturing and other structures observed in the field.

BACSE is particularly concerned that there is adequate characterization and evaluation of the MMRP sites, especially their impact on the bedrock underlying the base, as all (or virtually all) of the residential wells on land immediately adjacent to NAS Brunswick are installed in bedrock.

Additional geologic information will be collected, reviewed, and incorporated into the Final PA Addendum and Work Plan. Additionally, the shallow/overburden groundwater will be sampled during the SI. If the results of the SI indicate that additional characterization is needed, this will be addressed during the RI phase.

4. Sand and Gravel Aquifer. The description of the geology of the area around NASB in Section 3.3 of the Preliminary Assessment Addendum makes no mention of the sand and gravel aquifer that extends across the northern half of the base property. BACSE feels this is a major oversight that must be corrected, and addressed in the Work Plan and any future MMRP work. This aquifer is the location of the municipal water supply wells for the Town of Brunswick. The aquifer is shown on a map published by the Maine Geological Survey [C.D. Neil, et al, 1999, *Significant Sand and Gravel Aquifers, Brunswick Quadrangle, Maine*: Maine Geological Survey, Open-File No. 99-18.]

Furthermore, the database to support the statement on page 3-3 of the Addendum, that "*the unit found at NASB is largely sand that may be underlain by silt and clay*", should be cited. The number and location of monitoring wells on base are limited to previous site-specific investigations, primarily in the northeastern and central eastern portions of the base, where only two out of five MMRP areas of interest is located (see Figure 2.1-1). If there is additional information for other areas of the base, the sources must be cited. If there is not, the statement regarding base-wide overburden geology must be properly qualified.

Additional geologic information will be collected, reviewed, and incorporated into the Final PA Addendum and Work Plan The statement on page 3-3 of the PA Addendum will be supported or revised to reflect additional geologic information.

ATTACHMENT 1

DQO TEXT

3.1 QUALITY OBJECTIVES AND CRITERIA

The following sections outline the DQOs and project quality assurance objectives for the MEC visual survey and MC soil sampling at the six MRP-eligible sites at NASB.

3.1.1 Data Quality Objectives

The seven-step USEPA DQO process was used to develop the rationale for this field investigation. This process is further described in the DQO Process for Hazardous Waste Site Investigations (USEPA, 2000a) and Guidance for the DQOs Process (USEPA, 2000b).

The DQO process is a strategic planning approach to optimize data collection activities and define the criteria that the data collection design should satisfy. The results generate a scientific and resource-effective data-collection design. The DQOs selected are qualitative and quantitative statements that:

- clarify the study objectives
- define the appropriate types and amount of data to collect
- determine the appropriate conditions (e.g. location and time) for data collection
- specify the tolerable limits on decision errors

Using the DQO process to plan field activities assures that the type, quantity, and quality of data used in decision making is appropriate for its intended use. The DQO process comprises the following seven steps:

- state the problem
- identify the decisions
- identify the inputs to the decision
- define the boundaries of the study
- develop the decision rules
- specify the tolerable limits on decision errors
- optimize the design for obtaining data

These steps are further described in the following subsections.

3.1.1.1 State the Problem

As indicated in Section 2.0 of the SI Work Plan, there are six MRP-eligible sites to be addressed as part of this SI. The Navy and members of the planning team (see Section 3.1.1.2 below) want to determine if a no further action recommendation is appropriate for these sites or if a recommendation to proceed to a Remedial Investigation (RI) is warranted.

Based on historical records and previous visual inspections summarized in the Final PAs (Malcolm Pirnie, 2005, 2006, and 2007) the following is known:

- Former Munitions Bunker Area West Area
  - Munitions related security training was conducted at this site between 1980 and 2000.

- No MEC were observed during a site survey conducted by Malcolm Pirnie in January 2003.
- Potential for MEC is considered extremely low.
- Potential exists for MC in the surface soil.

- Machine Gun Boresight Range
  - The former range was used during the 1950s to align and test fire aircraft mounted guns.
  - The former range is not suspected to contain MEC.
  - Potential exists for MC in the surface soil.and potentially in the shallow subsurface soil, considering that the berm has been disturbed

- Skeet Range
  - The former range was used during the 1950s to train military personnel.
  - The former range is not suspected to contain MEC.
  - Potential exists for MC in the surface soil, sediment, and surface water.

- Site 12 EOD Area
  - The Site 12 EOD Area was used from 1981 through June 1, 2004 for disposal of small quantities of ordnance, pyrotechnics, privately manufactured explosive devices, and war souvenirs.
  - Historical and visual evidence suggest the MEC are present at the site.
  - Potential exists for MC associated with the MEC to be present at the site in the surface soil, sediment, and surface water.

- Quarry
  - The Quarry was used as a rock quarry in the 1940s and 1950s. Land spreading occurred at the Quarry from 1992 through 1995.
  - There is a report that the Quarry may have been used for past EOD activities.
  - Potential exists for MEC to be present at the Quarry.
  - Potential exists for MC in the surface soil, sediment, and surface water.

- Topsham Annex Skeet Range
  - The former range was used by the U.S. Air Force for training prior to the 1970s.
  - The former range is not suspected to contain MEC.
  - Potential exists for MC in the surface soil, sediment, and surface water.

Although groundwater contamination is not expected at any of the sites, with the possible exception of Site 12, it is important to verify this assumption.

3.1.1.2 Identify the Decisions

An instrument-assisted visual survey will be used to determine the answer to the following question:

- Is a no further action (NFA) recommendation appropriate for MEC at the Former Munitions Bunker West Area, the Site 12 EOD Area, and the Quarry where the potential for MEC is very low or uncertain?
- Is MEC present at Site 12 as the potential suggests and, if so, can it be addressed via a sweep and blast in place and, if so, is an NFA recommendation appropriate?

NFA for MEC will be recommended for these sites if no visual observations of MEC, items indicating MEC (e.g., MEC, target areas, patterned MEC debris, source areas) or MC sample results (e.g., sample concentration patterns) indicating surface MEC are present. Additional investigation with respect to MEC will be recommended if site investigation data for MEC or for MC indicate that surface or potential subsurface MEC may be present that pose an unacceptable risk to human or ecological receptors. An Immediate Response Action will be recommended if MEC is encountered that poses an immediate risk to human or ecological receptors. Considering that MEC is suspected at Site 12, provisions are included to address MEC during the SI.

Soil sampling and subsequent analysis will be used to help determine the answer to the following question:

- Is an NFA recommendation appropriate for MC at the six sites or is it warranted to proceed to an RI?

NFA for MC will be recommended for these sites if analytical results are below project action levels (see Section 3). Additional investigation for MC will be recommended if MC analytical results are greater than project action levels and are determined to represent an unacceptable risk to human or ecological receptors. An immediate remedial action will be recommended if analytical results indicate an immediate threat to human health or the environment.

3.1.1.3 Identify the Inputs to the Decision

The following inputs to the decision for the investigation at the six sites were identified:

Regarding surface MEC:

- Presence or absence of historical MEC use, such as observations of MEC or MEC related items (significant debris, munitions source areas, target areas).
- The spatial boundary of MEC, if MEC is detected.

Regarding MC:

- Presence or absence of MC
- If present, what are the concentration values for MC detected?
- The spatial boundary of MC, if MC is detected
- If MC is present in soil, has contamination migrated to surface water, sediment, or groundwater?

3.1.1.4 Define the Boundaries of the Study

3.1.2.4.1 Former Munitions Bunker Area West Area

The Former Munitions Bunker Area West Area is roughly bound by undeveloped land to the north and south, Outboard Runway to the east, and Perimeter Road to the west.

The vertical boundary for MEC is defined as the surface. The vertical boundary for MC is anticipated at 0 to 6 inches below ground surface (bgs).

3.1.2.4.2 *Machine Gun Boresight Range*

The Machine Gun Boresight Range is roughly bound by Building 52, Building 55, and a containment pond to the north, Building 50 to the south, open field and forested area to the east, and a parking lot and Orion Street to the west.

The vertical boundary for MC is 2 feet below ground surface (bgs).

3.1.2.4.3 *Skeet Range*

The Skeet Range is roughly bound by Building 211 to the north, forested area to the south and east, and Orion Street to the west.

The vertical boundary for MC is 0 to 12 inches below ground surface (bgs).

3.1.2.4.4 *Site 12 EOD Area*

The Site 12 EOD Area is roughly bound by undeveloped land to the north, undeveloped land and Buttermilk Mountain to the south, an unnamed stream and Maine State Route 24 to the east, and an access road and undeveloped land to the west.

The vertical boundary for MEC and MC is 3 to 4 feet below ground surface (bgs).

3.1.2.4.5 *Quarry*

The Quarry is roughly bound by a radar tower to the north, an access road and undeveloped land to the south, a runway to the east, and Maine State Route 123 (Harpswell Road) to the west.

The vertical boundary for MEC is defined as the surface. The vertical boundary for MC is 4 feet below ground surface (bgs).

3.1.2.4.6 *Topsham Annex Skeet Range*

The Topsham Annex Skeet Range is roughly bound by undeveloped land to the north, recreational fields and undeveloped land to the south, undeveloped land and residential property to the east, and residential property to the west.

The vertical boundary for MC is 0 to 12 inches below ground surface (bgs).

3.1.1.5 *Develop the Decision Rules*

The objective of this investigation is to evaluate the potential presence of MEC on the surface and the potential presence of MC on the surface and in the subsurface of the sites.

To support this objective, the following decision factors were developed:

Regarding MEC (Former Munitions Bunker Area West, Site 12, and Quarry):

- NFA for MEC at the surface will be recommended if no visual observations of MEC, items indicating MEC (e.g., MEC, target areas, patterned MEC debris, source areas) or MC sample results (e.g., sample concentration patterns) indicate MEC are present.
- NFA for MEC subsurface soil for the Munitions Bunker West and the Quarry, where the presence of MEC is very low or uncertain, will be recommended if no anomalies are encountered during the geophysical survey and MC sample results do not indicate

that MEC was present historically. For Site 12, where MEC is expected, an NFA recommendation is possible if any MEC encountered is addressed via a blast in place and MC sample results are uncontaminated.

- Further action to address MEC will be recommended if MEC items (e.g., MEC, target areas, patterned MEC debris, source areas, etc.) or MC sample results indicate MEC may be present.
    - Surface MEC: Except for Site 12, if MEC is encountered at the surface, locations will be flagged and recommendations will be made for an interim action for a blast in place on an expedited basis. For Site 12, MEC encountered will be addressed via a blast in place. Details will be included in the MEC Work Plan by TtNUS.
    - Subsurface MEC: Except for Site 12, if anomalies are encountered during the geophysical survey, these locations will be flagged and recommendations will be made for an interim action to conduct an intrusive subsurface investigation on an expedited basis. For Site 12, where MEC is expected, anomalies (up to 4 foot depth) will be investigated and if MEC is encountered it will be addressed via a blast in place. Details will be included in the MEC Work Plan by TtNUS.

Regarding MC (all sites):

- NFA for MC will be recommended for the site if MC is not present greater than project action levels (see Section 3).
- Additional investigation for MC will be recommended if MC analytical results are greater than project action levels and/or are determined to represent an unacceptable risk to human or ecological receptors.
- An interim action on an expedited basis will be required if MC concentrations indicate an immediate threat to human health or the environment.

In addition to the decision factors, the CSMs from the Final PAs (Malcolm Pirnie, 2005, 2006, and 2007) will be refined based on the visual survey and site investigation findings. The CSMs will present hypotheses regarding MEC and MC presence, potential routes of migration, and their potential impact on sensitive receptors. The CSM will provide a tool to evaluate and refine decisions regarding the site.

3.1.1.6 Specify Tolerable Limits on Decision Errors

The following major limits on decision errors were identified for the SI field activities:

Regarding MEC site survey:

- Decision errors related to equipment malfunction will be mitigated with the implementation of daily QA/QC procedures. Malfunctioning equipment will be repaired or replaced. Equipment includes the hand-held magnetometer (e.g. Schonstedt), Geonics EM61, and the Global Positioning System (GPS) receiving unit (e.g. Trimble).

- Decision errors related to location error will be limited by post-processing the GPS data and comparing the mapped locations to those noted in the field forms and log.

Regarding MC soil sampling:

- The two decision errors for this project are: deciding that the soil is MC impacted when it is really not (Type I error) and deciding that the soil is not MC impacted when it really is (Type II error). The consequence of a Type I decision error will be unnecessarily incurred project costs associated with a remediation action. The consequence for a Type II decision error will be continued liability and potential risk to human health and the environment. Precision and accuracy requirements for the data are presented in the QAPP. These requirements will be adequate for minimizing project decision errors.

3.1.1.7 Optimize the Design for Obtaining Data

The overall goal of the DQO process is to design a SAP that will meet the needs of the data user (i.e., the decision-maker).

Activities associated with supporting this objective include:

- Selection of the most resource-effective sampling design that satisfies the DQOs.
- Generation of sampling and analysis design alternatives based upon cost consideration, time constraints, sample selection constraints, access limitations due to safety, and other considerations.

TtNUS is preparing a separate Work Plan and HASP to address the MEC portion of the investigation, including geophysical surveys. For the MC investigation, a TtNUS UXO specialist will serve as an escort for avoidance purposes during sample collection.

ATTACHMENT 2

SOP TEXT

SOIL AND GROUNDWATER INVESTIGATION EQUIPMENT AND PROCEDURES

Soil borings and temporary wells will be completed by direct-push methods. A Geoprobe® or other similar direct-push technology (DPT) equipment will be used to collect subsurface soil samples and to install temporary groundwater monitoring wells. Hand augering will also be employed for surface and shallow subsurface soil sampling, as warranted by site conditions.

A summary list of standard operating procedures (SOPs) follows:

S1	HS-1.0 UTILITY LOCATING AND EXCAVATION CLEARANCE
S2	SA-2.5 DIRECT PUSH TECHNOLOGY
S3	SA-1.3 SOIL SAMPLING
S4	GH-1.5 BOREHOLE AND SAMPLE LOGGING
S5	SA-1-1 GROUNDWATER SAMPLE ACQUISITION AND ON-SITE WATER-QUALITY TESTING
S6	GH-1.2 EVALUATION OF EXISTING MONITORING WELLS AND WATER-LEVEL MEASUREMENTS
S7	GH-2.9 WELL ABANDONMENT
S8	SA-7.1 DECONTAMINATION OF FIELD EQUIPMENT AND WASTE HANDLING
S9	SA-6.3 FIELD DOCUMENTATION
S10	USEPA REGION 1 LOW-FLOW PURGING AND SAMPLING METHODS
S11	USEPA REGION 1 DRAFT CALIBRATION OF FIELD INSTRUMENTS
S12	SA-1.2 SURFACE WATER AND SEDIMENT SAMPLING
S13	GH-2.8 GROUNDWATER MONITORING WELL INSTALLATION

SITE UTILITY CLEARANCE AND DIGGING PERMIT

Before any intrusive drilling or subsurface sampling activities commence at the site, utility maps of the facility will be obtained from the Navy and thoroughly reviewed. A utility surveyor will locate and map the utilities in the field. Ground-penetrating radar (GPR) and/or other geophysical instruments will then be used to survey the area to more accurately locate the buried utility lines. No intrusive activities (i.e., drilling or hand augering) will occur until the proposed areas intended for drilling have been completely cleared in accordance with TtNUS SOP S1 (HS-1.0).

Direct-Push Methods for Surface and Subsurface Soil

The procedure for soil sampling using DPT is included in TtNUS SOP S2 (SA-2.5). Hand-augering will be employed where locations are not accessible for a drill rig and/or where only shallow subsurface samples are planned.

A new acetate liner will be advanced at each boring location using a 4 foot Macro-Core® or similar sampler and in accordance with procedures outlined in TtNUS SOP S3 (SA-1.3). The soil core will be visually inspected and logged by the field geologist, noting soil texture, grain size (sand, silt or clay), color (and any unusual discoloration), qualitative moisture content, and classification according to the Unified Soil Classification System (USCS). The field geologist will identify the soil depositional environment. Soil samples will be collected and logged in accordance with procedures outlined in TtNUS SOP S3 (SA-1.3) and S4 (GH-1.5).

All borings not converted into temporary well points will be backfilled with bentonite chips and patched with cement, cold-patch asphalt, or soil at grade to match the surrounding area. Care will be taken to return each location to its original condition after sampling. Each boring location will be revisited after backfilling is completed to re-apply soil, cement grout, or cold-patch, if necessary. After a boring has been sampled and backfilled, it will be identified by a wooden stake, driven into the center of boring. If two or more drive attempts were made to collect adequate sample volume, the stake will be driven in the center of the area represented by these borings. The locations and distances between the borings will be clearly described in the sample collection logs. The stake will have both brightly colored flagging attached to increase visibility, and be labeled using a waterproof marker with the soil boring number.

All direct-push equipment will be steam cleaned prior to the start of the field investigation, decontaminated between each borehole to prevent cross-borehole contamination and decontaminated prior to leaving the site.

Temporary Monitoring Point Installation

Borings for installation of temporary monitoring wells will be advanced using DPT and in accordance with TtNUS SOP S2 (SA-2.5) and the following described methodology. A GeoProbe® or similar DPT will be used to advance 2-inch outside diameter Macro-Core® or similar sampler of 4-foot length to several feet below the local water table. If cobbles or thick gravel layers are encountered and prohibit advancement of the sampler using DPT or if open boreholes can not be maintained that allow installation of temporary monitoring wells, small diameter hollow-stem augers (2.25-inch inside diameter minimum) will be used to advance the boring to the required depth.

The Macro-Core® sampler will be removed from the borehole, and a temporary polyvinyl chloride (PVC) well will be installed. A Geoprobe® pre-pack PVC well screen will have an inside diameter of approximately 0.75-inch, (outside diameter of approximately 1.4 inches), a slot size of approximately 0.01 inches (slot size openings), and a pre-pack well screen length of approximately 10 feet. The well screen will be installed in the upper surficial aquifer and will intersect the estimated depth of the groundwater

table, based on the presence of water observed during soil lithology sampling. The well screen will be capped at the bottom. PVC riser pipe (Schedule 40) will be attached to the screen to extend the well above grade. The screen and riser pipe will be flush jointed. Sand filter pack will be placed above the pre-pack well screen to a height of approximately 1 foot, followed by a bentonite seal (1 foot minimum thickness) installed above the sand pack. A pre-manufactured sand pack is recommended because the boring size is expected to be too small for the annulus between the well screen and the boring to allow free flow of sand. Only 100 percent, certified pure, sodium bentonite will be used for well construction. The remaining annulus above the bentonite seal will be backfilled with bentonite pellets or chips.

Each temporary well will be developed by pumping and surging to remove sediments from inside the casing and within the sand pack which surrounds the well screen. Each new well will be developed after waiting approximately 12 hours (minimum) after installation is complete. Each of the wells will be developed for approximately one-half hour.

The water levels in the temporary wells will be allowed to stabilize for 12 hours or more after development, depending on the permeability of the formation, before sampling. The temporary wells will be capped when left unattended to minimize the introduction of particles or contaminants into the well from the surface.

After TtNUS evaluation of water level data and analytical results and subsequent concurrence with the Navy and regulatory agencies, some of the temporary wells may be abandoned. For these wells to be abandoned, the riser and screen will be grouted. The grout will be emplaced from the bottom of the hole to the surface and consist of a cement-bentonite slurry. The surface at each temporary well location will be restored to its original condition (vegetated, asphalt or concrete).

Boring logs will be prepared for each temporary well. The boring logs will record installation methods, depths of installation, abandonment details, and other pertinent information. A map of the temporary monitoring well locations and appropriate data from the boring logs will also be recorded into the field logbook notes.

Surface Water and Sediment Sampling

Surface water and sediment samples will be co-located and collected in accordance with TtNUS SOP S12 (SA-1.2). Sediment samples will be collected from 0 to 1 foot depth.

GROUNDWATER PURGING, SAMPLING, WATER-LEVELS, AND ABANDONMENT OR COMPLETION

Groundwater Purging

Low-flow purging and sampling is being implemented because this method will provide the least disturbance to the surrounding formation (i.e., less turbulence in sampling and hence less turbidity) allowing for a more representative groundwater sample to be collected. Low-flow purging and sampling activities will not begin until at least 12 hours after the temporary monitoring wells have been developed. An adjustable-rate peristaltic pump will be used for purging and sampling purposes. New silicone pump tubing and polyethylene tubing will be attached to the pump and inserted near the mid point of the well screen before each well is purged so as not to disturb any sediment that may be located near the bottom of the well. Low-flow purging and sampling techniques will be utilized in accordance with TtNUS SOP S5 (SA-1.1) and general accordance with S10 (USEPA Region I methods).

During well purging, the water level in each well will be monitored every 5 to 10 minutes as purging occurs. A drawdown of less than 0.3 feet will be targeted. Initially, the pumping rate will be set at approximately 0.3 liters per minute (L/min) or lower if needed. The pumping rate will be reduced if turbidity is greater than 10 Nephelometric Turbidity Units (NTUs) after all other field parameters have stabilized. The measured pumping rates and groundwater level data will be recorded on the well purging log sheets.

Water quality parameters [pH, specific conductance, temperature, oxidation/reduction potential (ORP), salinity, and dissolved oxygen] will be measured during purging using a YSI Model 6820 water quality meter (or equivalent) and flow-through cell. Turbidity will be measured using a Lamotte 2020 (or equivalent) and the turbidity measurement will be performed from a sample collected from a "T" connector before the flow-through cell. Water quality measurements will be recorded on the well purging log sheet every 5 to 10 minutes.

Calibration and standards checks will be conducted on the water quality meter in accordance with the manufacturer's recommendations and SOP S11 (USEPA Region I methods). The flow-through cell will be decontaminated before initiation of well purging at each well. Precautions will be taken to prevent air entrapment and/or air leaks in the purging system so that potential problems with stabilizing dissolved oxygen are minimized. Precautions will include: (1) taking care to fill the entire flow-through cell with water, while minimizing air entrapment, before initiating purging and (2) maintaining a full cell of water by pinching the discharge line shut and elevating the discharge at the end of the tubing from the pump, above the cell.

Purging will continue until all of the parameters have stabilized to the criteria listed below and the minimum purge volume (one well volume) has been removed from a well. Stabilization criteria for the water quality parameters are as follows:

- pH ± 0.1 standard units
- turbidity ± 10 percent for values greater than 5 NTU
- specific conductance ± 3 percent
- temperature ± 3 percent
- ORP ± 10 millivolts
- dissolved oxygen ± 10 percent

If the parameters have not stabilized within 2 hours, then sampling will be initiated. The final values of water quality parameters measured just before sampling commences will be recorded on the groundwater sample collection log sheet.

Temporary Monitoring Well Sampling

After purging is completed, the flow-through cell will be disconnected from the pump discharge tube and samples will be collected directly from the discharge tube of the pump. Sample containers will be filled (following the method detailed in TtNUS SOP S2 (SA-2.5)) by allowing the groundwater to flow gently down the inside of the container with minimal turbulence. Except for perchlorate samples, the bottles should be filled completely with no head space remaining in the bottle then capped. All sample bottles will have the appropriate type and amount of preservative already added to the containers, prior to arrival at the site (i.e., preservatives are added to the bottles by the analytical laboratory before shipping to the site). TtNUS SOPs S5 (SA-1.1) and S10 (USEPA Region I methods) will be followed for purging and sampling activities.

Water-Level Measurements

One round of water levels will be manually measured in all temporary monitoring wells for a given site. These measurements will be made using an electric water-level indicator capable of 0.01-foot accuracy in accordance with TtNUS SOP S6 (GH-1.2). Water levels will be recorded on Groundwater-Level Measurement log sheets.

Temporary Well Abandonment

Select temporary wells will be properly abandoned after evaluation of water-level data and analytical results in concurrence with the Navy and regulatory agencies. Abandonment will comply with TtNUS SOP S7 (GH-2.9). The temporary wells will be tremie backfilled with a cement/bentonite grout for purposes of well abandonment. Each backfilled boring will be checked the following day for subsidence of the grout and finished smooth to existing grade. The grout sealant will consist of sodium bentonite.

The well riser and screen will be pulled from the borehole, and the borehole will be backfilled with bentonite/cement grout from the bottom of the borehole to the ground surface. If possible, the bottom of the well will be knocked out and used as a tremie pipe for emplacement of the grout to avoid collapse of the borehole. The well riser and screen will be steam cleaned at the decontamination pad before disposal.

Temporary Well Completion to Permanent Well

Select temporary wells will be converted to permanent monitoring wells after evaluation of water-level data and analytical results in concurrence with the Navy and regulatory agencies. Well completion will be conducted in accordance with TtNUS SOP S13 (GH-2.8), and will include a protective casing with lock, a concrete pad, and bollards where stick-up wells are completed in the vicinity of roadways.

SAMPLE HANDLING AND MANAGEMENT

The following sections outline the procedures that will be used by field personnel to document project activities and sample collection procedures. Detailed and accurate documentation is necessary in order to ensure data integrity.

Sample Custody

Documentation of field observations will be recorded in a field logbook and/or field log sheets including sample collection logs, boring logs, and monitoring well construction logs. Field logbooks utilized on this project will consist of a bound, water-resistant logbook. All pages of the logbook will be numbered sequentially and observations will be recorded with indelible ink. Field logbooks will be maintained according to SOP S9 (SA-6.3). Field sample log sheets will be used to document sample collection details, and other observations and activities will be recorded in the field logbook. Instrument calibration logs will be used to record the daily instrument calibration.

For sampling and field activities, the following types of information will be recorded as appropriate:

- Site name and location
- Date and time of logbook entries
- Personnel and their affiliations
- Weather conditions
- Activities involved with the sampling
- Subcontractor activity summary
- Site observations including site entry and exit times
- Site sketches made onsite
- Visitor names, affiliations, arrival and departure times
- Health & Safety issues including PPE

The following sections outline the information that will be documented in the field according to the media to be sampled and the activities to be performed.

Sample collection information for all media (soil, surface water, sediment, and groundwater) will be recorded on TtNUS sampling logs. The field logbooks and sample log sheets will remain on-site for the duration of the investigation. After the investigation, the field sampling log sheets will be organized by date and media and placed in the project file. The field logbooks for this project will be used only for this site, and will also be categorized and maintained in the project file 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 given titles based on date and activity.

Sample custody procedures are designed to provide documentation of preparation, handling, storage, and shipping of all samples collected. Field chain-of-custody procedures are described in TtNUS SOP S9 (SA-6.3). Laboratory chain-of-custody procedures are described in the laboratory SOPs.

Integrity of the samples collected during the site investigation will be the responsibility of identified persons from the time the samples are collected until incorporated into the final report. Stringent chain-of-custody procedures will be followed to document sample possession.

Field Custody

The TtNUS field operations leader (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.

Sample logs or other records will be signed and dated by the persons making the entries.

Chain-of-custody forms will be completed to the fullest extent possible before sample shipment. They will include the following information: project name, sample identification, date and time collected, analyses to be conducted, matrix, type of sample, grab or composite designation, preservative, and name of sampler. Sample salinity will be noted on chain-of-custody forms to aid the analytical laboratory in establishing dilution requirements for metals analyses where salinity could present an interference concern. Sample depths will be indicated on the chain-of-custody form also.

These forms will be filled out in a legible manner, using waterproof ink, and will be signed by the sampler. Similar information will be provided on the sample label, which will be securely attached to the sample bottle. The label will also include the general analyses to be conducted. In addition, sampling forms will be used to document collection, filtration, and preparation procedures. Copies of all forms used during field activities are provided in the SOPs.

Transfer of Custody

The following procedures will be used when transferring custody of samples:

Sample coolers will be custody-sealed for security according SOP S9 (SA-6.3) and accompanied by a chain-of-custody form. When transferring samples, the individuals relinquishing and receiving them will sign, date, and note the time on the chain-of-custody form. This record documents the sample custody transfer from the sampler to the laboratory, often through another person or agency (common carrier). Upon arrival at the laboratory, internal sample custody procedures will be followed as defined in the laboratory SOPs.

Prior to shipment to the laboratory for analysis, samples will be properly packaged. Individual custody records will accompany each shipment. Shipping containers will then be sealed for shipment to the laboratory. The methods of shipment, courier name, and other pertinent information will be entered in the "remarks" section of the custody record.

All shipments will be accompanied by the chain-of-custody form identifying the contents. The original record will accompany the shipment and a copy will be retained by the field sampler.

Proper documentation will be maintained (i.e. airbills) for shipments by common carrier.

Sample Shipment Procedures

The following procedures will be followed when shipping samples for laboratory analysis:

Samples requiring cooling to 4°C will be promptly chilled with ice or Blue Ice and will be packaged in an insulated cooler for transport to the laboratory. A temperature blank will be included in each cooler to be used as a temperature indicator. Each temperature blank will be clearly identified by the field sampling team. Ice will be sealed in containers to prevent leakage of water. Samples will not be frozen.

Only shipping containers that meet all applicable state and federal standards for safe shipment will be used.

The field chain-of-custody form will be placed inside the shipping container in a sealed, plastic envelope. Shipping containers will be sealed with nylon strapping tape, and custody seals will be signed, dated, and affixed, in a manner that will allow the receiver to quickly identify any tampering that may have occurred during transport to the laboratory.

Samples to be delivered to the laboratory will be made by a public courier. After samples have been collected, they will be sent to the laboratory within 72 hours. Under no circumstances will sample holding times be exceeded.

Laboratory Sample Custody

To ensure the integrity of a sample from collection through analysis, it is necessary to have an accurate, written record that traces the possession and handling of the sample. This documentation is referred to as the chain-of-custody form.

A sample is under custody if:

- The sample is in the physical possession of an authorized person.
- The sample is in view of an authorized person after being in his/her possession.
- The sample is placed in a secure area by an authorized person after being in his/her possession.
- The sample is in a secure area, restricted to authorized personnel only.

When samples are received, the chain-of-custody is signed and dated to acknowledge sample receipt. The sample custodian must examine the shipping containers and verify that the correct number of containers was received. The shipping containers are then opened and the enclosed sample paperwork is removed. Samples are removed from the shipping containers and the bottle condition, temperature blank, and sample salinity must be noted. The information on the chain-of-custody, the airbill, the containers, and the laboratory request is reviewed to note any discrepancies.

The laboratory will be required to fax the chain-of-custody forms and sample log-in information to the TtNUS project manager after every shipment.

All samples received by the laboratory must be stored at 4°C until analysis. Laboratory holding times are specified by the contract and presented in the QAPP.

If the equipment is new, the initial cleaning will consist only of a soapy water wash followed by a tap water and distilled water rinse. Disposable sampling materials (e.g., polyethylene or teflon tubing for pumps) that are individually packaged from the factory will not require decontamination before sampling.

To ensure that sampling equipment has been decontaminated properly, equipment rinsate blanks will be collected and analyzed.

EQUIPMENT DECONTAMINATION

TtNUS will obtain precleaned sample containers for laboratory analyses. These containers will meet the requirements of the USEPA Specification and Guidance for Contaminant-Free Sample Containers (OSWER Directive No. 9240.0-05A).

Equipment decontamination will follow the procedures described in the TtNUS SOP S8 (SA-7.1). Nondisposable equipment items that come in contact with the media to be sampled and will require decontamination include:

- stainless steel mixing bowls and spoons (in contact with soils)
- DPT tooling (in contact with soils)
- water level indicators (in contact with groundwater)

If the equipment is new, the initial cleaning will consist only of a soapy water wash followed by a tap water and distilled water rinse. Disposable sampling materials (e.g., tubing for peristaltic pumps) that are individually packaged from the factory will not require decontamination before sampling.

To ensure that sampling equipment has been decontaminated properly, equipment rinsate blanks will be collected and analyzed.

IDW MANAGEMENT

Five types of IDW will be generated during this investigation that could be potentially contaminated: soil cuttings, well development and purge water, DPT decontamination wastewater, sampling equipment

decontamination wastewater, and personnel protective equipment and clothing (PPE). Based on the historical site activities and types of contaminants present, none of these IDW materials is expected to present a significant risk to human health or the environment if properly managed.

Soil cuttings and excess soil core material resulting from the DPT drilling activity will be placed in 55-gallon labeled, sealable steel drums. The soils from each site will be segregated, so that if the soil analyses show that soils from one or more of the sites are clean, these soil materials can later be retrieved and disposed of as clean soil. The drums of soil IDW will be labeled, sealed, and transported to a central location designated by the Navy. Proper disposal of these wastes will be performed by the Navy once the analytical results of the soil samples are received from the laboratory and reviewed.

Well development waters and purge waters will be pumped into 55-gallon sealable drums, which will be properly labeled and transported to a central location to be designated by the Navy. Proper disposal of these wastes will be performed by the Navy once the analytical results are received from the laboratory and reviewed.

FIELD ANALYTICAL METHOD REQUIREMENTS

Water Quality Meters

Field Analytical Method Requirements – Water Quality Meters

Field measurements of pH, dissolved oxygen, specific conductance, salinity, temperature, turbidity, and ORP will be performed during low-flow purging for well sampling and pH, specific conductance, temperature, turbidity will be performed during development as described in TtNUS SOP S5 (SA-1.1). These measurements will be made using a YSI Model 6820 (or equivalent) for all parameters except turbidity. A Lamotte 2020 (or equivalent) will be used for the turbidity measurement. Each of these measurements will be performed using an electrode or sensor designed specifically for the parameter being measured. The calibration and use of the meter will be performed per TtNUS SOP S5 (SA-1.1) and SOP S11 (USEPA Region I method).

Field Analytical Instrument Calibration – Water Quality Meters

The multiprobe water quality meters will be calibrated per TtNUS SOP S5 (SA-1.1) and SOP S11 (USEPA Region I method). The meter will be calibrated at the beginning of each field day. The calibration will be checked against standard solutions at the end of each field day or more frequently if fluctuations in readings appear to be abnormal. Records of instrument calibration will be maintained on a field instrument calibration log sheet, provided in SOP S5 (SA-1.1). The field geologist will maintain

instrument manuals on site. If the meter does not maintain an acceptable level of calibration during a single day's use, then the meter will be calibrated more frequently or it will be replaced.

FIELD EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION REQUIREMENTS

Equipment, instruments, gauges, and other items requiring preventive maintenance will be serviced, when appropriate, by the equipment supplier, the FOL (or designee), and/or drilling subcontractor in accordance with the manufacturer's recommendations. Manufacturer's procedures identify the schedule for servicing critical items in order to minimize the downtime of the measurement system. It will be the responsibility of the FOL to adhere to this maintenance schedule and to arrange any necessary and prompt service required. Service of the equipment, instruments, tools, gauges, etc. shall be performed to the extent possible by the FOL (or designee). If the service requires a more qualified person, then the supplier or the manufacturer will be contacted for assistance. Logs shall be established by the FOL (or designee) to record maintenance, service procedures, and schedules. Maintenance records will be documented and traceable to the specific equipment, instruments, and gauges.

It will be the responsibility of the FOL (or designee) to inspect all supplies to be used as part of the field program during mobilization and use. Supplies to be inspected include sampling equipment, field meters, and sample containers.

If the FOL encounters any problem with the supplies, he or she will inform the TtNUS project manager and the laboratory supplying the containers. The TtNUS project manager, in consultation with the Navy project manager and QA/QC officer, will instruct the FOL on any corrective actions that should be implemented.

SURVEYING

The vertical elevations and horizontal locations of all temporary monitoring wells, soil sampling locations, and important site features will be surveyed by a Maine-licensed surveyor at the completion of the field investigation. Existing benchmarks will be used. All horizontal locations will surveyed to the nearest 0.1 foot. All elevations will be measured to the nearest 0.01 foot. The surveyor will determine elevations for the ground surface at all locations. Additionally, for the temporary monitoring wells, the elevation of the reference point (notch at the top of the riser pipe) will be surveyed for each of the temporary monitoring wells. The TtNUS Field Operations Leader will provide the correct sample location name to the surveyor so that database nomenclature is consistent with location names in the QAPP and the database. Upon receiving the survey data from the surveyor, the TtNUS Project Manager will check the survey data for consistency with the location names in the QAPP and the database.

For remote soil boring locations and/or surface water/sediment locations where land surveying is more difficult, a GPS unit may be used to collect coordinate data. However, all temporary wells will need to be surveyed by a land surveyor because of the required accuracy.

FIELD DOCUMENTATION AND DOCUMENTATION RESPONSIBILITIES

It will be the responsibility of the FOL to secure all documents produced in the field (e.g., sampling logs, calibration forms) at the end of each work day. Copies of all forms used during field activities are included in the SOPs. Sample logs and chain-of-custody records will be included as an appendix to the report that will be prepared based on results of this investigation.

At the completion of field activities, the FOL will send Ms. Linda Klink (the TtNUS project manager) all field records, data, field notebooks, logbooks, chain-of-custody forms, sample log sheets, daily logs, etc. The project manager will ensure that these materials are entered into the TtNUS document control system in accordance with appropriate administrative guidelines.

Changes in project operating procedures may be necessary as a result of changed field conditions or unanticipated events. A summary of the sequence of events associated with field changes is as follows:

- The FOL will notify the TtNUS project manager and TtNUS Maine Certified Geologist (CG) of the need for the change.

- If necessary, the project manager will discuss the change with the pertinent individuals (e.g., Maine CG, Navy project manager, TtNUS QA Manager) and will provide a verbal approval or denial to the FOL for the proposed change. The USEPA and MEDEP will be consulted by the Navy of any major scope changes that may occur while fieldwork is ongoing. Communications and correspondences to the Restoration Advisory Board (RAB) will be handled through inclusion on the distribution list or written correspondence and updates at RAB meetings.

- The FOL will document the change on a Task Modification Request form and forward the form to the TtNUS project manager at the earliest convenient time.

- The TtNUS project manager will sign the form and distribute copies to the Navy project manager, TtNUS QA Manager, FOL, and project file.

- A copy of the completed Task Modification Request form will be attached to the field copy of the affected document.

The possession of all records will be documented; however, only the project FOL or designee may remove field data from the site for reduction and evaluation.

D-4 MARCH 2009 RESPONSE TO STAKEHOLDER FOLLOW-UP COMMENTS

Klink, Linda

From: Klink, Linda
Sent: Tuesday, March 17, 2009 10:53 AM
To: Daly.Mike@epamail.epa.gov; Sait, Claudia B
Cc: 'Bober, Todd A CIV NAVFAC MIDLANT'; 'Burgio, Paul CIV OASN (I&E) BRAC PMO NE'; Race, Charles; Orient, Jeff
Subject: Navy reponse to EPA/MEDEP comments on SI MC Work Plan, NASB
Attachments: MC SI WP RTC for MEDEP&EPA Review NASB.doc; Empirical LCS AccuracyPrecisionLimits-13022009.pdf

Mike and Claudia,

The Navy would like to initiate the field program for the three small arms ranges (Skeet Range, Topsham Skeet Range, and Machine Gun Boresight Range) in May 2009. The associated SI Work Plan, as it currently stands, is in the Revised Draft Final phase dated February 2008. The Work Plan has been on hold as a result of discussions with the Stakeholders regarding MC sampling methodologies for the MEC sites (Site 12 EOD, Quarry, and Former Munitions Bunker West). For safety reasons and to provide a better basis for establishing MC sampling locations, the Navy is planning to conduct MEC clearance before accomplishing the MC sampling at the MEC sites As a result, the subject Work Plan for all six sites is being revised to only include the small arms ranges. A work plan for MC sampling of the three MEC sites will be issued at a later date, following evaluation of findings from the MEC clearance effort.

The Navy requests that The EPA and Maine DEP review the attached comment response letter and provide your concurrence or let us know, informally, of any remaining issues by March 24, 2009 if possible. The Navy will then formalize this comment response letter and finalize the Work Plan. Note that although the Work Plan will be submitted as final, any significant concerns, can still be addressed via revision pages so long as those concerns are identified to the Navy prior to accomplishing the fieldwork targeted for May 2009. Should you have any comments or concerns please feel free to contact Todd Bober or Paul Burgio at any time.

Thanks!

RESPONSE TO USEPA COMMENTS DATED MARCH 27, 2008
REVISED DRAFT FINAL SITE INSPECTION WORK PLAN, MUNITIONS CONSTITUENTS AT SIX MUNITIONS RESPONSE SITES, DATED FEBRUARY 2008
NAVAL AIR STATION BRUNSWICK, MAINE

Additional Notes: For safety reasons and to provide a better basis for establishing MC sampling locations, the Navy has now decided to conduct MEC clearance before conducting the MC sampling at the MEC sites (Site 12 EOD, Quarry, and Former Munitions Bunker West). As a result, the subject Work Plan for all six sites has been revised to only include the remaining small arms ranges (Skeet Range, Topsham Skeet Range, and Machine Gun Boresight Range). This will allow field work at these small arms ranges to proceed in the Spring of 2009. A work plan for the three MEC sites will be issued at a later date, following evaluation of findings from the MEC clearance effort, and associated comments included herein will be addressed at that time.

Note that where the comment response provides revised text, text additions are shown in italics and deleted text is shown as strikethrough.

1. **Comment:** Work Plan Cover Sheet; A lead agency (Navy) signature block should also be included along with the approval signatures of MEDEP and EPA representatives. Also, the dates of the signatures should also be included. EPA suggests that TtNUS circulate this cover page to all signatories once the document is finalized. Copies of the completed cover sheet can then be distributed as the final replacement page.

   Response: Agree. As discussed during the Data Quality Objectives (DQO) meeting of February 2008 on a different topic (1,4-dioxane/bedrock), the UFP-QAPP approval page will include approval signatures for the EPA (Michael Daly) and Navy BRAC PMO NE (Todd Bober). The MEDEP (Claudia Sait) signature will be removed.

2. **Comment:** Sections 3.3.2.1 (Soil/Sediment Sampling) & 3.3.2.2 (Surface Water/Ground Water Sampling and Water-Level Measurements); Sampling locations up-gradient of the sites need to be included in the work plan and clearly identified in Figures 2-2, 2-3, 2-4, 2-5, and 2-6) where surface water, sediment, and ground water will be collected. If it is not possible to locate samples up-gradient of the sites, this should be clearly identified in the work plan. For the purposes of the ground water investigation portion of the work plan, all available ground water potentiometric surface maps should be included in the work plan to justify the monitoring well network spatial design for each of the sites. EPA understands that for some sites undergoing MEC investigations prior to the MC investigation, the results from that phase will be incorporated into the MC final sampling design. EPA's general comment on this issue is that the sampling plan should attempt to characterize the quality of these environmental media coming onto the study areas as they are currently defined. This will allow the Navy to make more informed conclusions as to whether potential releases from MEC/MC-related activities require further investigation.

   Response: The original plan was to rely on the results from the facility background study being conducted by Tetra Tech/Navy under a separate project rather than collecting samples as part of the MC SI effort. The facility background study schedule is on track to be accomplished Spring 2009. As part of this study, the Navy will include a few upgradient samples for the two skeet ranges and MGBR as necessary. Rationale for each media follows:

Soil: Surface soil background samples for TAL metals (and PAHs/SVOCs where applicable for a given site) will be collected at each of the three sites. Because explosives/propellants constituents should not be present in native soil, these analyses will not be included. Because the Skeet Range and Machine Gun Boresight Range are adjacent to each other, one surface soil sample will suffice for both and will be analyzed for TAL metals and PAHs. The location for this background sample was already included in the SI work Plan, Section 2.0 sampling location figure but will be relocated in response to MEDEP Comment #15. A background surface soil sample will also be collected for Topsham Skeet Range; the location for the sample (for metals and PAH analysis), which has been added to the sample location map, will be to the west of the skeet range, between the school to the south and the Laskey/Ofarell Street area to the north.

Surface Water/Sediment: The two skeet ranges have the surface water/sediment media present. The Skeet Range is pond-like where upgradient locations are not applicable. The Navy agrees to collect an upgradient surface water/sediment sample at the Topsham Skeet Range and analyze the samples for metals and PAHs. The upgradient location has been added to the sample location map.

Groundwater: Note that the Work Plan will be revised to indicate that temporary monitoring wells will be installed at the NASB Skeet Range and Topsham Skeet Range only as necessary depending on XRF results for lead or other considerations, as agreed upon during a conference call held among the Navy, EPA, and MEDEP on January 29, 2009.

In general, groundwater flow direction is not definitively known since the planned SI will be the first investigation. The plan, in general, is to encompass all four directions from a site source area, as well as the source area(s) itself to determine groundwater flow direction and upon evaluation of the resulting potentiometric map, the well serving as the upgradient well can be identified as such. The groundwater sampling locations are shown on the sampling location figures. Text will be added to presume the groundwater flow direction based on existing information in the area. The following additions will be made to the Appendix A SAP, Section 3.1.1.1 Supplemental Information, for each of the three sites, as follows:

> *Machine Gun Boresight Range*, new bullet: "....Numerous other wells and other historical samples are available (Appendix C-1). *Based on data from Site 9 located less than ¼ mile to the south, groundwater flow direction is presumed to be to the southeast.*" Also, the Site 9 interpreted groundwater surface contour map from September 2008 from the Monitoring Event 33 Report (ECC, December 2008) will be added to Appendix C-1.
>
> *Skeet Range*, 2nd bullet: "....Numerous other wells and other historical samples are available (Appendix C-2). *Based on adjacent Site 9 data, groundwater flow direction is presumed to be to the southeast.*" Also, the Site 9 interpreted groundwater surface contour map from September 2008 from the Monitoring Event 33 Report (ECC, December 2008) will be added to Appendix C-2.
>
> *Topsham Skeet Range*, 2nd bullet: "....Historical data is available from 2004 and 2006 investigations of the Skeet Range and nearby areas at Topsham Annex. *Of note, reference figures in Appendix C-3 associated with the 2004 Investigation Report (EA/ECC, February 2006) show inferred groundwater flow direction to the west based*

on regional groundwater flow. However, the subsequent 2006 Investigation Report (Tetra Tech, draft) text indicates that groundwater flow direction, determined from water levels during monitoring well construction and survey data, is to the southeast."

The changes above will affect sample quantity updates to the following sections:

- SI Work Plan: Section 3.3.2 and Tables 3-2 and 3-3
- Appendix A SAP: Table 3-1, Section 4.3, Tables 4-3, 4-4, and 4-6
- Appendix A SAP Attachment 1 QAPP, Attachment 1-A, Worksheet #20

3. **Comment:** Table 3-1, Pages 3-3 & 3-4; EPA suggests eliminating this table and incorporate the activities in this table into Table 3-2. This table creates yet more confusion as to what work is to be conducted under the MEC work plan (previously submitted under a separate cover) versus the work covered by this MC work plan.

   Response: The Navy agrees with eliminating Table 3-1, considering that the MEC sites have now been removed from this SI MC Work Plan. Subsequent tables in the Section will be renumbered.

4. **Comment:** Page 3-10, last¶; Synoptic water level measurements should include some subset of existing LTM program wells (i.e., Site 9) that would provide greater ground water potentiometric surface control.

   Response: Agree, if it is determined by the team that groundwater needs to be evaluated as part of this investigation. If that is the case, then the following Site 9 monitoring wells will be included in synoptic water level gauging: MW-NASB-072, -075, -076, -077 and -078. These wells are completed in the upper sand aquifer, consistent with the aquifer targeted for new well installations at the Skeet Range Site. These wells will be added and shown as existing wells on the Section 2.0 MC sampling location figure. The wells will also be added to the text and the sample quantities. Note there one other well, MW-NASB-069, would have provided good information except it was decommissioned and/or destroyed as a result of the Removal Action, as per the Draft Site 9 Monitoring Event 33 Report for September 2008 (ECC, December 2008).

   As per the Navy response to EPA Comment #2, note that temporary monitoring wells will be installed at the NASB Skeet Range and Topsham Skeet Range only as necessary depending on XRF results for lead and other factors.

5. **Comment:** Section 6.0, Project Deliverables; For some of the six sites that will undergo a MEC investigation, the results from that investigation will be relied upon to determine where sampling efforts should be focused as part of this MC work plan. Due to this phased approach, the Navy will need to transmit to MEDEP and EPA a final sampling design for these sites. EPA suggests that the Navy submit draft page changes for the work plan as a possible solution. The number samples currently specified in the work plan for the MEC sites may also change depending on results from the MEC investigation and this should be acknowledged in the work plan.

   Response: References to the MEC sites will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites.

6. **Comment:** Appendix A, Section 3.1.1.4.5 – Quarry; The MC vertical boundary for the Quarry site is defined here as 4 feet bgs where soil spreading has occurred and 1 foot bgs elsewhere. In **Section 2.8 - Contaminants of Concern**, it is stated that additional chemical analyses are planned to account for the fact that the site appears to have been historically used as a dumping area. For this reason, the vertical boundary must be expanded deeper (at least 10 feet bgs, pre-soil spreading topography, and perhaps deeper if waste materials exist and extend beyond the 10 feet) to account for the operational history of the Quarry site. The work plan, as it applies to subsurface soil sampling, needs to be revised accordingly.

   Response: References to the Quarry, a MEC site, will be removed from the subject SI MC Work Plan and will be addressed in a separate future MC Work Plan submittal for MEC sites.

7. **Comment:** Appendix A, Section 4.3.4 - Site 12 EOD Area; In its 12 February 2008 letter to the Navy on the Draft MEC work plan, EPA questioned the Navy proposal to only conduct geophysical surveys of the current and historical EOD berm areas, which represents a very small percentage of cleared Site 12 land area. EPA will need to review the Navy's response to this comment before it can make a determination on the adequacy of the field work proposed in this MC work plan.

   Response: References to the Site 12 EOD Area, an MEC site, will be removed from the subject SI MC Work Plan and will be addressed in a separate future MC Work Plan submittal for MEC sites.

8. **Comment:** Appendix A, Section 4.4 -Sampling Overview, ¶3 and QAPP; Surface water, ground water, and sediment samples should be collected according to the MEDEP/EPA approved Navy Base-Wide Long-Term Monitoring Program Quality Assurance Project Plan (QAPP). The Navy should ensure that these approved base-wide LTM SOPs are consistently used for all investigation and LTM program sampling.

   Response: The Base-wide monitoring QAPP is not cited because the objectives of the long-tem monitoring differ from those of the subject MC investigation. The Navy will, however, collect groundwater and surface water and sediment samples in accordance with the Basewide QAPP. Any sampling protocols not specifically provided in the Basewide QAPP are provided in the MC Work Plan.

9. **Comment:** Appendix A, Page 4-13 - Sampling and Analysis Plan and QAPP; EPA requests that field water quality meters (pH, dissolved oxygen, etc.) be calibrated according to the procedures provided in Appendix E of the Navy Base-Wide QAPP unless the specific manufacturer's procedure for a field instrument is equivalent to the Base-Wide QAPP procedure.

   Response: The Navy will follow the specific manufacturer's procedure for calibration of a given field instrument.

10. **Comment:** Appendix A, Page 4-14 - Temporary Monitoring Well Sampling; According to Lab SOP No. HPLC-Perchlorate and QAPP Worksheet #19, perchlorate samples are not to be field preserved. This paragraph suggests that perchlorate samples will be preserved. Please provide clarification in the text.

Response: There are no perchlorate samples proposed for the skeet ranges. This comment will be incorporated (with clarifications) to address the Machine Gun Boresight Range for the subject MC sampling plan and, in the future, for the MC sampling plan for the MEC sites (Site 12 EOD, Former Munitions Bunker West, and the Quarry), as applicable.

RESPONSE TO MEDEP COMMENTS DATED APRIL 10, 2008
REVISED DRAFT FINAL SITE INSPECTION WORK PLAN, MUNITIONS CONSTITUENTS AT SIX MUNITIONS RESPONSE SITES, DATED FEBRUARY 2008
NAVAL AIR STATION BRUNSWICK, MAINE

Additional Notes: For safety reasons and to provide a better basis for establishing MC sampling locations, the Navy has now decided to conduct MEC clearance before conducting the MC sampling at the MEC sites (Site 12 EOD, Quarry, and Former Munitions Bunker West). As a result, the subject Work Plan for all six sites has been revised to only include the remaining small arms ranges (Skeet Range, Topsham Skeet Range, and Machine Gun Boresight Range). This will allow field work at these small arms ranges to proceed in the Spring of 2009. A work plan for the three MEC sites will be issued at a later date, following evaluation of findings from the MEC clearance effort, and associated comments included herein will be addressed at that time.

Note that where the comment response provides revised text, text additions are shown in italics and deleted text is shown as strikethrough.

General Comments

1. **Comment:** Screening criteria for explosives, perchlorate, and propellants in surface water and sediment will need to be approved for the base. The current document does not include those compounds. The Navy should evaluate if there are sufficient existing values in the literature or if criteria will have to be developed for stakeholder approval.

   Response: The Navy used surface water and sediment values developed by EA as criteria; however, no values were developed by EA for explosives, perchlorate, or propellants. There are no surface water/sediment samples associated with the Machine Gun Boresight Range, and explosives, perchlorate, and/or propellants are not being collected for surface water/sediment samples at the two skeet ranges. Also, this comment will be incorporated into the future MC Work Plan for the MEC sites (i.e., Site 12 EOD, Former Munitions Bunker West, and Quarry), as applicable.

2. **Comment:** The Work Plan references the 2002 and 1996 CRREL documents as the basis for the soil sampling, but does not specify how samples will be collected. The two documents suggest somewhat different approaches, and differ on the number of composite aliquots. The text should also specify what soil depths will represent "surface soils" and how the subsurface samples will be collected. The 2002 document included in the draft-final plan and a recent May 2007 guidance from USACE-CRREL both suggest that up to 30 subsamples are needed to prepare a composite sample for surface soils. MEDEP recently attended a USEPA-sponsored Explosives Investigation training that also recommended multi-increment composite samples of 30 -100 aliquots. Based on the references provided and more recent CRREL documents, undisturbed surface samples for the three MEC sites should be taken from the top 0-15 cm (0-6 inches), with at least 30-part composites. This approach may not be possible at the Quarry where the potentially impacted soil is covered with up to 3 feet of land farmed soils. Once we know whether the land farmed soil can be distinguished from the underlying surface soil we will need to decide how best to approach this site for MC sampling. The VOC samples at the Quarry must be collect separately as discrete samples.

Response: References to the three MEC sites (Site 12 EOD Area, Former Munitions Bunker West, and the Quarry) will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites.

3. **Comment:** Discrete samples are acceptable for subsurface samples as proposed. The text must clarify if the discrete subsurface samples will be composited from the 1-5 foot interval, assuming collection by a 4-foot direct-push core sleeve. It may take more than one core depending upon the sample volume requirements.

   Response: The MEC sites will be removed from the subject MC Work Plan; the referenced 1-5 foot interval was noted for the Quarry, an MEC site.

4. **Comment:** Discrete samples are also acceptable for the locations where XRF will be performed as a pre-screen of the site. If no XRF will be used at the NASB Skeet Range, then surface soil samples there need to be composited also, although a smaller number of aliquots is acceptable. (These comments apply to Section 3.3.2, page 3-8, Soil Sampling Approach, and throughout)

   Response: The Navy agrees to conduct XRF sampling as a pre-screen at both the Skeet Range and Topsham Skeet Range. XRF sampling has already been accomplished by the USEPA at the Machine Gun Boresight Range (results will be included as an appendix to the MC Work Plan). Therefore, surface and subsurface soil samples collected at all three of these sites will be discrete samples.

5. **Comment:** The surface water samples at the Quarry and Site 12 are from small ponds, with little or no flow. The central or deepest portion of the pond has greater potential for deposition of sediment and impacted water than the edge. The surface water samples at those sites need to be collected in the middle of the water bodies and must be collected from the mid to deep portion of the water column, where there is direct interaction with contaminants in the sediment. A composite water sample of 2-3 depths is needed. The Navy may want to keep the independent water samples preserved for future analysis if contaminants are detected in the composite sample. The sediment must be collected in the deeper central portion or collection points within the surface waters bodies, and that two or three samples must be composited to improve the characterization.

   Response: References to the Site 12 EOD Area and Quarry, MEC sites, will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites. This comment will be incorporated into the future MC Work Plan for the MEC sites (i.e., Site 12 EOD, Former Munitions Bunker West, and Quarry), as applicable.

   This comment applies to sample collection at the NASB Skeet Range impoundment ponds, which is further detailed in response to MEDEP Comment #37.

Specific Comments:

6. **Comment:** SI Section 2.2 page 2-2; Phosphorous, presumably white phosphorous, from smoke-generating grenades is listed as a potential contaminant for the former munitions

bunker; however, a test for this constituent was not included in the QAPP. Please either justify why a test is not needed or include a test for white phosphorous.

Response: References to the Former Munitions Bunker West, an MEC site, will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites.

7. **Comment:** Section 2.3, page 2-3, Machine Gun Boresight Range: It is MEDEP's understanding that for machine guns that every fifth round or so there was a tracer round. The tracer round had pyrotechnics so that the bullet stream could be "seen". Therefore perchlorate must be included to the contaminants of concern for this site in groundwater. Methods include IC 6860 or LCM 6850.

   Response: Agree. The Navy agrees that tracers would have been used in ammunitions at the Machine Gun Boresight Range associated with perchlorate. Although perchlorate was not originally included because it is not likely a primary contaminant when compared with lead at the site, it will now be added to address this comment. Perchlorate will be analyzed using Method 6850 and filtered at the laboratory rather than in the field.

8. **Comment:** Section 2.4, page 2-4, para. 4; "Analyses for black powder constituents are omitted due to "the use of chemical constituents of black powder in pesticides,..."

   Black powder in shot guns has not been used in since the early 1900s when smokeless powder became widely available. During the period of these skeet ranges smokeless powder would have been used. Smokeless powder used nitrocellulose, nitroglycerine, and nitroguanidine, depending upon whether the powder was single-, double- or triple-based. Usually it is single or double based. Therefore method 8330B can be used for single and double based propellants for both soil and groundwater. For the plasticizers, such as dinitrophthalate, SVOC 8270 must be used. Since these propellants breakdown into nitrates, it must be added to the analyte list for groundwater. If nitrates are found above screening levels it may be necessary to perform additional sampling for nitroglyercine and other components, such as dinitrotoluene, of smokeless powder. Soil sample locations at the Naval Air Station and Topsham Annex skeet ranges must be added or relocated to collect soil in the area where any propellants emitted from the muzzle blast would have most likely fallen.

   Response: The Navy agrees that smokeless powder would have been used at both skeet ranges (NASB and Topsham) and could have resulted in the release of propellants, although these are not likely primary contaminants as compared to lead and PAHs. Because lead is the primary contaminant associated with skeet ranges and because of lead's documented toxicity to human and ecological receptors, lead can be used as a marker for contamination and other metals are expected to be spatially correlated with lead. For delineation of lead contamination, the use of both field analyses via XRF and laboratory analyses will be included.

   To address the comment, the following modifications will be made (or an explanation provided if the Navy does not agree with the requested modification):

- Nitroglycerin (propellant used in double, and triple, base powders) and burn rate modifier dinitrotoluene will be added to the sampling suite for soil and groundwater samples in and around the firing point.
- Nitrates will be added to groundwater samples throughout.
- Nitrocellulose will not be included because no criteria are available for screening of data results. Nitrocellulose is formed by nitrating cellulose (usually cotton or wood pulp) by treating it with concentrated sulfuric/nitric acid or sulfuric acid/potassium nitrate and subsequently removing most of the acid by washing or centrifuging. When used as a propellant or low-order explosive, it is also known as guncotton. Nitrocellulose is of low toxicity because it consists mainly or cotton (or wood pulp) with minor amounts of acid.
- Nitroguanadine will not be included because it is associated with triple-based propellants and so is not suspected at the two skeet ranges.
- Dibutylphthalate, an inert plasticizer used to make the grains less brittle, is a secondary contaminant not expected at significant concentrations and the screening criteria is relatively elevated (RSL of 6100 mg/kg for residential soil and 3,700 ug/L for tap water and so is not included.

Throughout the document for the NASB Skeet Range and Topsham Skeet Range, nitroglycerine (Method SW846 8330A), dinitrotoluene (Method SW846 8330A), and nitrates (Method SW846 9056 or EPA 300, groundwater only) will be added.

Also, indicator parameters useful in evaluating fate and transport potential will be added for four soil samples at each of the skeet ranges; these parameters will be analyzed for pH (SW846 9045D), total organic carbon (TOC) (Lloyd Kahn Method), and cation exchange capacity (CEC) (Method 9081); the selected samples will be biased toward locations with evidence of contamination (visual observations of fragments/shot and the results of field screening). [Of note, because the Machine Gun Boresight Range is adjacent to the NASB Skeet Range, the indicator parameters collected will be applicable to both sites]. Note that no field duplicates of indicator parameters are necessary.

The laboratory Worksheets #15, #19, #23 through #28, and #30 (Appendix A, Attachment 1A Worksheets) have also been updated to reflect the revised analytical suite and updated laboratory SOPs.

Note that the Work Plan will be revised to indicate that temporary monitoring wells will be installed at the NASB Skeet Range and Topsham Skeet Range only as necessary depending on XRF results for lead, as agreed upon during a conference call held among the Navy, EPA, and MEDEP on January 29, 2009.

The following text will be included concerning smokeless powder: "*Black powder was supplemented by smokeless powder early in the 20th century. Smokeless powder is named because the combustion products are mainly gaseous compared to black powder. Propellants (nitrocellulose and possibly nitroglycerine) and plasticizers (dinitrotoluene and dibutyl phthalate) are associated with smokeless powder.*"

9. **Comment:** Section 2.5, and Tables 2-1, 3-2, 3-3:

a.) Site 12 & Quarry - Propellants must be included as analytes at the EOD range due to the wide range of potential munitions disposed of at the site, and the presence of blank cartridge casings noted on the perimeter road by Malcom-Pirnie.

b.) Quarry – Analysis for soil DRO is needed based on the potential waste disposal at the site, and to evaluate if the land farming was successful in treating the soil.

c.) TOC analysis for soil may also be useful for evaluating distribution of munitions related compounds if a site proceeds to a Remedial Investigation.

Response:

a.) and b.) References to Site 12 EOD Area and the Quarry will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites.

c.) Several TOC soil samples will be collected at the three non-MEC sites, as described in the response to MEDEP Comment #8.

10. **Comment:** Section 2.7, page 2-6, Topsham Annex Skeet Range; "The majority (27 acres) of the SDZ extends of-base onto private property transferred to the MSAD No. 75."

This isn't quite correct. Only a small portion of the former skeet range was transferred to the Maine School Administrative District (MSAD). MEDEP suggests the following language: "The majority (27 acres) of the SDZ extends of-base onto private property *and onto land* transferred to the MSAD No. 75."

Response: Agree. The text will be revised as suggested.

11. **Comment:** Figure 2-4, Skeet Range (BNAS); No samples should be taken within the Site 6 area. The soils within this area were excavated as part of the remediation for Site 6 so any lead shot would have already been removed. (The outline for Site 6 needs to be shifted to the north following the cleared area.) Therefore the monitoring well can be dropped. MEDEP suggests moving one soil sampling locations to the northwest within the treed wedge northwest of Site 6 and the other south of Neptune Drive mid way between the road and the northern edge of the lower impoundment. Since this is the area where the two skeet ranges overlapped there is more likelihood of finding contamination. See comment 15.b. below.

Response: Agree. The text and MC sampling location figure will be revised as follows, as suggested:

- The Site 6 outline will be shifted to the north.
- The monitoring well previously shown within Site 6 will be eliminated.
- One of the surface soil samples within Site 6 will be moved to the northwest within the treed wedge.

- The other surface soil sample within Site 6 will be moved between Neptune Drive and the northern edge of the lower impoundment.

The changes above will affect sample quantity updates to the following sections:

- SI Work Plan: Section 3.3.2 and Tables 3-2 and 3-3
- Appendix A SAP: Table 3-1, Section 4.3, Table 4-4
- Appendix A SAP Attachment 1 QAPP, Attachment 1-A, Worksheet #20

12. **Comment:** Figure 2-5 (Site12); The Navy's rationale for the northernmost surface water/sediment sampling location is valid. However an additional surface water and sediment sample needs to be added so that there is one in the center of the pond and one at the southern end also needs to be moved east to the middle of the pond. Also see comments 5 above and 15.c below.

    Response: References to Site 12 EOD Area, an MEC site, will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites.

13. **Comment:** Figure 2-6, Quarry; See comment 5 above. The number of sample points and spacing is adequate however the surface water and sediment samples need to be relocated southeastward to the middle of the water body.

    Response: References to the Quarry, an MEC site, will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites.

14. **Comment:** Figure 2-7, Topsham Annex Skeet Range; The southern most sediment sample should be moved approximately 175-200 feet northward (downgradient to account for flushing of the stream sediment. Also the components of single and double based propellants must be sampled for down range of the firing point in soil and groundwater. (See comment 8 above.)

    Response: The Navy agrees to move the southernmost sediment sample approximately 175-200 feet northward (downgradient to account for flushing of the stream sediment. As per the response to Comment 8, nitroglycerine and dinitrotoluene will be sampled for in soil and groundwater but only for samples at and near the firing point (i.e., in front of the shotfall zone).

15. **Comment:** Section 3.3.2, Table 3-2;

    a.) **Machine Gun Boresight Range** – Based on the disturbed nature of the soils near the site, the uncertainty regarding the placement of the berm soils, and the proximity to the skeet range and fire training area, the background sample cannot be collected within the bounds of the range. A site should be chosen in the undeveloped portion of the range, or possibly to the east near the base boundary. The historic aerial photographs can be used to determine if the selected area has been undeveloped and therefore

suitable as a reference location. Also it is not clear whether the background sample is just for the Machine Gun Boresight Range or for other MMRP sites. Please clarify.

Response: One surface soil background sample was planned to suffice for both the Skeet Range and Machine Gun Boresight Range, which are adjacent to each other. The sampling location map shows the MC sampling locations, including the planned background sample located south of the surface danger zone and beyond the dirt pile. This location will be moved approximately 175 feet to the southeast and clearly labeled as a background sample for both the Skeet Range and the Machine Gun Boresight Range. A note will be added to the Skeet Range sampling location map referencing the common sample on the Machine Gun Boresight Range figure. Text and tables will be checked to make sure this is clear. Considering that facility background development is underway, the intent was not to overdo it on collection of site-specific samples.

b.) Skeet Range – It is unclear to MEDEP why an XRF investigation is not also being performed on the Skeet Range at NAS-B. It would provide invaluable information for establishing the range shotfall fan and locations for MC sampling. However, if no XRF will be performed to select locations, surface soil samples must be composites rather than discrete grabs.

Response: It was agreed upon during the October 2007 site visit meeting that XRF was not necessarily needed at the SI phase and there was an objective at the time to minimize changes to the original Malcolm Pirnie Work Plan. However, the Navy agrees on the utility of XRF sampling and this task will be added to the Work Plan for the Skeet Range.

c.) Site 12 – See comments 5 and 12 above. The southern sample point for surface water/sediment must be moved to the central portion of the pond, where there is the greatest likelihood particulate matter has collected as the pond is flushed by precipitation events and another sample point added to the center of the pond. The sediment sample must be a composite of three aliquots to reduce the impact of heterogeneity in the sediment.

Response: References to Site 12 EOD Area, an MEC site, will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites.

d.) Quarry (also reference Figure 2-6 and Appendix A, Section 3.1.1.1, page 3-9, Bullet 1) – The table indicates 10 surface and 15 subsurface soil samples will be collected. The text in the appendix clarifies that some locations will target the former land surface beneath the land farmed soil; please add this information to the main body of the text as well. (Also see comments 26 and 35 below.)

Response: References to the Quarry, an MEC site, will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites.

16. Comment: Section 3.3.2.1, Soil/Sediment Sampling:

a.) Correct per additions and deletions noted above in comments 9 & 11.

Response: Revisions will be made in accordance with the Navy response to Comment 9 and 11.

b.) For the NAS-B skeet range, the sediment sample on the small tributary needs to be moved closer to the pond. Also for the lower impoundment pond, MEDEP assumes that the pond was dammed and flooded and not dredged like the upper impoundment pond. If so, then the samples must be taken within the impoundment not on the edge. MEDEP suggests one within the northern most area of the impoundment about 50 feet out from the edge, one just north west of the weir about 50 feet NW, and the last in the middle of the impoundment roughly centered between the other two points.

Response: The Navy agrees to move the sediment sample on the small tributary closer to the pond. The Navy also agrees to collect the sample from the middle of the lower impoundment pond rather than the edge. The Navy agrees to collect an additional two sediment samples within the impoundment at the MEDEP recommended locations. This revision will necessitate a change in sampling methodology since the samples will need to be collected using waders or via a small watercraft.

The changes above will affect sample quantity updates to the following sections:

- SI Work Plan: Section 3.3.2 and Tables 3-2 and 3-3
- Appendix A SAP: Table 3-1, Section 4.3, Table 4-4
- Appendix A SAP Attachment 1 QAPP, Attachment 1-A, Worksheet #20

The change in sample location from the edge to the middle of the impoundment will necessitate a modification of the sample collection methodology. Please refer to the details in response to MEDEP Comments #5 and #37.

17. **Comment:** Section 3.3.2.2, page 3-9, Surface Water/Groundwater Sampling…; One well is proposed for outside the shotfall zone surrounding the target area. The purpose for this well is unclear; please clarify.

Response: The following sentence will be added to Section 3.3.2.2, end of 2nd paragraph:

"*The well outside of the shotfall zone will aid in determining the extent of contamination, if any, beyond the shotfall zone.*"

18. **Comment:** Section 3.3.2.3 and Appendix A Section 3.0; Method 8330 was updated in February 2007 to 8330A, please revise as needed.

Response: Empirical Laboratory, will be conducting Method 8330A analysis for explosives as per Worksheet #30 (Appendix A SAP, Attachment 1-A UFP-QAPP Worksheets) Analytical Services Table. A search and replace of the Work Plan text (including the Appendix A SAP) will be conducted to add the "A" suffix for any instance where it is not already present.

19. **Comment:** Table 3-3, pages 3-11 & 3-12, Sample Summary Table;

a. Please add perchlorate to the Machine Gun Boresight Range.

Response: The Navy agrees to add perchlorate for groundwater analysis at the Machine Gun Boresight Range.

b. Please add propellants and plasticizers for both the skeet ranges.

Response: Please see the response to MEDEP Comment #8. Nitroglycerine and dinitrotoluene will be added for those samples at the firing point (all samples in front of the shotfall zone) at both skeet ranges and nitrates will be added to groundwater throughout for both of the skeet ranges.

c. Please add propellants for Site 12 and the Quarry.

Response: References to Site 12 EOD Area and the Quarry will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites.

d. All TAL metals must be analyzed for both Skeet Ranges due to the other metals constituents associated with shotgun pellets. (See section 2.4 of the workplan.)

Also, only lead was proposed for analysis of the Brunswick skeet range sediment samples. To be consistent w/ the other sites, it would make sense to do the TAL metals list for this site as well.

Response: Section 2.4 of the Work Plan notes the primary contaminants expected, including lead. For the purposes of the SI, TAL metals are identified for soil and groundwater analysis at both Skeet Ranges (Table 2-1, Table 3-2, Table 3-3, etc.) and no changes are necessary. For sediment, the Topsham Skeet Range includes TAL metals. It was only the sediment samples at the on-base Skeet Range that were limited to TAL lead because the samples will be collected from impoundment ponds where other metals may be present that are not attributable to the Skeet Range (similar to the exclusion of PAH samples from the impoundment ponds). However, the Navy agrees to revise the Work Plan to analyze both surface water and sediment samples at the Skeet Range for TAL metals rather than just lead. The ratio of lead to other metals present in these samples may aid in the evaluation of whether or not contamination, if any, is site related.

20. **Comment:** Section 3.3.2.6, XRF Investigations; The text needs to specify how the surface soil samples will be collected. Specifically, describe what area will represent each location and to what depth will soil be collected.

Response: The "how" is discussed in the Appendix A SAP and will be specific for each site in describing areas represented and soil depths. The Appendix A SAP discussion has been expanded as per the response to this MEDEP comment and MEDEP Comments #33 and #37. For Section 3.3.2.6, the addition of XRF at the NASB Skeet Range will be added, as follows:

"At the NASB Skeet Range, aerial photographs are available showing the site location. However, the site has been disturbed during construction of the impoundment ponds and the disturbance may have extended beyond the edges of the impoundment ponds. The site is further complicated in that the orientation of the skeet range was changed. Maximum concentrations are expected within the shotfall zone(s), working radially outward from the center. Up to 90 surface soil samples are planned for collection via XRF, with a minimum of 30 surface soil samples. The minimum 30 samples would be sufficient if it can be established that maximum lead concentrations present exceed the residential standard of 400 mg/kg and the source area location (i.e., shotfall zone) is consistent with that of the current-day conceptual site model, because the decision to move forward to an RI would be evident at this point and the data collected could be built upon during the RI. Otherwise, up to 90 samples will be collected via XRF to meet the objective. During field analysis, the XRF lead data will be reviewed on a ½ day basis (approximately every 15 samples) to support the continuing effort of determining sample locations."

Also, the following sentence will be added to the end of the last paragraph of Section 3.3.2.6: *"Additional details on XRF Sampling are provided in the Appendix A SAP, Section 4.7.4 XRF Sampling of Lead in Soil."*

21. **Comment:** Section 6.0, Project Deliverables; As with other investigations, the State requires that the data be submitted electronically conforming to the Department's Version 5 Electronic Data Deliverable (EDD) format.

    Response: Agree. The following bulleted item will be added to the list of additional project deliverables:

    "- *Electronic Data Deliverable (EDD) format, Version 5, submitted to MEDEP electronically.*"

Appendix A-Sampling Analysis Plan (SAP):

22. **Comment:** Section 3.0, page 3-1, para 4; The Maine Remedial Action Guidelines for soil are not conservative enough to be used for screening criteria. Please delete the reference unless it is lower than all the other screening criteria. If that is the case, please add a footnote to that affect.

    Response: USEPA screening criteria are also specified in the same sentence and, in fact, USEPA soil screening criteria are prioritized in Worksheet #15 (Appendix A SAP, Attachment 1-A UFP-QAPP Worksheets) Reference Limits and Evaluation Table.

23. **Comment:** Section 3.1.1.1, page 3-4, Bullet 1; MEDEP expects that a follow-up MC investigation will need to be completed for vegetated areas after any additional MEC screening is implemented, in order to clear the property for transfer.

    Response: References to the MEC sites will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites.

24. **Comment:** Section 3.1.1.1, page 3-10, final sentence; If the waste disposal and EOD activities at the Quarry are confirmed, MEDEP considers that site another likely candidate for groundwater contamination.

Response: References to the Quarry, an MEC site, will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites.

25. **Comment:** Section 3.1.1.2, page 3-11, MC, para 3, 1st sentence; It is unclear which section 3 is being referenced and it is not section 3 of the workplan or of appendix A. Please specify.

Response: The reference will be corrected to...."(see ~~Section 3~~ *Worksheet #15 [Appendix A SAP, Attachment 1-A UFP-QAPP Worksheets] Reference Limits and Evaluation Table*)"

26. **Comment:** Section 3.1.1.4.5, Quarry - page 3-13; "The vertical boundary for MEC is defined as the surface."

Please clarify this statement because it is unclear whether the Navy is discussing the current ground surface or the surface prior to the land farming. If the 1 to 5 foot interval refers to the current ground surface, then the samples should be biased to the 3 to 5 foot depth and the limits of land farmed soil need to be identified where possible in the field. (See comments 15.d. & 35.)

Response: References to the Quarry, an MEC site, will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites.

27. **Comment:** Section 3.1.1.4.5, Site 12 - page 3-14; "For the Site 12 EOD Area, where MEC is anticipated, an NFA recommendation is unlikely following SI. However, an NFA recommendation for MEC may be possible if addressed via a future interim measures removal effort for MEC and no MC is identified in samples collected from the site."

Until bedrock groundwater is investigated for perchlorate MEDEP cannot concur with a NFA for Site 12. Please revise. MEDEP suggests the following language: "...and no MC, *including COCs in bedrock groundwater,* is identified in samples collected from the site."

Response: References to Site 12 EOD Area, an MEC site, will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites.

28. **Comment:** Section 3.1.1.4.5, Surface MEC - page 3-14; Please add BIP (blow in place?) to the acronym list

Response: References to the Quarry, an MEC site, will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites.

29. **Comment:** Section 3.1.1.4.5, Regarding MC - page 3-14, Bullet 1; This bullet references section 3. There are number of section 3's in this workplan. Please be more specific. (Also regarding NFA, see comment 27 above.)

Response: The reference will be corrected to...."(see ~~Section 3~~ *Worksheet #15 [Appendix A SAP, Attachment 1-A UFP-QAPP Worksheets] Reference Limits and Evaluation Table*)"

Also, please see the response to MEDEP Comment #27.

30. **Comment:** Section 3.1.1.6, page 3-15, Regarding MC Sampling: The text and QAPP does not evaluate the overall decision error tolerance for the proposed sample plans, and this assessment must be discussed, at least generally in the text.

Response:

The following text will be added to the end of Section 3.1.1.6:

"Further discussion of data usability assessment is provided in the QAPP (Appendix A, Attachment 1) Section 4.2.3."

31. **Comment:** Section 4.3.1, page 4-1; Based on the references provided and on others reviewed by MEDEP the highest concentrations of explosive residues are likely present in the top 15 cm or 6 inches of soil at Site 12 and the Munitions Bunker West site, rather than the top foot. All of these references also discuss the limitations of discrete sampling versus composited samples. The surface soil samples need to be collected in accordance with these recommendations, specifically they should be at least 30-aliquot composites of the 6-inch interval for a given location. (Also correct/clarify in Section 4.4.)

Response: References to the MEC sites will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites.

32. **Comment:** Section 4.3.1, page 4-1 and Section 4.3.4, page 4-3; *"...one or more wells may be placed to aid in evaluating groundwater flow direction rather than investigating a potential MEC anomaly."*

MEDEP will be very conservative when considering shifting points from MC evaluation to improving characterization of groundwater flow, particularly given the limited number of groundwater monitoring points at each site. The lack of MEC does not preclude potential dissolved constituents at the site. Additional piezometers may be proposed to improve triangulation of groundwater flow if needed. Also see comment 27 above.

Response: References to the MEC sites will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites.

33. Comment: Section 4.3.2, page 4-2 and Section 4.7.4, page 4-12: The text should describe how the XRF locations will be used to locate the laboratory sample collection. It is unclear if the same soil will be subsampled, or if the location will be re-occupied and additional sample collected, or some other method applied. Please clarify.

Response: Sections 4.3.2 and 4.7.4 will add a reference to Section 4.7.4 XRF Sampling of Lead in Soil for additional details.

Section 4.7.4 will be rewritten as follows:

"4.7.4 XRF Sampling of Lead in Soil

X-Ray Fluorescence (XRF) spectrometry investigations will be conducted at two sites – ~~Machine Gun Boresight Range~~ *NASB Skeet Range* and Topsham Skeet Range. Discrepancies and or lack of information *and/or site disturbance* on these two sites have led to uncertainty on the site boundaries. Therefore, XRF investigations may aid in establishing the locations for MC sampling; in particular, by obtaining a greater number of real time samples resulting in a higher level of confidence in identifying area(s) of maximum contamination for fixed-based laboratory analysis. At each of the two sites, a grid will be established over the site area and samples will be analyzed in the field for lead by XRF, beginning with the grid squares expected to have the maximum concentrations and working outward. ~~The USEPA will conduct the XRF investigation at the Machine Gun Boresight Range.~~ TtNUS will conduct the XRF investigation*s* at the Topsham Skeet Range *and NASB Skeet Range.*

An XRF investigation was conducted by the USEPA at the Machine Gun Boresight Range on May 1, 2008 and results are provided in Appendix B. At the Machine Gun Boresight Range, the disposition of the former berm is unknown and a dirt pile on site may or may not be the remnants of the former berm. The existing dirt pile boundary will be mapped in the field using a hand-held surveying Global Positioning System (GPS) device. The distance to the dirt pile has been measured but the entire outline of the dirt pile should be mapped. Maximum concentrations are expected at the existing dirt pile and/or the former berm location based on historical aerial photographs. *Between 30 and* ~~Up to~~ 90 soil samples ~~are~~ *were* planned for collection *and screening* via XRF~~, with a minimum of 30 soil samples~~. The minimum 30 samples may be sufficient if it can be established that maximum lead concentrations present exceed the *MEDEP RAGS* residential standard of *375* mg/kg *(USEPA action level of 400 mg/kg)* and the source area locations (i.e., former berm location, existing dirt pile, and area in between) are consistent with those of the current-day conceptual site model~~, because the decision to move forward to an RI would be evident at this point and the data collected could be built upon during the RI~~. Otherwise, up to 90 samples *were to* ~~will~~ be collected via XRF to meet the objective. During field analysis, the XRF lead data ~~will be~~ *were* reviewed on a ½ day basis (approximately every 15 samples) to support the continuing effort of determining sample locations. *As shown in Appendix B, of the 50 XRF samples collected (which excludes 6 duplicate XRF samples and 6 fixed-base laboratory confirmation samples), only 3 had lead concentrations exceeding the 375 mg/kg MEDEP RAGS residential standard. These XRF samples included two samples at location MBR-SB-01 (surface soil at 1640 mg/kg and 12-16" interval at 610 mg/kg) and location MBR-SB-22 at 401 mg/kg. These results will be further assessed in combination with the planned soil sampling results in evaluating site contamination.*

At Topsham Skeet Range, very little information is available on the ~~exact location~~ *actual operation* of the range and aerial photographs do not resolve this issue. Any remaining

historical features, such as remaining wooden posts, etc., will be mapped via a hand-held surveying GPS device. Maximum concentrations are expected within the shotfall zone, working radially outward from the center. *For both the Topsham Skeet Range and the NASB Skeet Range, all field visual observations including physical observation of lead shot, bullets, and pieces of clay targets will be recorded on sample log sheets. Prior to collection of the XRF samples, a site "walk over" will be conducted by the FOL to identify any obvious areas of fragment/lead shot accumulation. Additional discretionary surface soil samples may be collected based on visual observation of accumulated fragments or shot in the area to be sampled. Emphasis will be placed on collecting samples required to delineate contaminated areas. Additional "step-out" samples will also be collected at the boundaries of the area to be sampled if lead concentrations greater than the field action level of 300 mg/kg are detected based on the results of the XRF field screening analysis of the initial surface soil samples. An XRF field action level of 300 mg/kg will be used as the discriminator between contaminated and uncontaminated locations, which corresponds to the MEDEP level of 375 mg/kg reduced by a 20 percent margin of error to minimize the potential that contaminated areas go undetected. The margin of error was selected based on recent experiences with field XRF analyses at other small arms ranges.*

For the skeet ranges, the initial horizontal study boundary encompasses the area that, based on historical information, is most likely to have been impacted by site activities as shown on the sample location maps. Lateral expansion of this horizontal study boundary may be necessary if the XRF concentrations in the samples collected along this boundary exceed 300 mg/kg. The study area has the following three sub-zones: the firing point, an area downrange of the firing line where clay targets (i.e., missed targets) and clay target fragments actually hit by shotguns would have accumulated (spent lead shot would also be present in this area), and the shotfall zone that is the downrange area where the greatest concentrations of lead shot would accumulate although targets and target fragments are unlikely to be present. The sampler will try to ensure good spatial coverage of the targeted contamination areas when selecting XRF sampling locations to validate the sampling design; areas outside of those expected to be contaminated will also be sampled and if the sampling design is correct, it is anticipated that the additional locations will exhibit lesser, and perhaps even non-detectable, concentrations of lead than are found in the targeted contamination areas.

~~Similar to the Machine Gun Boresight Range~~, *Between 30 and* ~~Up to~~ 90 soil samples are planned for collection *and screening* via XRF *at each of the two skeet ranges*~~, with a minimum of 30 surface soil samples~~. The minimum 30 samples may be sufficient if it can be established that maximum lead concentrations present exceed the ~~residential standard of 400 mg/kg~~ *MEDEP level of 375 mg/kg* and the source area location (i.e., shotfall zone) is consistent with that of the current-day conceptual site model~~, because the decision to move forward to an RI would be evident at this point and the data collected could be built upon during the RI~~. Otherwise, up to 90 samples will be collected via XRF to meet the objective. During field analysis, the XRF lead data will be reviewed on a ½ day basis (approximately every 15 samples) to support the continuing effort of determining sample locations.

The XRF results will aid in locating the samples for MC fixed-based laboratory analysis, which will be collected at the locations of highest concentrations established by the XRF investigation. The XRF results will be used for screening purposes only and so no confirmation sampling for the XRF samples are planned prior to collecting the MC soil samples for fixed-based laboratory analysis. *However, all XRF sample data will be later correlated with fixed-base laboratory data, when received. Performance criteria (correlation coefficients) for correlation of lead XRF data to fixed-base laboratory data for lead is based on a correlation coefficient greater than or equal to*

0.65 and less than or equal to 1.00. These correlations will be used to translate field XRF values into equivalent laboratory lead concentrations. Correlation analyses are not anticipated to be generated for the other metals.

Planned soil sampling locations shown on the figures may be adjusted based on XRF results. *After collection of all XRF samples and confirmation or adjustment of the shotfall zone, the actual samples for fixed-base laboratory analyses will be determined in accordance with the sampling design shown on the figure. Soil sampling methodology is discussed in Section 4.7.1. All sample locations will be marked with a wooden stake or brightly colored pin flag indicating the sample location. Coordinates will be determined by GPS at each individual sample location which will allow for future repeatable investigations or guide in any remedial action. All sample location markers will be removed prior to demobilization from the site.*

Each soil sample collected for XRF analysis will be removed and placed in either an unused, labeled, large zip-lock bag (gallon sized) or a decontaminated stainless steel mixing bowl. The soil will be thoroughly mixed using a kneading motion if in a zip-lock bag or with a stainless steel spoon (or equivalent) if in a bowl. Any gravel, rocks, or other large debris items will be removed during the mixing procedure. *Any bullets, bullet fragments, or lead shot observed in a sample will be recorded in a logbook and then removed so as not to bias lead results.* Mixing will be performed for approximately 2 minutes or until the sample material appears to be well mixed. After the soil sample is thoroughly mixed, the soil will be transferred to an unused, labeled large zip-lock bag (if not already) and stored in a cooler that is always maintained under custody procedures. A representative aliquot of the homogenized soil (approximately 1.5 cubic inches) will be collected and placed in an unused smaller zip-lock bag. The large zip-lock bag will be returned to the cooler. The representative aliquot will be dried in a toaster oven at a temperature not to exceed 150 degrees Celsius. After the sample is thoroughly mixed and dried, the sample will be analyzed with XRF in accordance with the TtNUS XRF SOP (S-16). The XRF results for lead for each sample will be recorded on a daily sample analysis form. After each day of XRF analysis, the data file generated by the XRF spectrometer will be downloaded for review by the FOL and the Project Manager.

Upon validation, laboratory lead concentrations that are co-located with field XRF lead concentrations will be compared through a correlation plot. The field data will be plotted against the laboratory data, and a linear least-squares fit will be applied to generate an estimate of the "best" line through the data. If the data set exhibits more than one region where there appears to be a linear fit to the data but the regions have different slopes, the data may be subdivided into separate regions for fitting. No more than three separate regions of the correlation will be fit in this manner, and as few regions as possible will be used to adequately represent a linear fit. If a correlation coefficient (commonly denoted as "r") of 0.65 or greater is obtained for a region, the fit will be considered adequate to additionally translate and use field XRF results to the equivalent laboratory lead concentrations with confidence. A correlation coefficient less than 0.65 will be considered unacceptable for making this translation and reliance on the laboratory data only will be used. An ideal correlation coefficient would be 1.0. The use of subdivisions of the overall concentrations range to represent a best linear fit will be most important for concentrations in the range of about 300 to 450 mg/kg because the 375 mg/kg risk screening value for lead is within this range."

34. Comment: Section 4.3.3, page 4-2;

a.) If the sample locations at the Skeet Range will not be guided by XRF screening and therefore should be composites rather than discrete samples since the same heterogeneity in the environment applies for the lead pellets. The seven-point wheel approach would be acceptable here.

Response: Discrete samples will be collected at the Skeet Range guided by XRF, which will be added as per the response to MEDEP Comment #33.

b.) Table 3-3 indicates that PAHs will be included as part of the sediment analyses. Please correct table 3-3.

Response: Table 3-3 (of the Work Plan) will be corrected to delete "2 surface water" and "2 sediment" under the PAHs column for the NASB Skeet Range.

35. Comment: Section 4.3.5, page 4-4; *"Subsurface samples will be collected from depth intervals of one to five feet."*

Is the intent to homogenize the soils from that interval for collection (except for VOCs)? The approach is acceptable for non-land farmed areas. The soil of interest is the former ground surface below the land farmed soil, which may be up to three feet deep. The text needs to clarify that the target soil interval surface may be up 3 feet below ground surface. The land farmed soil should not be sampled for MC determination unless there is no way to distinguish it from the other fill in the Quarry. If direct-push methods are used, multiple points may be needed to get sufficient soil volume for the intended analyses.

Response: References to the Quarry, an MEC site, will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites.

36. Comment: Section 4.3.6, page 4-4 and Figure 2-7; *"The surface water/sediment samples will be collected from the brook running through the site with one sample located at the brook and the other within the shotfall zone."*

Please revise to note both surface water/sediment locations will be within the shotfall zone, and correct based on the revisions outlined in comment 11 above.

Response: Agree as per the Navy's response to MEDEP Comment #11. The sentence will be revised as follows: "The surface water/sediment samples will be collected from the brook running through the site with ~~one sample located at the brook and the other within the shotfall zone~~ *both samples collected within the shotfall zone."*

37. Comment: Section 4.4, page 4-5:

a.) The soil sampling procedures described in Appendix B of the ERDC/CRREL TR-02-1 document and other recent guidance reviewed by MEDEP all recommend composites of 30 aliquots and the rigorous processing of the resulting sample at the laboratory,

which includes taking 30+ sub-aliquots to make the final aliquot to be analyzed. Navy must follow these procedures for the three MEC-suspected sites to reduce the potential heterogeneity of the soil data.

Response: References to the MEC sites, including the associated seven-wheel sampling approach and associated SOP appendices, will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites.

b.) In order to sample the sediment and surface water in the central portion of the surface water bodies as needed at Site 12 or the Quarry, the sample technique will have to be modified per the techniques in SOP S12.

Response: References to the MEC sites will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites. However, the comment still applies to the NASB Skeet Range and will be incorporated into Section 4.4 and SOP S12 (SA-1.2), as per the Navy's response to part d of this comment.

c.) It is not clear from the Standard Operation Procedure (SOP) for soil sampling just how deep the proposed samples for the Skeet Ranges are to be collected. Surface soil samples for skeet ranges should not be deeper than 3 inches since it unlikely that the pellets penetrated the soil deeper than that. Please clarify the sampling depth in the text and in the SOP. The seven point wheel approach is acceptable per sampling location due to the heterogeneity of the pellets. (See comment 34 above.)

Response: Discrete sampling will be used, as guided by XRF, as per the Navy's response to MEDEP Comment #34.

Concerning sample depth intervals, for trap and skeet ranges, ammunition is dispersed as pellets over a small area in the direction of fire. Pellets dispersed from a shotgun would be deposited on the ground surface and not significantly penetrate the ground unless disturbed. Because both of the Skeet Ranges may have been disturbed, the Navy recommends a 0 to 6-inch depth interval for surface soil samples at the Skeet Ranges rather than the requested 0 to 3-inch depth interval. The Navy requests assurances from the EPA/MEDEP that the 0 to 6 inch depth interval for surface soils will be considered as being representative of surface soil and usable in the future for risk assessment purposes, if necessary. A given subsurface soil sample will be collected at a 6 to 12 inch depth interval bgs directly below the associated surface soil sample.

d.) In regard to discrete samples for sediment and surface water samples, see comment 5 above.

Response: Agree. Site 12 EOD and the Quarry are MEC sites and MC sampling is deferred but the comment is applicable for the NASB surface water/sediment sampling at the impoundment ponds. As requested in MEDEP Comment #5, the surface water samples will be collected in the middle of the water body and will be collected from the mid to deep portion of the water column, where there is direct interaction with contaminants in the sediment. Composite surface water samples will be collected from two to three depths. Composite sediment samples will be collected from two samples to improve the characterization. The SAP will also note that the samples will be collected by field personnel using waders or, if too deep, a small watercraft to access the middle of the water body. Text changes to the Work Plan will be made accordingly.

For surface water at the NASB Skeet Range, field parameter data will be collected (as per the SOP S12 Surface Water and Sediment Sampling) and this will be added to the text (SAP Appendix A and Section 4.7.3, page 4-10).

38. **Comment:** Section 4.7.2, page 4-10 and Section 4.8.4, page 4-15; Where possible the screen and riser should be pulled out when the temporary points are abandoned, as is stated in Section 4.8.4. The previous Section only indicates that the PVC will be grouted in place. Please revise Section 4.7.2.

    Response: Agree.

39. **Comment:** Section 4.14, page 4-22;

    a.) Please submit an electronic table of the survey data with the report for the investigation.

    Response: Agree. The following sentence will be added after the 2nd sentence of the Work Plan, Section 3.4 Report, page 3-16 and in Section 4.14 Surveying, page 4-22, of the Appendix A SAP:

    "*Survey data coordinates (both GPS and land surveyor based) will be provided in the report, as well as an electronic version.*"

    b.) "Additionally, any areas with concrete debris must be mapped..." Please change "should" to "will".

    Response: Agree.

40. **Comment:** Table 4-4, page 4-27, Quantities of Analyses for the Skeet Range, Soil; According to Table 3-2 there should be 28 field samples and 3 duplicates. Please correct.

    Response: Agree. The subsurface soil samples were inadvertently left out of Table 4-4 and will be corrected.

41. **Comment:** Section 5.1, DQCR, page 5-1; Please add DQCR to the acronym list and change "Malcolm Pirnie" to "Tetra Tech NUS".

    Response: Agree.

Attachment 1:

42. **Comment:** Section 2.1.2.4, Field Equipment...page 2-2; "If equipment that is out of calibration cannot be brought into calibration, the equipment will not be used."

    Please add: "If the equipment cannot be used then new equipment must be acquired before proceeding."

Response: Agree.

Attachment 1-A:

43. **Comment:** QAPP Section 1.6.2.2 page 1-7 Accuracy/Bias: Although laboratory control charts are a good way to assess whether or not a laboratory system is in control, the control limits generated by these charts may or may not be adequate for meeting the goals of this project. The site specific accuracy limits must be set for the project. Note that in Worksheet 28 under Method/SOP QC Acceptance Limits, language [presumably] from the laboratory does state that nominal limits may be set on a project specific basis.

    Response: The following sentence will be added: "*Nominal limits will be set on a project-specific basis (see Worksheet #28).*"

44. **Comment:** Section 1.6.2.3 page 1-8 Sensitivity: Sensitivity is an important issue for this project since data will be used to evaluate whether or not the site qualifies for no further action. A low-level [at or near the quantitation limit] laboratory control sample must be processed and analyzed each day samples are processed and analyzed. These low level laboratory control samples should meet nominal acceptance limits.

    Response: The following sentence will be added to the Work Plan and also added to the lab scope:

    "*A low-level [at or near the quantitation limit] laboratory control sample must be processed and analyzed each day samples are processed and analyzed. These low level laboratory control samples should meet nominal acceptance limits.*"

45. **Comment:** Section 2.5.2 page 2-7 Data Package Deliverables: Results must be provided to MEDEP electronically in the Department's Environmental Data Deliverable (EDD) format for the Environmental and Geographic Analysis Database (EGAD). This should include field parameter data, water level and flow data, and laboratory analytical data for soil and groundwater. Laboratory analytical data should include field quality control sample results, surrogate recoveries in percent, and matrix spike/matrix duplicate recoveries in percent. All reports that include discussion of new data should include that data in the EDD format as an electronic deliverable. Specific EDD formats and additional information can be found on the Department's EGAD web page at http://www.maine.gov/dep/rwm/egad/. Any additional questions about the EDD or EGAD should be referred to Erika Bonenfant, the Department's groundwater database manager at 207-287-5767. According to the Department's legal authorities 10 MRSA section 9418(1) and 9418(2)(A) provide statutory authority for the Department to require electronic submission of data in a specified format.

    Sample locations and other geographic location data must be provided to sub-meter accuracy in a map file or table for uploading to the Department's GIS (Arc GIS). Universal Transverse Mercator (UTM) coordinates based on North American Datum (NAD) 1983 are preferred but at a minimum the projection, units, and datum must be submitted.

Response: Both analytical and mapping location data will be provided to MEDEP as requested and text from the comment will be added to Section 2.5.2.

46. **Comment:** Section 4.2.2.1 Validation Activities; Please state what level of data validation will be employed. Worksheet 36 references Region I data validation guidelines. Those guidelines provide for a tiered approach, with levels given as tier 1, tier 2 or tier 3 validation. Please state which tier will be followed, and whether or not there will be some modification of this level of validation.

    Response: The following sentence will be added "*Region I, Tier 3 data validation will be employed.*"

47. **Comment:** Section 4.2.3 page 4-5 Step III; Please clarify that a suspected data outlier will not be removed from consideration unless a sampling or analytical error can be identified. It is not enough to say the data point represents a physical quantity that is different from the rest of the data set and therefore should be removed.

    Response: The following sentence will be added: "*A suspected data outlier will not be removed from consideration unless a sampling or analytical error can be identified.*"

48. **Comment:** Section 4.2.3.1 page 4-5 Data Limitations; Grouping soil and sediment samples may or may not be appropriate for statistical analyses of the data. Sediments may contain much more moisture than soils.

    Response: Agree. Sediments with high percent moisture (>50%) will be discussed separately from soil as these types of sediment cannot be considered part of the same population as soil samples. Text will be revised accordingly.

49. **Comment:** QAPP Worksheet 3, page 10; Please correct the spelling of "sait" in the e-mail address.

    Response: Agree.

50. **Comment:** QAPP Worksheet 6, page 17: Please update the Navy RPM (2 places) to Todd Bober.

    Response: Agree.

51. **Comment:** Worksheet 12 page 26 and all other Worksheets 12 where statistically derived measurement performance criteria are listed; Measurement performance criteria [MPC] for accuracy/ bias need to be site specific. The laboratory derived limits may or may not be

applicable to site requirements. Note that in Worksheet 28 under Method/SOP QC Acceptance Limits, language [presumably] from the laboratory does state that nominal limits may be set on a project specific basis.

Response: The laboratory derived limits have been evaluated and are appropriate for this site. The limits are attached to this comment response letter for your perusal.

52. **Comment:** Worksheet 12 page 27 and all other Worksheets 12; Sensitivity must be assessed with a low-level laboratory control sample rather than simply relying on previously derived quantitation limits.

Response: Sensitivity will be determined by the low level calibration standard for all organic SW-846 methods.

53. **Comment:** Worksheet 12 page 26 and all other Worksheets 12; The measurement performance criteria for bias/ contamination allows an exception for common contaminants. This exception does not seem applicable to most analyses, explosives, for instance. This exception should be removed for all analyses except perhaps volatile organic compounds and semi-volatile organic compounds. Even in these instances, blank contamination for analytes of interest may interfere with project goals. The laboratory should be made aware of any potential problem analytes and the laboratory should be careful to minimize contamination of blanks and samples. Note that VOC criteria listed in Worksheet 28 is different, and more acceptable: "No target analytes ≥ ½ QL; with the exception of common lab contaminants which should be < RL."

Response: Agree. Note, however, that volatiles and semivolatiles (with the exception of the PAH subset of semivolatiles) are not included in the analysis for the two skeet ranges and the Machine Gun Boresight Range. The laboratory will be made aware of any potential problem analytes.

54. **Comment:** Worksheet 12 page 28; Accuracy/ Bias MPC of 50-120% recovery for nitrocellulose should be reconsidered. Limits should be closer to 78–105% acording to *A Reproducible Method For Determination Of Nitrocellulose In Soil* by Denise K. MacMillan, Chelsea R. Majerus, Randy D. Laubscher, John P. Shannon, Available online at www.sciencedirect.com.

Response: Nitrocellulose has been removed from analysis (see the Navy's response to MEDEP Comment #8.).

55. **Comment:** Worksheet 12 page 35; Accuracy/ Bias MPC of 30-130% recovery for nitrocellulose should be reconsidered. Limits should be closer to 78–105% according to *A Reproducible Method For Determination Of Nitrocellulose In Soil* by Denise K. MacMillan,

Chelsea R. Majerus, Randy D. Laubscher, John P. Shannon, Available online at www.sciencedirect.com.

Response: Nitrocellulose has been removed from analysis (see the Navy's response to MEDEP Comment #8.).

56. **Comment:** Worksheet 15 page 60; Please clarify how method detection limits [MDL] and quantitation limits were derived. The definition for MDL given in section 1.6.2.3 does not correspond with the definition of MDL derived from 40 CFR Part 136 Appendix B.

Response: The text in Section 1.6.2.3 will be revised as follows:

"Sensitivity is the ability of the method or instrument to detect the target analytes at the level of interest. The quantitation limit *will be the low level calibration standard.* ~~is the mimimum concentration of an analyte that can be routinely identified and quantified above the method detection limit (MDL) by a laboratory.~~ The *Method Detection Limit (*MDL*)* is the minimum concentration of an analyte that can be ~~reliably distinguished from background noise for a specific analytical method~~ *reported with 99% confidence that the analyte concentration is greater than zero.*

57. **Comment:** Worksheet 15 page 64; Sensitivity for metals in surface water is not adequate for the project goal to evaluate whether or not the site qualifies for no further action. Another method, EPA SW846 method 6020, should be considered for metals in surface water.

Response: Agree. However, Method 6010B was scoped out for this field effort by Malcolm Pirnie and their planning documents had been through several review cycles, hence the method was carried forward. It is recommended to continue with Method 6010B for surface water and groundwater for the project because the selected laboratory does not have Method 6020 capabilities. To address the comment, however, the laboratory will perform a four times concentration on these samples, which will reduce the MDL and QLs. The revised aqueous MDLs and QLs are provided in revised Worksheet 15.

58. **Comment:** Worksheet 15, page 64, Metals (Surface Water); The project action limit (PALs) for antimony should be 5.5 µg/L based on the State Water Quality Criteria (SWQCs) for Human Health - consumption of water and organisms. Please revise.

Response: Agree.

59. **Comment:** Worksheet 15, page 66, PAHs (Surface Water); The PAL for benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i,)perylene, benzo(k)fluoranthene, chrysene, dibenzo(a,h)anthracene and indeno(1,2,3-cd)pyrene should be 0.003 µg/L based on the State Water Quality Criteria for Human Health - consumption of water and organisms. Please revise.

Response: Note that Tetra Tech was originally directed to use EA developed PALs for surface water/sediment regarding ecological values. MEDEP's request for revisions to PALs where State AWQC is more stringent will be accomplished if possible. Based on conversations with the analytical laboratory, the PALs for PAHs will be revised downward on Worksheet 15. Please note however that it is not technically feasible to attain the requested level of detection by any of the best available analytical technology.

60. **Comment:** Worksheet 15, page 67, Volatiles in Groundwater; Also 1,1-dichloroethane PAL should be 70 µg/L and 1,1,1-trichloroethane PAL should be 200 µg/L based on the State Maximum Exposure Guidelines (MEGs) (September 2007). Please revise.

    Response: VOCs are not included in the analytical suite for the two skeet ranges or Machine Gun Boresight Range. Worksheet #15 will be revised to reflect just those analytes that are relevant. Also, the PALs will be revised as necessary considering the change from Region 9 PRGs to the new regional screening levels (Oak Ridge).

61. **Comment:** Worksheet 15, page page 68, Volatiles in Surface Water; Based on the SWQCs for Human Health - consumption of water and organisms, please revise the following PALs:

1,1,2-trichloroethane	0.58 µg/L
1,1,2,2-tetrachloroethane	0.16 µg/L
1,2-dichloroethane	0.38 µg/L
1,2-dichloropropane	0.50 µg/L
chloroform	5.4 µg/L
methylene chloride	4.6 µg/L
tetrachloroethane	0.58 µg/L
Trans-1,2-dichloroethene	140 µg/L
trichloroethene	2.37 µg/L
Vinyl chloride	0.025 µg/L

Page10 of 12

    Response: VOCs are not included in the analytical suite for the two skeet ranges or Machine Gun Boresight Range. Worksheet #15 will be revised to reflect just those analytes that are relevant.

62. **Comment:** Worksheet 15, page 71,Semivolatiles in Surface Water; Based on the SWQCs for Human Health - consumption of water and organisms please revise the following PALs:

2,6-dinitrotoluene	2.11 µg/L
3,3-dichlorobenzidine	0.013 µg/L
hexachlorobenzene	0.0002 µg/L
hexachlorobutadiene	0.43 µg/L
hexachloroethane	1.04 µg/L
isophorone	35 µg/L
nitrobenzene	16.7 µg/L
n-nitrosodiphenylamine	2.23 µg/L

n-nitro-di-n-propylamine	0.005 µg/L
Pentachlorophenol	0.25 µg/L

Response: With the exception of 2,6-dinitrotoluene, the listed analytes are not included in the analytical suite for the two skeet ranges or Machine Gun Boresight Range. For 2,6-dinitrotoluene, soil samples will be collected for this analyte at the two skeet ranges (at the firing points) but no surface water samples will be collected for this analyte. Worksheet #15 will be revised to reflect just those analytes that are relevant.

63. **Comment:** Worksheet 15, page 73, Pesticides/PCBs (Groundwater);

a.) Lindane has an MEG of 0.2ppb which is the total of the all the Lindane or HCH parent and breakdown products. Please add a footnote regarding the totaling.
b.) Endosulfan I, Endosulfan II, and Endosulfan Sulfate are all breakdown products of Endosulfan and for comparing to the MEGs must be totaled. Please add a footnote regarding the totaling and comparing to the MEG of 42 µg/L.
c.) Endrin, Endrin Aldehyde, and Endrin Ketone must also be added and compared to the MEG of 2 µg/L.
d.) The Aroclors must also be totaled and compared to the PCB MEG of 0.5 µg/L.

Response: Pesticides/PCBs are not included in the analytical suite for the two skeet ranges or Machine Gun Boresight Range. Worksheet #15 will be revised to reflect just those analytes that are relevant. Also, the PALs will be revised as necessary considering the change from Region 9 PRGs to the new regional screening levels (Oak Ridge).

64. **Comment:** Worksheet 15, page 74, Pesticides/PCBs (Surface Water;: Based on the State Water Quality Criteria for Human Health - consumption of water and organisms please revise the following PALs:

4,4-DDD	0.00017 µg/L
4,4-DDE	0.00012 µg/L
4,4-DDT	0.00012 µg/L
Aldrin	0.000027 µg/L
Alpha-BHC	0.0017 µg/L
Beta-BHC	0.006 µg/L
dieldrin	0.00019 µg/L
Endosulfan sulfate	39 µg/L
endrin	0.032 µg/L
Endrin aldehyde	0.16 µg/L
Gamma-BHC (Lindane)	0.68 µg/L
heptachlor	0.000043 µg/L
heptachlorepoxide	0.000021 µg/L
toxaphene	0.000015 µg/L
Total aroclors (PCBs)	0.000035 µg/L

Response: Pesticides/PCBs are not included in the analytical suite for the two skeet ranges or Machine Gun Boresight Range. Worksheet #15 will be revised to reflect just those analytes that are relevant.

65. **Comment:** Worksheet 15, page 75, Explosives (Soil); Please revise the 2,6 dinitrotoluene to 0.72mg/kg per the Region 9 limits for dinitrotoluene mixture.

Response: The PAL will be revised to 0.71 mg/kg on revised Worksheet 15 as per the new regional screening levels (Oak Ridge).

66. **Comment:** QAPP Worksheet #19, page 93 and Attachment 1-B, SOP S14 CRREL TR-02-1, Appendix E Sample Preservation; Based on the information in the appendix, TNT and related compounds may be subject to degradation after 1-2 days without preservation. MEDEP suggests that due to the likelihood of microbial activity in the surface water at Site 12 and the Quarry, that the samples for nitroaromatics be preserved as suggested in the guidance to reduce potential degradation.

Response: Nitroaromatics analysis for surface water is not included in the analytical suite for the two skeet ranges or Machine Gun Boresight Range. However, the comment does apply to the MEC sites and will be addressed as part of that subject future Work Plan.

67. **Comment:** Worksheet 23 page 103; Analytical SOP References Table lists the Emperical SOP-105 Analytical Group as *mercury*. This should be changed to *metals*. Also, the most current version of EPA SW846 ICP method for metals is 6010C.

Response: The typographical errors (TAL Metals and mercury were switched) have been corrected in Worksheet 19. The Navy agrees that the most current version of 6010 is revision C. When this work plan was prepared and the laboratory was procured, Method 6010B was the most current revision, hence that is the method that was written into the plan. Since that time the laboratory can provide revision C, so the work plan will be updated.

68. **Comment:** Worksheet 28 page 115 and all other worksheets where statistically derived measurement performance criteria are listed: Measurement performance criteria [MPC] for accuracy/ bias need to be site specific. The laboratory derived limits may or may not be applicable to site requirements.

Response: The laboratory derived limits have been evaluated and are appropriate for this site. The limits are attached to this comment response letter for your perusal.

69. **Comment:** Worksheet 28 page 123: Please define LODV and add to acronym list.

Response: LODV is "Limit of detection verification." The definition will be added to Worksheet 28 and the acronym list.

Attachment 1-B

70. Comment: SOP S3, Soil Sampling:

a.) The sample methodologies for VOCs are "crossed out", they must be retained for the Quarry investigation.

b.) MEDEP recommends collecting both methanol-preserved and freeze-preserved or sodium-bisulphate preserved samples to ensure adequate sample volume is available.

Response: References to the Site 12 EOD Area, an MEC site, will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites. Therefore, references to VOC analyses will also be removed.

71. Comment: SOP S12, Surface Water and Sediment Sampling:

a.) The sections for pond sampling are "crossed out" and must be retained in the SOP for Site 12 and the Quarry.

Response: References to the Site 12 EOD Area and Quarry, MEC sites, will be removed from the subject SI MC Work Plan and will be addressed in a separate future SI MC Work Plan submittal for MEC sites. However, the comment still applies to the NASB Skeet Range and will be retained in the SOP.

b.) For sampling surface water at depths MEDEP strongly suggests the Kemmerer sampler.

Response: Agree. The Kemmerer sampler for surface water sampling will be incorporated into the sampling plan.

c.) MEDEP also strongly suggests the petite ponar dredge sampler for sampling sediment at depth.

Response: Agree. The petite ponar dredge sampler for sediment sampling will be incorporated into the sampling plan.

General Comment: Additional references cited or reviewed by MEDEP;

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

ERDC/CRREL TR-07-11 Environmental Assessment of Lead at Camp Edwards, Massachusetts Small Arms Ranges, Dated May 4, 2007 for the Massachusetts Army National Guard.

Explosives Investigation and Technology Workshop training document for February 12-14 2008 short-course.

ERDC/CRREL TR-06-18 Conceptual Model for the Transport of Energetic Residues from Surface Soil to Groundwater by Range Activities, November 2006.

Response: No response necessary.

Empirical Laboratories, LLC **LCS Accuracy/Precision Limits**

Explosives 8330A	Water LCS Limits	Water RPD Limits	Soil LCS Limits	Soil RPD Limits
1,3,5-Trinitrobenzene	65-140	30	75-125	30
1,3-Dinitrobenzene	45-160	30	80-125	30
2,4,6-Trinitrotoluene	50-145	30	55-140	30
2,4-Dinitrotoluene	60-135	30	80-125	30
2,6-Dinitrotoluene	60-135	30	80-120	30
2-Amino-4,6-dinitrotoluene	50-155	30	80-125	30
2-Nitrotoluene	45-135	30	80-125	30
3-Nitrotoluene	50-130	30	75-120	30
4-Amino-2,6-dinitrotoluene	55-155	30	80-125	30
4-Nitrotoluene	50-130	30	75-125	30
HMX	80-115	30	75-125	30
Nitrobenzene	50-140	30	75-125	30
RDX	50-160	30	70-135	30
Tetryl	20-175	30	10-150	30
Nitroglycerin (8332)	60-120	30	70-135	30
Nitrocellulose (LAB)	50-120	30	30-130	30
1,2-Dinitrobenzene(surr)	40-145		55-140	

Empirical Laboratories, LLC **LCS Accuracy/Precision Limits**

PAHs Low Level 8270C	Water LCS Limits	Water RPD Limits	Soil LCS Limits	Soil RPD Limits
Naphthalene	30-115	30	40-105	30
2-Methylnaphthalene	35-115	30	45-105	30
1-Methylnaphthalene	35-115	30	45-105	30
Acenaphthylene	40-115	30	45-105	30
Acenaphthalene	35-120	30	45-110	30
Fluorene	40-120	30	50-110	30
Phenanthrene	40-130	30	50-110	30
Anthracene	45-120	30	55-105	30
Fluoranthene	45-125	30	55-115	30
Pyrene	35-140	30	45-125	30
Benzo(a)anthracene	45-120	30	50-110	30
Chrysene	45-120	30	55-110	30
Benzo(b)fluoranthene	35-130	30	45-115	30
Benzo(k)fluoranthene	30-135	30	45-125	30
Benzo(a)pyrene	45-120	30	50-110	30
Indeno(1,2,3-cd)pyrene	30-140	30	40-120	30
Dibenz(a,h)anthracene	30-140	30	40-125	30
Benzo(g,h,i)Perylene	25-135	30	40-125	30
Nitrobenzene-d5(surr)	30-110		30-110	
2-Fluorobiphenyl(surr)	35-110		35-110	
Terphenyl-d14(surr)	55-125		40-120	

Empirical Laboratories, LLC **LCS Accuracy/Precision Limits**

Metals 6010B	Water LCS Limits	Water RPD Limits	Soil LCS Limits	Soil RPD Limits
Aluminum	80-120	20	80-120	20
Antimony	80-120	20	80-120	20
Arsenic	80-120	20	80-120	20
Barium	80-120	20	80-120	20
Beryllium	80-120	20	80-120	20
Cadmium	80-120	20	80-120	20
Calcium	80-120	20	80-120	20
Chromium	80-120	20	80-120	20
Cobalt	80-120	20	80-120	20
Copper	80-120	20	80-120	20
Iron	80-120	20	80-120	20
Lead	80-120	20	80-120	20
Mercury	80-120	20	80-120	20
Magnesium	80-120	20	80-120	20
Manganese	80-120	20	80-120	20
Nickel	80-120	20	80-120	20
Potassium	80-120	20	80-120	20
Selenium	80-120	20	80-120	20
Silver	80-120	20	80-120	20
Sodium	80-120	20	80-120	20
Thallium	80-120	20	80-120	20
Zinc	80-120	20	80-120	20

CAS

LCS Accuracy/Precision Limits

	Water LCS Limits	Water RPD Limits		
Perchlorate 6850	80-120	15		

Empirical TCLP Water Limits

Semivolatiles 8270C	Limits	
2,4-Dinitrotoluene	50-120	
Hexachlorobenzene	50-110	
Hexachlorobutadiene	25-105	
Hexachloroethane	30-95	
2-Methylphenol	40-110	
4-Methylphenol	30-110	
Nitrobenzene	45-110	
Pentachlorophenol	40-115	
Pyridine	10-110	
2,4,5-Trichlorophenol	50-110	
2,4,6-Trichlorophenol	50-115	
Volatiles 8260B		
Benzene	80-120	
2-Butanone	30-150	
Carbon Tetrachloride	65-140	
Chlorobenzene	80-120	
Chloroform	65-135	
1,4-Dichlorobenzene	75-125	
1,2-Dichloroethane	70-130	
1.1-Dichloroethene	70-130	
Tetrachloroethene	45-150	
Trichloroethene	70-125	
Vinyl chloride	50-145	
Pesticides 8081A		
Endrin	55-135	
Gamma-BHC	25-135	
Heptachlor	40-130	
Heptachlor epoxide	60-130	
Methoxychlor	55-150	
Chlordane	45-119	

Toxaphene	41-126	
Herbicides 8151		
2,4-D	35-115	
Silvex	50-115	

D-5 MAY 2009 RESPONSE TO STAKEHOLDER FOLLOW-UP COMMENTS

Klink, Linda

From: Sait, Claudia B [Claudia.B.Sait@maine.gov]
Sent: Friday, May 29, 2009 11:16 AM
To: Carol G. Warren; Catherine Guido; Daly.Mike@epamail.epa.gov; DWChipman@Suscom-maine.net; Evans, Chris; sjohnson@me-law.com; lepage, carolyn; Klink, Linda; michael.fagan1@navy.mil; paul.burgio@navy.mil; Scott Libby ; todd.bober@navy.mil; victoriab@mrra.us
Cc: Van Dercook, Amy L CIV NAVFAC Atlantic
Subject: MMRP MC SI Workplan
Attachments: MMRP_3-09MCSIWPso_5-29-09.pdf

Todd,

Attached is MEDEP sign off on the follow up RTCs for the MC site investigation for the 6 Munitions Response Site. Please contact me if you have any issues or concerns.

Claudia Sait
Maine Department of Environmental Protection
Federal Facilities Unit
(207) 287-7713
claudia.b.sait@maine.gov

STATE OF MAINE
DEPARTMENT OF ENVIRONMENTAL PROTECTION

JOHN ELIAS BALDACCI
GOVERNOR

DAVID P. LITTELL
COMMISSIONER

May 29, 2009

Mr. Todd Bober
Department of Navy
Base Realignment and Closure
Program Management Office-Northeast
4911 South Broad Street
Philadelphia, PA 19112-1303

Re: Draft Final Site Inspection Workplan for Munitions Constituents
At Six Munitions Response Sites
Naval Air Station, Brunswick, Maine

Dear Mr. Bober:

Pursuant to Section VI of the Naval Air Station, Brunswick, Maine Federal Facility Agreement (Oct 1990), as amended, the Maine Department of Environmental Protection (MEDEP) has reviewed the Navy's responses received via email on May 21, 2009, to MEDEP's comments, dated April 6, 2009, for revised draft final "Site Inspection (SI) Work Plan, Munitions Constituents at Six Munitions Response Sites", February 2008), prepared by Tetra Tech NUS.
Based on that review, MEDEP has no further comments provided that:

- Once the soil data is available the groundwater sampling requirements and locations will be discussed;
- And the proposed revisions and additions are incorporated into the final report along with regulator comments, responses and this letter.

Please contact me at (207) 287-7713 or claudia.b.sait@maine.gov, if you have any questions or comments.

Respectfully,

Claudia Sait

Claudia Sait
Project Manager-Federal Facilities
Bureau of Remediation & Waste Management

Cf: Hard copy
File
Ed Benedikt

Electronic Copy
Chris Evans-MEDEP
Carol Warren
Catherine Guido-ECC
Vicki Boundy-MRRA
Mike Fagan-BNAS
Mike Daly-EPA
Linda Klink – Tetra Tech NUS
David W. Chipman
Susanne Johnson
Scott Libby
Carolyn Lepage-Lepage Environmental

AUGUSTA
17 STATE HOUSE STATION
AUGUSTA, MAINE 04333-0017
(207) 287-7688 FAX: (207) 287-7826
RAY BLDG., 28 TYSON DR.

BANGOR
106 HOGAN ROAD
BANGOR, ME 04401
(207) 941-4570 FAX: (207) 941-4584

PORTLAND
312 CANCO ROAD
PORTLAND, ME 04103
(207) 822-6300 FAX: (207) 822-6303

PRESQUE ISLE
1235 CENTRAL DRIVE, SKYWAY PARK
PRESQUE ISLE, ME 04769-2094
(207) 764-0477 FAX: (207) 764-1507

web site: www.maine.gov/dep

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Klink, Linda

From: Daly.Mike@epamail.epa.gov
Sent: Wednesday, May 27, 2009 3:19 PM
To: Klink, Linda
Cc: Race, Charles; Sait, Claudia B; Orient, Jeff; 'Fagan, Michael CIV NAS Brunswick, Environmental'; Blanken, Michelle; Burgio, Paul CIV OASN (I&E) BRAC PMO NE; Bober, Todd A CIV NAVFAC MIDLANT
Subject: Re: Navy's response to MEDEP and EPA follow-up comments, MC Work Plan Skeet Ranges and MGBR, NASB, CTO 69

Hi Linda,

I've reviewed the RTCs on the subject document and have no further comments.

Thanks for addressing EPA's comments!

Thanks,

Mike

"Klink, Linda" <Linda.Klink@tetratech.com>

05/21/2009 04:46 PM

To
"Sait, Claudia B" <Claudia.B.Sait@maine.gov>, Mike Daly/R1/USEPA/US@EPA

cc
"Bober, Todd A CIV NAVFAC MIDLANT" <todd.bober@navy.mil>, "Burgio, Paul CIV OASN (I&E) BRAC PMO NE" <paul.burgio@navy.mil>, "Orient, Jeff" <Jeff.Orient@tetratech.com>, "Race, Charles" <Charles.Race@tetratech.com>, "Blanken, Michelle" <Michelle.Blanken@tetratech.com>, "'Fagan, Michael CIV NAS Brunswick, Environmental'" <michael.fagan1@navy.mil>

Subject
Navy's response to MEDEP and EPA follow-up comments, MC Work Plan Skeet Ranges and MGBR, NASB, CTO 69

Hi, Claudia and Mike,

We would appreciate it if you could look over the Navy's attached response to your follow-up comments on the MC Work Plan., Todd has spoken with Claudia on the major points so I think we are good to go but want to make sure before finalizing the document., Thank you.

By the way, I am out of the office tomorrow through next week on vacation.

[attachment "for MEDEP&EPA review MC SI WP followupRTC_052109.doc" deleted by Mike Daly/R1/USEPA/US]

Klink, Linda

From: Klink, Linda
Sent: Thursday, May 21, 2009 4:46 PM
To: 'Sait, Claudia B'; Daly.Mike@epamail.epa.gov
Cc: 'Bober, Todd A CIV NAVFAC MIDLANT'; Burgio, Paul CIV OASN (I&E) BRAC PMO NE; Orient, Jeff; Race, Charles; Blanken, Michelle; 'Fagan, Michael CIV NAS Brunswick, Environmental'
Subject: Navy's response to MEDEP and EPA follow-up comments, MC Work Plan Skeet Ranges and MGBR, NASB, CTO 69

Hi, Claudia and Mike,

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for MEDEP&EPA review MC SI WP ...

RESPONSE TO USEPA FOLLOW-UP COMMENTS VIA e-MAIL DATED APRIL 13, 2009
REVISED DRAFT FINAL SITE INSPECTION WORK PLAN, MUNITIONS CONSTITUENTS AT SIX MUNITIONS RESPONSE SITES, DATED FEBRUARY 2008
NAVAL AIR STATION BRUNSWICK, MAINE

Response to EPA Comment #2: EPA concurs with the recommendation to defer ground water investigations at the skeet ranges until the XRF & soil investigation portion of the SI is completed and the data are evaluated to determine if and where ground water sample collection should be conducted.

Follow-up Response: No response necessary.

Response to EPA Comment #9: A important aspect of whether a calibration procedure is equivalent to EPA and the NAS Brunswick Base-Wide LTM QAPP procedures is that it uses, at a minimum, two standards to bracket the measurement range. The only exception to this is the ORP measurement which uses one standard (Zobell standard). If TetraTech uses a field instrument and it's manufacturer's calibration procedure does not meet this basic requirement, the manufacturer's procedure is not acceptable. Please provide the specific field instrumentation that will be used and whether the manufacturers' calibration procedures meet this basic SOP requirement.

Follow-up Response: Tetra Tech typically rents a YSI 650 MDS handheld/date logger with flow-through cell. It measures pH, ORP, specific conductance, temperature, and dissolved oxygen. Turbidity is measured with a LaMotte Turbidity meter. The calibration procedure for all parameters except ORP, will use two standards. The QAPP will be clarified accordingly.

Response to MEDEP Comment #7: While EPA believes it is reasonable to filter perchlorate samples in a lab environment instead of in the field at the time of collection, this step should be completed as soon as reasonably possible. EPA recommends that samples be filtered within 24 hours of field collection. The step of sterilely filtering the sample using a 0.2 micron filter is done to remove potential perchlorate-degrading microbes from the sample. Please provide EPA with more specifics about when sample filtering will be conducted.

Follow-up Response: Agree. The plan will be revised to clearly state that samples will be shipped within 12 hours of sample collection and the laboratory will filter immediately upon receipt, which is targeted to occur within 24 hours of sample collection.

Response to MEDEP Comment #20: Based on the site conceptual model for the skeet ranges, EPA agrees with MEDEP that XRF sampling should target the first 2 or 3 inches of surface soil. Based on the review of XRF field data for this top soil horizon, the Navy should target deeper soil horizons down to 1 foot in depth at select locations to determine if the lead contamination exists below this top layer. EPA also agrees with MEDEP that a conference call could be convened to discuss the XRF sampling design specifics to resolve any questions before the workplan is finalized.

Follow-up Response: For the two skeet ranges, the Navy agrees to collect surface soil samples from the 0 to 3-inch interval and subsurface soils from the 3- to 12-inch interval. Should any XRF samples from the 3- to 12-inch interval exceed 375 mg/kg for lead, an additional subsurface soil from the 12 to 18-inch interval will additionally be collected.

RESPONSE TO MEDEP FOLLOW-UP COMMENTS VIA e-MAIL DATED APRIL 6, 2009
REVISED DRAFT FINAL SITE INSPECTION WORK PLAN, MUNITIONS CONSTITUENTS AT SIX MUNITIONS RESPONSE SITES, DATED FEBRUARY 2008
NAVAL AIR STATION BRUNSWICK, MAINE

Of note, in addition to addressing the MEDEP follow-up comments below, the Navy would like to clarify its original response to MEDEP Comment #67 concerning the most current version of metals analysis. Method 6010B is planned (with the four times concentration described in the response to MEDEP #57). The most current version of Method 6010 is now 6010C. The project analytical laboratory, Empirical, is capable of but not approved by the Navy for 6010C and so the work plan will retain 6010B.

Response to EPA Comment #2, Groundwater: While EPA did agree to sampling groundwater only as necessary, MEDEP did not. Groundwater sampling must be performed to ensure there is no question of the former skeet ranges impacting groundwater. Please revise or delete this statement.

Follow-up Response: Comment acknowledged. The Navy will discuss potential groundwater sampling requirements and locations with the EPA and MEDEP after conducting site reconnaissance and soil sampling activities.

Response to EPA Comment #2, Groundwater, para 3&4: Site 9 is located north of the former machine gun bore sight range and the skeet ranges. Groundwater flow direction while locally controlled by the impoundment ponds is toward the north (toward the impoundment pond), northeast and southeast toward the lower impoundment pond and the unnamed tributary. Please correct the text and figures accordingly.

Follow-up Response: The Machine Gun Boresight Range response text will be corrected to read: "Based on data from Site 9 located less than ¼ mile to the ~~south~~ *north...*"

The Machine Gun Boresight Range and Skeet Range text will be revised to read: "Based on adjacent Site 9 data, groundwater flow direction is presumed to be ~~to the southeast~~ *generally to the north toward the impoundment ponds while locally controlled, then probably shifting to the northeast toward the west arm of Picnic Pond; further to the south, groundwater flow direction is expected to shift to the southeast toward Merriconeag Stream.*"

Response to EPA Comment #4, para1: MEDEP agrees with MW-NASB-72, -75, and -77 but rather than MW-NASB-76, if not already included in the workplan, MEDEP suggests MW-NASB-74, -78 and -320 and -82.

Follow-up Response: MW-NASB-76 and -78 are existing monitoring wells slated for groundwater sampling and so will need to be included in the synoptic water-level gauging. Considering the complex hydrogeology, as influenced by local effects, the Navy agrees to add NW-NASB-74, -320, and -82 to the list of wells for synoptic water-level gauging.

Response to EPA Comment #4, para2: While EPA did agree to sampling groundwater only as necessary, MEDEP did not. Groundwater sampling must be performed to ensure there is no question of the former skeet ranges impacting groundwater. Please revise or delete this statement.

Follow-up Response: Comment acknowledged. The Navy will discuss potential groundwater sampling requirements and locations with the EPA and MEDEP after conducting site reconnaissance and soil sampling activities.

Response to MEDEP Comment #4 & 11: In RTC 4 the Navy agrees to use an XRF to determine where to collect discrete samples; this contradicts RTC 11 where the Navy agrees to relocate specific sampling locations for the NAS-B skeet range. This contradiction needs to be resolved. (Also an XRF sample for a range is typically a composite from within the gridded section and the lab sample would be from that same composite.)

Follow-up Response: Soil samples will be collected as per the sampling scheme, relative to the skeet range location shown on the figures; the sample locations shown provide full site coverage of the shotfall zone and firing point. However, it may become apparent during XRF sampling, for example, that the actual skeet range location is not as depicted on the figures and so the sampling locations may be shifted as necessary while still maintaining the same coverage.

Response to MEDEP Comment #8, Bullet 5: The response for the dibutylphthalate is acceptable for the SI however if other contaminants of concern are found at levels of concern then it will be necessary to include dibutylphthalate in the Remedial Investigation.

Follow-up Response: Agree. If warranted, dibutylphthalate will be included in the future during an RI and/or remediation.

Response to MEDEP Comment #8, para 6: While EPA did agree to sampling groundwater only as necessary, MEDEP did not. Groundwater sampling must be performed to ensure there is no question of the former skeet ranges impacting groundwater. Please revise or delete this statement.

Follow-up Response: Comment acknowledged. The Navy will discuss potential groundwater sampling requirements and locations with the EPA and MEDEP after conducting site reconnaissance and soil sampling activities.

Response to MEDEP Comment #15.a: This is acceptable if there is no evidence of fill or other historic soil disturbance in the area.

Follow-up Response: Agree. Lithology will be recorded during the field effort.

Response to MEDEP Comment #20 & 33: At the Naval Air Station, the Navy will need to concentrate on the areas where the two shot fall fans overlap and will likely need more than the minimum 30 soil samples proposed due to the larger area to be covered.

It is not clear from the RTC, Section 4.7.4 of the QAPP (Appendix A), or the XRF SOP how the soil is to be collected and while Section 4.7.1 of the QAPP is cited in paragraph 6 of the response this section is applicable to direct push soil sampling not XRF. Please provide specific information on the soil collection including soil sampling depths (discussed in RTC 37.c, but still

to be resolved), grid size, and whether the sample will be discrete or composite, and the number of samples per composite.

At another Navy site where XRF was used extensively a composite was collected and field homogenized for each location, and an aliquot taken for XRF with the remainder held in reserve until fixed lab samples were selected. The number of readings per sample on the XRF must also be determined.

Follow-up Response: The Navy's response follows (changes will be incorporated into the revised document):

- As stated in the Navy's initial response to Comment 33, the revised text notes that 30 to 90 XRF samples are planned. The Navy agrees that the number of samples will be on the upper end for the NASB Skeet Range, considering the changed orientation over the years resulting in a larger area. The Navy also agrees to concentrate on the overlap area of the two shotfall fans.
- The XRF soil samples will be discrete in order to "match" the discrete sampling methodology for the samples to be sent to the fixed-base analytical laboratory.
- Soil samples collection depths will be changed as requested. Samples will be collected from the 0- to 3-inch interval and 3- to 12-inch interval. Should any XRF samples from the 3- to 12-inch interval exceed 375 mg/kg for lead, an additional subsurface soil from the 12 to 18-inch interval will additionally be collected. Because the soil samples are now so shallow, a trowel or hand auger will be utilized rather than a drill rig.
- Since the contaminant of concern is lead and the project action level is 375 mg/kg, a handheld XRF is sufficiently accurate. Three measurements per aliquot are planned and the average of the three recorded values will be calculated and reported along with the three measurements.

Response to MEDEP Comment #33, para 8: In order to get a good correlation between the XRF and fixed lab lead concentrations, an adequate number and range of lead concentrations must be analyzed by the lab, not just the highest. Therefore, a cross section of concentrations from XRF samples must be sent to the lab for analyses. Based on MEDEP experience at other sites, the XRF and fixed lab lead correlations should achieve greater than a 0.80 correlation coefficient, depending upon the range of data values. The evaluation needs to consider whether the correlation data set requires (for example) a high and low range conversion, or has outliers in the dataset if the linear correlation is between 0.65 and 0.80.

Follow-up Response: The Navy and regulators previously agreed we have an adequate number of set locations for fixed based analysis as shown on the sampling figures; the XRF samples will confirm the skeet range location and will provide supplemental data. Each soil sample location on the figure will have an associated XRF sample(s). Each soil sample location analyzed by the fixed-based lab will be as per the figures to address full coverage of the firing point and shotfall zone as shown on the figures. Although maximum concentrations for lead during the XRF effort will aid in defining the shotfall zone of the skeet ranges, the relative locations of the samples shown on the figures will be the basis for the fixed based laboratory samples. The fixed based laboratory will stand on its own and XRF data is considered supplemental data and will be added to the data set only if there is a good correlation.

Response to MEDEP Comment #37.c: Without evidence that the area has been disturbed the pellets are, as stated in the RTC, likely to be on the surface of the soil and therefore taking a 6" deep sample dilutes the concentration. MEDEP stands by its comment that the sample should not be more than 0-3" however since an XRF is going to be utilized, a number of stratified

samples could be taken to determine if contamination lies within the 0-6" interval. Soil samples could be collected in the most likely places and samples collected 0-3", 3-6" 6-9" and 9-12". This will answer whether the skeet ranges have been disturbed and the lead is deeper than expected.

If this is done then MEDEP assures the Navy it will accept 0-3" as surface for future risk assessment purposes.

Follow-up Response: Soil samples collection depths will be changed as requested. Samples will be collected from the 0- to 3-inch interval and 3- to 12-inch interval. Should any XRF samples from the 3- to 12-inch interval exceed 375 mg/kg for lead, an additional subsurface soil from the 12 to 18-inch interval will additionally be collected.

Response to MEDEP Comment #44: This response is acceptable as long as the Accuracy/Precision Limits attached as part of the RTCs are the nominal acceptance limits referenced.

Follow-up Response: Correct. The Accuracy/Precision Limits table attached to the comment response letter are the nominal acceptance limits referenced.

Response to MEDEP Comment #45: This response is acceptable as long as the field parameters are part of the analytical data to be supplied with the Environmental Data Deliverable.

Follow-up Response: Agree.

Response to MEDEP Comment #52: This response conflicts with RTC 44. MEDEP accepts RTC 44 as long as the nominal acceptance limits are clarified. Please revise as necessary.

Follow-up Response: Agree. The Accuracy/Precision Limits table attached to the comment response letter are the nominal acceptance limits.

Response to MEDEP Comment #57: This response is acceptable as long as the revised MDL meet the necessary screening levels.

Follow-up Response: Using the four times concentration described in the response to MEDEP #57, MDLs achievable by the analytical laboratory, Empirical, are much improved. However, there are four metals that still do not meet the PALs, including arsenic [PAL=0.045 ug/L; MDL=0.75 ug/L], cobalt [PAL=1.1 ug/L; MDL=5 ug/L], mercury [PAL=0.063 ug/L; MDL=0.08 ug/L], and thallium [PAL=0.5 ug/L; MDL=0.75 ug/L].