

**Quality Assurance Project Plan**  
for  
**Phase III Investigation**  
for  
**Area A Wetland - Site 2B**  
  
**Naval Submarine Base**  
**New London**  
Groton, Connecticut



**Naval Facilities Engineering Command**  
**Mid-Atlantic**

**Contract Number N62467-04-D-0055**

**Contract Task Order 439**

October 2007

QUALITY ASSURANCE PROJECT PLAN  
FOR  
PHASE III INVESTIGATION  
FOR  
AREA A WETLAND - SITE 2B

NAVAL SUBMARINE BASE - NEW LONDON  
GROTON, CONNECTICUT

COMPREHENSIVE LONG-TERM  
ENVIRONMENTAL ACTION NAVY (CLEAN) CONTRACT

Submitted to:  
Naval Facilities Engineering Command  
Mid-Atlantic  
9742 Maryland Avenue  
Norfolk, Virginia 23511-3095

Submitted by:  
Tetra Tech NUS, Inc.  
234 Mall Boulevard, Suite 260  
King of Prussia, Pennsylvania 19406

CONTRACT NUMBER N62467-04-D-0055  
CONTRACT TASK ORDER 439

OCTOBER 2007

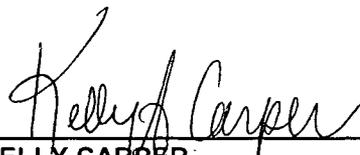
PREPARED UNDER DIRECTION OF:



---

AARON BERNHARDT  
PROJECT MANAGER  
TETRA TECH NUS, INC.  
PITTSBURGH, PENNSYLVANIA

APPROVED FOR SUBMISSION BY:



---

KELLY CARPER  
QUALITY ASSURANCE MANAGER  
TETRA TECH NUS, INC.  
PITTSBURGH, PENNSYLVANIA

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE NO.</u>
ACRONYMS.....	vi
<b>1.0 PROJECT MANAGEMENT AND OBJECTIVES .....</b>	<b>1-1</b>
1.1 DOCUMENT FORMAT.....	1-1
1.1.1 Document Control Format.....	1-1
1.1.2 Document Control Numbering System.....	1-2
1.1.3 QAPP Identifying Information.....	1-2
1.2 DISTRIBUTION LIST AND PROJECT PERSONNEL SIGN-OFF SHEET.....	1-2
1.2.1 Distribution List.....	1-2
1.2.2 Project Personnel Sign-Off Sheet.....	1-2
1.3 PROJECT ORGANIZATION.....	1-2
1.3.1 Communication Pathways.....	1-3
1.3.2 Personnel Responsibilities And Qualifications.....	1-3
1.3.3 Special Training Requirements And Certifications.....	1-3
1.4 PROJECT PLANNING AND PROBLEM DEFINITION.....	1-3
1.4.1 Project Planning.....	1-3
1.4.2 Site History, Background, and Problem Definition.....	1-4
1.5 PROJECT QUALITY OBJECTIVES AND MEASUREMENT PERFORMANCE CRITERIA.....	1-15
1.5.1 Development of Project Quality Objectives Using the Systematic Planning Process.....	1-15
1.5.2 Measurement Performance Criteria.....	1-15
1.6 SECONDARY DATA EVALUATION.....	1-20
1.7 PROJECT OVERVIEW AND SCHEDULE.....	1-20
1.7.1 Project Overview.....	1-21
1.7.2 Project Schedule.....	1-21
<b>2.0 MEASUREMENT/DATA ACQUISITION.....</b>	<b>2-1</b>
2.1 SAMPLING TASKS.....	2-1
2.1.1 Sampling Process Design and Rationale.....	2-1
2.1.2 Sampling Procedures and Requirements.....	2-1
2.2 ANALYTICAL TASKS.....	2-3
2.2.1 Analytical SOPs.....	2-3
2.2.2 Analytical Instrument Calibration Procedures.....	2-3
2.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures.....	2-3
2.2.4 Analytical Supply Inspection and Acceptance Procedures.....	2-3
2.3 SAMPLE COLLECTION DOCUMENTATION, HANDLING, TRACKING, AND CUSTODY PROCEDURES.....	2-3
2.3.1 Sample Collection Documentation.....	2-4
2.3.2 Sample Handling and Tracking System.....	2-4
2.3.3 Sample Custody.....	2-5
2.4 QUALITY CONTROL SAMPLES.....	2-6
2.4.1 Sampling Quality Control Samples.....	2-6
2.4.2 Analytical Quality Control Samples.....	2-6

## TABLE OF CONTENTS

<u>SECTION</u>		<u>PAGE NO.</u>
2.5	DATA MANAGEMENT TASKS .....	2-7
2.5.1	Project Documentation and Records.....	2-7
2.5.2	Data Package Deliverables .....	2-7
2.5.3	Data Reporting Formats .....	2-8
2.5.4	Data Handling and Management.....	2-8
2.5.5	Data Tracking and Control .....	2-10
<b>3.0</b>	<b>ASSESSMENT AND OVERSIGHT.....</b>	<b>3-1</b>
3.1	ASSESSMENT AND RESPONSE ACTIONS .....	3-1
3.1.1	Planned Assessments.....	3-1
3.1.2	Assessment Findings and Corrective Action Responses.....	3-2
3.2	QA MANAGEMENT REPORTS .....	3-3
3.3	FINAL PROJECT REPORT .....	3-4
<b>4.0</b>	<b>DATA REVIEW .....</b>	<b>4-1</b>
4.1	OVERVIEW.....	4-1
4.2	DATA REVIEW STEPS.....	4-1
4.2.1	Step I: Verification .....	4-3
4.2.2	Step II: Validation .....	4-3
4.2.3	Step III: Data Usability Assessment .....	4-4
4.3	STREAMLINING DATA REVIEW.....	4-8
4.3.1	Data Review Steps to be Streamlined.....	4-8
4.3.2	Criteria for Streamlining.....	4-9
4.3.3	Amounts and Types of Data Appropriate for Streamlining.....	4-9
<b>REFERENCES.....</b>		<b>R-1</b>

### APPENDICES

<b>A</b>	<b>FIELD AND LABORATORY STANDARD OPERATING PROCEDURES</b>
<b>B</b>	<b>UFP-QAPP WORKSHEETS</b>
<b>C</b>	<b>UPDATED ECOLOGICAL RISK ASSESSMENT</b>
<b>D</b>	<b>SUMMARY OF AREA A LANDFILL MONITORING PROGRAM</b>
<b>E</b>	<b>LABORATORY STATEMENT OF WORK</b>

## TABLES

### NUMBER

- 1-1 Summary of Historic Surface Soil Samples
- 1-2 Summary of Historic Sediment Samples
- 1-3 Summary of Historic Surface Water Samples
- 1-4 Summary of Historic Biological Tissue Samples
- 2-1 Laboratory Data Package Elements

## FIGURES

### NUMBER

- 1-1 Site Location Map
- 1-2 Historical Soil and Sediment Sampling Locations
- 1-3 Historical Surface Water Sampling Locations
- 1-4 Proposed Sampling Locations

## ACRONYMS

%R	Percent Recovery
4,4'-DDD	1,1-dichloro-2,2-bis(4-chlorophenyl)ethane
4,4'-DDE	1,1-dichloro-2,2-bis(4-chlorophenyl)ethene
4,4'-DDT	1,1,1-trichloro-2,2-bis(4-chlorophenyl)ethane
BERA	Baseline Ecological Risk Assessment
BGOURI	Basewide Groundwater Operable Unit Remedial Investigation
CLEAN	Comprehensive Long-term Environmental Action Navy
CLP	Contract Laboratory Program
COC	Contaminant of Concern
CTDEP	Connecticut Department of Environmental Protection
CTO	Contract Task Order
DDTR	DDT and its derivatives (e.g., DDD, DDE)
DGI	Data Gap Investigation
DOD	Department of Defense
DQO	Data Quality Objective
DVM	Data Validation Manager
EDD	Electronic Data Deliverable
ERA	Ecological Risk Assessment
ESLs	Ecological Screening Levels
FFS	Focused Feasibility Study
FOL	Field Operations Leader
FS	Feasibility Study
FWENC	Foster Wheeler Environmental Corporation
GC	Gas Chromatography
GIS	Geographical Information System
HASP	Health and Safety Plan
HHRA	Human Health Risk Assessment
IDL	Instrument Detection Limit
IRA	Interim Remedial Action
LCS(D)	Laboratory Control Sample (Duplicate)
LIMS	Laboratory Information Management System
LQAP	Laboratory Quality Assurance Plan
MDLs	Method Detection Limits
MPCs	Measurement Performance Criteria
MS	Mass Spectroscopy

MS(D)s	Matrix Spike(s) (Duplicates)
NEBA	Net Environmental Benefits Analysis
NELAP	National Environmental Laboratory Accreditation Program
NFESC	Naval Facilities Engineering Service Center
NIRIS	Naval Installation Restoration Information Solution
NSB-NLON	Naval Submarine Base – New London
OBDA	Overbank Disposal Area
OSHA	Occupational Safety and Health Administration
PAHs	Polycyclic Aromatic Hydrocarbons
PARCCS	Precision, Accuracy, Representativeness, Comparability, Completeness, Sensitivity
PCBs	Polychlorinated Biphenyls
PDSs	Post-Digestion Spikes
PM	Project Manager
PQOs	Project Quality Objectives
PPE	Personal Protective Equipment
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
QLs	Quantitation Limits
QSM	Quality Systems Manual
RI	Remedial Investigation
RME	Reasonable Maximum Exposure
RPD	Relative Percent Difference
SDG	Sample Delivery Group
SERA	Screening Level Ecological Risk Assessment
SIM	Selected Ion Monitoring
SOP	Standard Operating Procedure
TAL	Target Analyte List
TCL	Target Compound List
TOC	Total Organic Carbon
TtNUS	Tetra Tech NUS, Inc.
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plan
USEPA	United States Environmental Protection Agency
U.S. Navy	United States Navy

## 1.0 PROJECT MANAGEMENT AND OBJECTIVES

This Quality Assurance Project Plan (QAPP) for the Phase III Investigation for Area A Wetland - Site 2B at the Naval Submarine Base – New London (NSB-NLON), Groton, Connecticut has been prepared by Tetra Tech NUS, Inc. (TtNUS) on behalf of the United States Navy (U.S. Navy) Naval Facilities Engineering Command Mid-Atlantic (NAVFAC Mid-Atlantic) under the Comprehensive Long-term Environmental Action Navy (CLEAN) Contract Number N62467-04-D-0055, Contract Task Order (CTO) 439. The QAPP contained herein was generated for and complies with applicable State of Connecticut, Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP), and United States Environmental Protection Agency (USEPA) Region I requirements, regulations, guidance, and technical standards.

The purpose of this QAPP is to ensure that data to be collected are of the correct type, quantity, and quality to support the attainment of project objectives. This QAPP outlines the organization, project management and objectives, planned activities, measurement/data acquisition (including field work), assessment/oversight, and data review procedures associated with the Phase III Investigation to be conducted at Area A Wetland – Site 2B at NSB-NLON in Groton, Connecticut.

The investigation procedures used comply with applicable TtNUS Standard Operating Procedures (SOPs), which are included in Appendix A of this QAPP.

Figure 1-1 is the site map for NSB – NLON, with the location of Area A Wetland – Site 2B denoted, and Figure 1-2 depicts the historical surface soil and sediment sampling locations. Historical surface water sampling locations for Area A Wetland – Site 2B can be found in Figure 1-3.

The field activities conducted under this QAPP shall meet the requirements of the Health and Safety Plan (HASP), which will be prepared as a separate document.

### 1.1 DOCUMENT FORMAT

#### 1.1.1 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. Text, tables, and figures in this QAPP include a header indicating the revision number and date. The footer indicates the page number within each section. Revision 0 with the month and year will be used as part of the header, for the draft, draft final, and final QAPP versions. If necessary, any revisions made after submittal of the final will be identified with appropriate revision number and date.

### **1.1.2 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).

### **1.1.3 QAPP Identifying Information**

The QAPP identifying information (found on the QAPP cover and signature pages) identifies the key project staff, previous site work, and the program for which the current project is being performed. The QAPP title and approval page is on Worksheet #1 and identifying information is provided on Worksheet #2. The UFP-QAPP worksheets are presented in Appendix B.

## **1.2 DISTRIBUTION LIST AND PROJECT PERSONNEL SIGN-OFF SHEET**

### **1.2.1 Distribution List**

The QAPP distribution list includes personnel from: Connecticut Department of Environmental Protection (CTDEP), USEPA Region I, U.S. Navy, U.S. Fish and Wildlife Service, and TtNUS. The distribution list for this QAPP is summarized in Worksheet #3. Each person listed in QAPP Worksheet #3 will receive a copy of this QAPP (Revision 0) and any subsequent revisions. Revisions may be in the form of a complete document reissue or individual pages that have been changed.

### **1.2.2 Project Personnel Sign-Off Sheet**

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

## **1.3 PROJECT ORGANIZATION**

An organization chart depicting the personnel involved with the project as it relates to the QAPP is shown in Worksheet #5. The lead agency for this site, the U.S. Navy, and their contractor, TtNUS, will implement this QAPP.

### **1.3.1 Communication Pathways**

Routes of communication have been established for the exchange of project-related information and alterations that may be required because of unforeseen or changing circumstances, including potential revisions to project methods and SOPs, schedules, requirements, etc. Communication pathways are depicted in QAPP Worksheet #6.

### **1.3.2 Personnel Responsibilities And Qualifications**

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

### **1.3.3 Special Training Requirements And Certifications**

All field personnel will have received the appropriate training required 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 CFR 1910.120(b)(4). As indicated on Worksheet #8, the only training requirements are Health-and-Safety-related and will be covered in the Health and Safety Plan.

Katahdin Analytical Services, Inc. (Katahdin) will perform the chemical analysis of sediment samples and has successfully completed the laboratory evaluation process required as part of the Naval Facilities Engineering Service Center (NFESC) Quality Assurance Program, as described in the Department of Defense Quality Systems Manual (DOD QSM) (January 2006). Katahdin is additionally certified by the National Environmental Laboratory Accreditation Program (NELAP).

## **1.4 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.

### **1.4.1 Project Planning**

Project planning meetings were conducted as needed to prepare the QAPP (refer to Worksheet #9 for a list of meetings and participants). The project problem definition and data quality objectives are outlined in Worksheet #10.

## 1.4.2 Site History, Background, and Problem Definition

### 1.4.2.1 Site History and Background

Area A Wetland is a 25.76-acre site located within NSB-NLON (see Figures 1-1 and 1-2). Figure 1-2 is an aerial photograph of the Area A Wetland. This portion of NSB-NLON remained undeveloped wooded land, and possibly wetland, until the late 1950s. Dredge spoils from the Thames River were then pumped into the area and contained within an earthen dike that extends from the Area A Landfill to the south side of the Area A Weapons Center. The total volume of dredged material in the wetlands is estimated to be 1.2 million cubic yards. A small pond is located in the southern portion of the wetland, and between one and three feet of standing water is present during all seasons. In the 1960s, pesticide "bricks" consisting of formulated (water-soluble) DDT were placed on the wetland ice during winter and allowed to dissolve as a mosquito control measure. These placements were made prior to the ban on DDT in 1972. The Area A Wetland discharges to the Area A Downstream Watercourses and subsequently into the Thames River. The levels of contaminants in the sediment prior to placement into the wetland are not known. The finer particulates, which typically have greater chemical concentrations than the coarser sediments, may have deposited in the area near the dike.

To the northwest of the Area A Wetland lies the Area A Weapons Center. This site consists of Building 524, which was primarily used for administration, minor torpedo assembly, storage of simulator torpedoes, and weapons storage in bunkers. Small quantities of chemicals and chemical wastes were generated by the activities in Building 524 and were stored in 1- to 5-gallon containers in seven metal storage cabinets located on a paved area to the south of the building. These chemicals included cleaning and lubricating compounds, paints, and adhesives, many of which were classified as corrosive or flammable. Liquid fuels placed in the storage bunkers included Otto fuel, JP-10, and TH Dimer (kerosene).

Two drainage culverts (one along the northwest side and one along the southeast side of the Area A Wetland) collect runoff from the surrounding hillsides and from the Area A Weapons Center and discharge it to the Area A Wetland. The drainage culvert along the northwest side eventually discharges to a storm sewer which passes along the southern side of the site and discharges into the Area A Wetland. The drainage culvert along the southeast side of the wetland collects runoff from the hillside north of the site and continues along the southeast side of the site, eventually discharging to the Area A Wetland. Water typically flows in these drainage culverts immediately following precipitation events.

Originally, runoff from the Area A Landfill drained as overland flow to the north into the Area A Wetland. As a result of the installation of the landfill cap in September 1997, surface water flow patterns at the landfill have been significantly altered. A storm water management system is incorporated into the landfill cover system to minimize ponding and potential leaching of contaminants to the groundwater through

infiltration. The storm water management system was designed to direct runoff from a storm event around the cover system and into the Area A Wetland and to intercept a portion of the shallow groundwater flowing into the landfill from the southern slope. Runoff from the landfill discharges through a culvert into Site 3 to the northwest and, ultimately, into the Thames River. The system consists of five surface water diversion channels, two reinforced concrete culverts, and a riprap channel to convey the water. Four of the surface water diversion channels are asphalt-lined to funnel surface water flow, and the fifth channel consists of riprap to provide appropriate erosion protection prior to discharge into the Area A Wetland. The two culverts were installed to allow vehicular access to the landfill at Thresher Avenue and at the access gate located north of Building 460. Contaminants that migrate from these areas in surface runoff are likely to accumulate in the Area A Wetland. Contamination in the Area A Wetland may also result from the leaching of materials from the dredge spoils that were historically pumped into the area. Further contaminant migration into groundwater may also occur with subsequent transport in groundwater in downgradient flow directions.

In 1997, Phase I and II Remedial Investigations (RIs) and a Focused Feasibility Study were conducted for the Area A Wetland (Brown and Root Environmental, 1997). The scopes of these investigations are summarized in the following sections. Tables 1-1 through 1-4 list the surface soil, sediment, surface water, and biological tissue samples that were collected as part of these investigations, and the analyses that were conducted on each of the samples, respectively. Figure 1-2 presents the locations of the samples, except for the biological tissue samples (frogs and fledgling catbirds), since frogs and catbirds are mobile, and the exact locations of these biological samples were not documented. Note that Figure 1-2 also presents the locations of the surface water samples collected as part of the Area A Landfill monitoring program, as well as some sediment samples collected as part of investigations at the Area A Weapons Center. In addition, it is noted that in 1999, a few soil sample locations on Figure 1-2 were collected during the installation of monitoring wells at the Area A Wetland.

A human health risk assessment (HHRA) and a screening level ecological risk assessment (SERA) were conducted for the Area A Wetland as part of the Phase II Remedial Investigation (RI) for NSB-NLON (Brown and Root Environmental, 1997). The results of the HHRA are presented in the Phase II RI (Brown and Root Environmental, 1997). The SERA concluded that chemicals in the surface water, sediment, and surface soil of the Area A Wetland represent a potential risk to both aquatic and terrestrial receptors. Before proceeding further in the SERA process, the Navy determined that the SERA should first be updated using current methodologies and toxicity data, because the risk assessment is more than 10 years old. Appendix C presents the results of the updated SERA that were used to determine additional data needs, as discussed in this section. The results are summarized in Section 1.4.2.2.

Since 1999, a Groundwater Monitoring Program for Area A Landfill has been conducted to evaluate the effectiveness of an Interim Remedial Action (IRA) completed in September 1997 by Foster Wheeler Environmental Corporation (FWENC).

The following subsections present a summary of the sampling that was conducted as part of the investigations for the Phase I RI, the Phase 2 RI, the FFS, the Area A Landfill Monitoring Program, and the Area A Weapons Center. The subsections discuss the investigations; however, summary tables are provided only for the surface soil, sediment, surface water, and tissue samples, since only those data were evaluated in the updated SERA in Appendix C.

### Phase I RI

A Phase I RI of four sites was completed at NSB-NLON by Atlantic Environmental Services, Inc., from 1990 through 1992. Additional investigations were recommended for three of the sites, including Area A – Site 2 of which the Area A Wetland is a part.

A total of 41 soil/sediment samples were collected from the Area A Wetland during the Phase I RI, including 16 samples, plus 4 field duplicates, from depths within 2 feet of ground surface (considered to be surface soil/sediment samples) and 25 samples, plus one field duplicate, from depths starting at greater than 2 feet below ground surface (bgs), considered to be subsurface soil samples (see Tables 1-1 and 1-2 and Figure 1-2). All soil samples collected from depth intervals which began in the 0- to 2-foot bgs range and went no deeper than 4 feet bgs were considered surface soils, as shown on Table 1-1. The 41 soil/sediment samples consisted of the following types of samples. Eight composite sediment samples (2WSD1 through 2WSD8) plus one sediment field duplicate were collected from various onsite areas, and a ninth grab sediment sample (2WSD9) was collected from the drainage culvert coming from the Area A Weapons Center. The seven areas where composite samples 2WSD1 through 2WSD4 and 2WSD6 through 2WSD8 were collected were also each evaluated via the installation of a test boring to investigate deeper soils. Five surface and 19 subsurface soil samples (plus two surface and one subsurface field duplicates) were collected from the seven test borings in these areas. Additional soil samples (two surface soils plus one surface soil field duplicate and six subsurface soils) were collected from four monitoring well borings located throughout the Area A Wetland. All soil/sediment samples collected within the limits of the wetland were from depths at or below the groundwater table. Chemical concentrations in the subsurface soil samples were much lower than the concentrations in the surface soil samples (Brown and Root Environmental, 1997).

Seven groundwater samples were collected from three shallow monitoring wells and four deep wells in the Area A Wetland during the Phase I RI. Two surface water samples (plus one field duplicate) were also collected: one located near Route 12, and the other located near the dike outlet (see Table 1-3 and

Figure 1-3). Several avian and amphibian ecological samples (i.e. fledging catbirds and frogs) were also collected during the Phase I RI (see Table 1-4).

#### Phase II RI

The Phase II RI was conducted as part of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) of 1980 (also known as Superfund) and U.S. Navy IRP RI programs. The RI's purpose was to determine the nature and extent of contamination, to assess the human health and environmental risks posed by contamination. Remediation alternatives were recommended for 13 sites identified at NSB-NLON, including the Area A Wetland.

Four new groundwater monitoring wells were installed, and two rounds of groundwater sampling were completed. Ten groundwater samples were collected from four shallow (overburden) and six deep (bedrock) wells during Round 1, and ten samples (including one field duplicate) were collected from three shallow and six deep wells during Round 2. Twenty nine sediment samples were collected for analysis of 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT (see Table 1-2 and Figure 1-2). Six of those samples were analyzed at a fixed-base laboratory. In addition to the two Phase I RI surface water sample locations, seven additional surface water samples were collected from additional locations along Route 12, along the Area A Landfill boundary, and in the northeast portion of the site (east of the Area A Weapons Center) (see Table 1-3 and Figure 1-3).

#### Focused Feasibility Studies (FFSs)

Focused feasibility studies (FFSs) were prepared for the Area A Downstream/OBDA and the Area A Landfill to select remedial actions for mitigation of risks to human health and the environment associated with contaminated sediments and soils at the site. Some sample collection for both FFSs occurred within the Area A Wetland boundary.

Four sediment samples (2WSD23, 2WSD24, 2WSD25, and 2WSD26) were collected from the southwest portion of the Area A Wetland (along the earthen dike) as part of the Area A Downstream/OBDA FFS (see Table 1-2 and Figure 1-2). Twenty additional sediment samples were collected from ten transects straddling the Area A Landfill/Area A Wetland boundary as part of the Area A Landfill FFS (see Table 1-2 and Figure 1-2). Two sediment samples were collected from each transect: one was collected from the wetland boundary, and one was collected approximately 20 feet from the wetland boundary, within the wetland area.

### Area A Landfill Monitoring Program

An Interim Remedial Action (IRA) was completed in 1997 at the Area A Landfill site to reduce the risk from direct exposure to landfill materials and to minimize the risk of migration of chemicals of concern from the landfill to the surrounding areas via groundwater. The IRA consisted of capping the site with a multi-layer low-permeability cover system and installing a surface water and shallow groundwater interception and diversion system upgradient of the cover system.

In October 1999, the Navy implemented a monitoring program for groundwater and surface water at the Area A Landfill to assess the effectiveness of the IRA previously described (TtNUS, 1999). Monitoring activity at the Area A Landfill was initially conducted quarterly. During Year 3 (2001/2002), the monitoring frequency was reduced to semi-annually. Annual reports were issued for each year of the monitoring program, summarizing the results from water quality testing. Appendix D summarizes the surface water sampling portion of the monitoring program. The locations of the surface water samples are presented on Figure 1-3. Note that surface water samples were not collected at all locations during all rounds.

### Area A Weapons Center

The Area A Weapons Center was investigated during the Phase I RI, Phase II RI, Basewide Groundwater Operable Unit Remedial Investigation (BGOURI), and BGOURI Data Gap Investigation (DGI). The results of these investigations showed minimal contamination of the groundwater and surface water at the site, but indicated that the soil and sediment at the site may be a contaminant source to the Area A Wetland. A remedial action of a small removal of less than 200 cubic yards was conducted at the site in 2001 to reduce the PAH and arsenic contamination in the soil and sediment at the site. Soil and sediment were removed from Drainage Area 1, while contamination levels in Drainage Areas 2 and 3 were less than cleanup levels and did not require remediation. Several sediment samples were collected as part of the investigations at the Area A Weapons Center. Figure 1-2 shows the locations of the sediment samples that are either in, or adjacent to, the Area A Wetland.

#### **1.4.2.2 Problem Definition**

Project Data Quality Objectives (DQOs) were developed in accordance with USEPA Guidance for the DQO Process, commonly known as QA/G-4 (USEPA, February 2006). The following discussion provides information on the project planning conducted to develop the DQOs. The project definition, the project quality objectives, and the measurement performance criteria are identified, based on the DQOs, and are provided in this QAPP.

Problem Statement

In 1997, the results of a SERA performed for the Area A Wetland indicated that the chemicals detected in the surface soil, sediment, and surface water represented a potential risk to ecological receptors, both aquatic and terrestrial. It was necessary to update the 1997 SERA using current ERA methodologies to determine whether the collection of additional data is necessary under the current ERA requirements. This updated SERA is presented in Appendix C. Based on the revised SERA, additional sample collection is required to obtain current site data to fully characterize ecological risks and to more characterize the nature and extent of contamination prior to performing a feasibility study (FS). These additional data will support the implementation of any future feasibility studies designed to evaluate remedial options for reducing or eliminating risk from site contaminants.

The Area A Landfill lies to the southwest of the Area A Wetland, and the Area A Weapons Center is located to the northwest of the wetland. Both borders are clearly defined. Attachment 1 (Appendix C) presents figures that show the locations of concentrations of selected chemicals in sampled media. The sample locations are shaded green if the chemical concentrations in sediment or soil are less than the ecological sediment screening values used in the SERA, shaded red if the concentrations are greater than the "higher effects levels" for sediment, and shaded yellow if the detected concentrations are between those values. The chemical concentrations in the soil samples were compared to the sediment screening values, because some of the soil locations may be periodically submerged, and/or the soil may erode into the sediment. If a chemical was not detected in a sample, a value equal to one-half of the detection limit was used as the sample concentration, with the exception of total PAHs and total DDTRs, which used positive detections only. Figure 10 in Attachment 1 of Appendix C depicts total organic carbon (TOC) concentrations in sediment samples; TOC is used to evaluate the potential bioavailability of the chemicals. A review of these figures shows that the greatest chemical concentrations were generally found in the western portion of the wetland, adjacent to the Area A Weapons Center and the Area A Landfill. Other portions of the wetland generally had lower chemical concentrations with some elevated detections that have no discernable distribution pattern.

The updated SERA identified potential risks to ecological receptors from chemicals in the sediment and surface water. The contaminants causing the greatest risk were PAHs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, PCBs (Aroclor-1260), and metals in the sediment. Some sediment sample locations, which had the highest chemical concentrations, were near the Area A Landfill. These locations may have been covered by the landfill cap since the date of sampling. Therefore, additional sampling in that area is needed to determine whether the sediments with previously elevated levels of chemicals are still exposed. Also, because most of the sediment samples used in the updated SERA were collected in 1990 and 1993, it is possible that the chemical concentrations in sediments have changed over time.

Although some chemicals were detected in the surface water at concentrations that exceed the ecological screening levels, it is assumed that the sediment is the medium of greater concern, because any remedial actions at the site would likely focus on the sediment, and not surface water. Also, surface water samples are currently being collected as part of the long-term monitoring program for the Area A Landfill and were used in the ERA in Appendix C.

As presented in the Phase II RI, the chemical concentrations in the surface soil/sediment were much higher than in the subsurface soil. However, the subsurface soil samples were not located in the areas where the greatest sediment concentrations were found. Analytical data for subsurface sediment are necessary from these areas to evaluate options in the FS, should one be needed. On top of the dredge spoils in the wetland, a layer of organic material has formed; however, the thickness of the organic material is not known. Characterizing this thickness is important, because a thicker layer of organic material would more likely isolate the dredge spoils from ecological receptors and would also adsorb chemicals and decrease their bioavailability.

#### Goals of the Study

The following decision statements were developed to address the issues identified in the problem statement.

**Decision Statement No. 1a:** After the collection of Phase III data for analytes of concern and conducting a SERA for ecological receptors of concern (based on existing data and Phase III data), determine whether an unacceptable ecological risk persists at the Area A Wetland – Site 2B:

- If the SERA shows an unacceptable ecological risk exists, recommend that additional data (i.e., Phase IV) be collected as part of a Baseline Ecological Risk Assessment (BERA), using the SERA to refine the analytes and receptors of concern.
- If the SERA shows that risks are acceptable, no further action is necessary.

**Decision Statement No. 1b:** After the collection of additional data (i.e., Phase IV) for analytes of concern and completion of the BERA for ecological receptors of concern using all data (including Phase IV), determine whether an unacceptable ecological risk persists at the Area A Wetland – Site 2B:

- If the BERA indicates an unacceptable ecological risk, develop site-specific cleanup goals and perform a Feasibility Study, including a Function and Values Assessment and a Net Environmental Benefits Analysis.
- If the BERA indicates no further ecological risk, no further action is necessary.

**Decision Statement No. 2:** Determine whether the chemical concentrations in the Phase III sediment samples are similar to those from the previous data set, and determine whether the extent of contamination is adequately defined:

- If the chemical concentrations in the Phase III sediment samples are similar to the concentrations in the previous data set, and the extent of contamination is adequately defined, do not collect additional data.
- If the chemical concentrations in the sediment samples are not similar to the concentrations in the previous data set, or the extent of contamination is not adequately defined, determine what additional data is needed.

**Decision Statement No. 3:** Determine the thickness of the overlying organic layer that has formed above the dredge spoils:

- If the data collected are adequate to determine the thickness of the overlying organic layer, no further action is required.
- If the data collected are not adequate to determine the thickness of the organic layer, obtain additional data to allow an accurate profile of the organic layer thickness.

#### Information Inputs

The following data and evaluations are needed to determine the ecological risks and the nature and extent of contamination in the Area A Wetland sediment:

#### **Decision Statements 1a and 1b**

Historic and Phase III (and Phase IV, if necessary) data for the following media will be used to determine the ecological risk in the Area A Wetland:

- Soil
- Sediment
- Surface Water
- Biological Tissue

The concentrations of chemicals in the surface soil were relatively low and unlikely to pose a significant risk to terrestrial ecological receptors. Therefore, additional surface soil samples do not need to be

collected during this Phase III investigation. Based on the information in the problem statement, the collection of additional sediment samples is required to determine whether or not the chemical concentrations have changed over time.

As discussed above, although some chemicals were detected in the surface water samples at concentrations that exceed screening levels, there is generally a smaller likelihood of ecological risk from chemicals in the surface water compared to sediment; therefore, the collection of surface water samples during this Phase III investigation is not recommended. Based on the results of food chain modeling in the updated SERA (see Appendix C), risks to wildlife were relatively low, so the collection of tissue samples during this Phase III investigation should not be necessary. Biological/toxicity testing is not proposed at this time. If a BERA is pursued, toxicity and/or bioaccumulation tests may be conducted.

Based on the updated SERA, it was determined that the primary risk drivers were the following chemicals in sediments: PAHs; 4,4'-DDD; 4,4'-DDE; 4,4'-DDT; PCBs (Aroclor-1260); and metals. Therefore, analytical results that are accurate for concentrations of these chemicals in sediments are required. In addition, total organic carbon (TOC) and pH data are needed to help evaluate the potential bioavailability of the chemicals in sediments. Standard EPA SW-846 analytical methods are adequate for this investigation, including Gas Chromatography/Mass Spectroscopy/Selected Ion Monitoring (GC/MS/SIM) for PAHs.

#### **Decision Statement 2**

The target analytes for the samples collected as part of the Phase III investigation are the same as they are for Decision Statements 1a and 1b.

#### **Decision Statement 3**

Decision Statement 3 addresses the characterization of the organic layer that has formed above the Area A Wetland dredge spoils. The thickness of the organic layer overlying the dredge spoils is required to determine whether or not the dredge spoils are isolated from ecological receptors, and to evaluate the likelihood that the organic carbon in the organic layer can adsorb the chemicals, thereby decreasing their bioavailability.

The determination of the organic layer thickness will be primarily visual; however, TOC measurements are needed to evaluate the potential for the organic layer to adsorb chemicals.

### Study Boundaries

The study boundaries are defined for each of the decision statements established above.

### Decision Statement 1

The exposure unit is, and sampling will occur within the Area A Wetland, as delineated on Figure 1-2. The sediment samples will be collected from a depth of 0 to 4 inches, based on the approximate depth of the biotic zone for sediment invertebrates. However, if the site progresses to a FS, subsurface sediment analytical data (2-4 feet) would be required. The sampling area will be focused in the western portion of the site, between the Area A Wetland and the Area A Weapons Center and the Area A Landfill, covering approximately half of the area of the wetland, because as discussed above, the available data indicates that this is the area where contamination and risks are the greatest. Chemical concentrations in the other portions of the wetland are relatively low, or are elevated in sporadic locations.

The sediment area to be sampled was selected to determine current chemical concentrations in the areas where the concentrations were greatest in the historic samples, and to determine whether the construction of the landfill cap and the associated rip-rap along the toe of the landfill resulted in covering the sediment that previously had elevated chemical concentrations. The area to be represented will not extend past the border of the landfill due to the riprap at the landfill toe. It is important not to disturb the landfill cover. An area adjacent to the Area A Weapons Center has been remediated; however, the area immediately adjacent to the Area A Wetland has not been remediated, and some of the higher chemical concentrations are found in these sediment samples (see figures in Appendix C, Attachment 1).

### Decision Statement 2

The lateral boundaries for the extent of contamination in surficial sediment are the same as those for Decision Statement 1. For the subsurface sediment samples, the lateral boundaries are the locations where the greatest concentrations of chemicals are expected to be found in the surface sediment.

The vertical boundaries are 0 to 4 inches for the surficial sediment samples and a maximum depth of 4 feet for the subsurface sediment samples. The depth of 4 feet was selected as the likely maximum depth that would be evaluated for removal in the FS.

The surficial sediment depth of 0 to 4 inches is different than the sampled depths in the previous sediment samples (0 to 6 inches in the Phase I and Phase II RIs and from 0 to 12 inches in the FFS). Therefore, comparisons made between the two data sets will be somewhat limited. However, the depth of 0 to 4 inches was selected because the primary purpose of the investigation is to determine the ecological

risks to sediment invertebrates and the biologically active zone is typically within the top 10 cm (0 to 4 inches). The chemical concentrations in this zone may be lower than the concentrations in the previous samples if the more contaminated sediment was covered with cleaner sediment. Conversely, the chemical concentrations in this zone may be greater than the concentrations in the previous samples if the subsurface sediment was less contaminated.

### **Decision Statement 3**

The thickness of the overlying organic layer needs to be assessed across the entire 23.6 acres of the Area A Wetland, but the primary focus should be where the chemical concentrations and risks are greatest. Access to the center of the site is difficult due to its boggy nature. A high degree of spatial resolution is not necessary, so areas representative of various habitats were selected for sampling. Existing core data for the Area A Wetland will also be combined with the new data to help determine the organic layer thickness.

#### **Develop the Analytical Approach**

### **Decision Rules for Decision Statement No. 1 (Evaluation of Ecological Risk)**

The Phase III data is being collected to update the ecological risk assessment and to determine whether or not a BERA is necessary. A technical memorandum will be drafted to be given to regulators. If toxicity and/or bioaccumulation tests are conducted to support a BERA, these tests will be selected based on the results of the updated SERA results in consultation with regulators.

If, using professional judgment, the Phase III SERA identifies an unacceptable ecological risk, additional data will be collected, and a BERA will be conducted using all available data. Otherwise, no further action is necessary. Consequently, if the BERA shows unacceptable risk, based on professional judgment, conduct a Feasibility Study (including a Functions and Values Assessment and NEBA). Otherwise, no further action is necessary.

### **Decision Rules for Decision Statement No. 2 (Nature and Extent of Contamination)**

If, based on professional judgment, Phase III data collection adequately bounds the Area A Wetland contamination, so that contaminated areas are distinguishable from uncontaminated areas, and the nature and extent of the contamination are adequately established, collect no more data. Otherwise collect more data to adequately define the nature and extent of contamination.

The Area A Landfill and Area A Weapons Center boundaries, and the dike in the northwestern portion of the wetland will be used as physical boundaries. Factors to be considered will be the overall

contamination pattern compared to the action levels (i.e., background concentration and risk-based screening levels developed during the BERA, if necessary) and estimates of where chemical concentration gradients appear to decrease to concentrations less than the action levels.

### **Decision Rules for Decision Statement No. 3 (Organic Layer Thickness)**

Using visual observation to distinguish the organic layer from the dredge spoils material, if the top of the dredge spoils has not been reached, continue to dig until either a depth of 4 feet has been reached, or the layer has been reached. Otherwise, stop digging.

### **Specify Performance or Acceptance Criteria**

The decisions to be made require subjective input, in the form of professional judgment, as well as objective data, such as analyte concentrations. Therefore, a statistical analysis of decision uncertainty is not beneficial to this project.

### **Develop the Detailed Plan for Data Collection**

The remaining sections of this QAPP comprise the detailed plan for data collection.

## **1.5 PROJECT QUALITY OBJECTIVES AND MEASUREMENT PERFORMANCE CRITERIA**

This section summarizes the Project Quality Objectives (PQOs) for the sediment sampling activities. The PQOs were developed in accordance with the USEPA Guidance for the DQO Process, commonly known as QA/G-4 (USEPA, 2006). The following discussion provides information on the project planning conducted to develop the DQOs, the project definition, the project quality objectives, and measurement performance criteria identified based on the DQOs.

### **1.5.1 Development of Project Quality Objectives Using the Systematic Planning Process**

The PQOs outlined in Worksheet #11 address the type, quantity, and quality of data to be determined using the DQO process.

### **1.5.2 Measurement Performance Criteria**

Measurement performance criteria (MPCs) are the PARCCS parameters (i.e., precision, accuracy, representativeness, comparability, completeness, sensitivity), which 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 Worksheet #12.

#### 1.5.2.1 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. A fundamental tenet of using precision measurements for quality control (QC) is that precision will be bounded by known limits.

Two samples generated by removing representative portions from a single mass or volume of material are called 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, the 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 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 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).

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. The RPD between a sample or Matrix Spike (MS)

(Sample 1) and its duplicate or matrix spike duplicate (MSD) (Sample 2) is calculated for chemical analyses using the following formula:

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

#### 1.5.2.2 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted 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 typically ensured through control over the sample collection and handling and through routine instrument calibration. Accuracy is also typically monitored through the use of blanks to detect cross-contamination and by monitoring adherence to procedures that prevent sample contamination or degradation. Equipment rinse blanks shall be collected at a frequency of 1 per day or 1 per 20 samples per matrix, whichever is less, for this investigation to assess cross-contamination via sample collection equipment. Ambient condition blanks will not be collected unless site conditions during sampling (e.g., generation of fugitive dust) indicate a need to assess infiltration of airborne contaminants into sampling containers. Source water blanks (field blanks) will be collected to monitor the purity of water used to decontaminate sampling equipment. 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 of a spiked sample or LCS result to a known or calculated value 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 organic 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 a 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 percent recovery (%R) for a spiked sample (including surrogate compounds) is calculated by using the following formula:

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

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

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

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 QC limits for evaluating accuracy.

During data validation, any data not meeting accuracy specifications are 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.

### 1.5.2.3 Sample 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 without bias. Adherence to the standardized sample collection, handling, preparation, analysis, and 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.

#### 1.5.2.4 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 (QLs) and method detection limits (MDLs) are sufficiently low to satisfy project action limits for the duration of the project. The QLs and MDLs anticipated for this project are presented in 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.

#### 1.5.2.5 Completeness

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. Completeness will be computed in accordance with the following equation:

$$\% \text{ Completeness} = \frac{\text{Number of Valid Measurements}}{\text{Number of Measurements Planned}} \times 100 \%$$

For relatively clean, homogeneous matrices, 100 percent 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 planned) 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 percent completeness), either with respect to the number of samples or an individual analysis,

resampling to replace the deficient data points may be necessary. The U.S. Navy and TtNUS will determine whether resampling is necessary.

#### **1.5.2.6 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 (QL) is the minimum concentration of an analyte that can be routinely identified and quantified above the minimum detection limit (MDL) by a laboratory. The MDL is the minimum concentration of an analyte that can be reliably distinguished from background noise for a specific analytical method.

Analytical methods were chosen for their ability to achieve the appropriate sensitivity for a specific matrix at this site. Analyte MDLs and QLs have been compared to project action limits [i.e., Ecological Screening Levels (ESLs)] to determine if the chosen analytical method is sensitive enough to meet the PQOs. Table/Worksheet #15 displays the comparison of Katahdin Analytical Services, Inc. laboratory's analytical MDLs and QLs to project action limits by method and matrix. The methods of analysis chosen can meet most of the project action limits for PAHs, pesticides, PCBs, and metals, as shown in Table/Worksheet #15. PAH, pesticide, and PCB results will be reported to QLs, while metals will be reported to instrument detection limits (IDLs).

### **1.6 SECONDARY DATA EVALUATION**

Secondary data are data that were collected previously by external and/or independent parties that are subsequently transmitted to the current data user. Worksheet #13 lists data collected during previous rounds of investigation, as relevant to this Phase III sampling event.

Secondary data from the Phase I RI, Phase II RI, and the FFS, that can be found in the Phase II RI Report for NSB-NLON (Brown and Root Environmental, 1997) will be combined with current investigation data (fully validated) and used in support of the updated RI/FS Report. The data were subjected to a series of technical reviews and were approved by the U.S. Navy and regulators and, therefore, are useable as reported.

### **1.7 PROJECT OVERVIEW AND SCHEDULE**

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

**1.7.1 Project Overview**

Sediment samples will be collected in order to complete this Phase III Investigation. Tasks include implementation of field sampling programs outlined in this QAPP, and completion of a RI/FS report, unless additional data are required prior to completing that report. A summary of project tasks is shown in Worksheet #14. Worksheet/Table #15 displays QLs and MDLs in comparison to project action limits.

**1.7.2 Project Schedule**

The project schedule is displayed in Worksheet #16.

TABLE 1-1

SUMMARY OF SAMPLING AND ANALYTICAL PROGRAM FOR SOILS<sup>(1)</sup> - PHASE I  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT

Sample ID	Sample Depth (feet below ground)	Analyses			
		Target Compound List (TCL)			
		Volatiles	Semivolatiles	Pesticides/ PCBs <sup>(2)</sup>	TAL <sup>(3)</sup> Metals
<b>Phase I RI</b>					
2WTB2	0-2	• <sup>(4)</sup>	•	•	•
090690-2WTB9 (4-6) <sup>(5)</sup>	0-2	•	•	•	•
2WTB4	0-2	•	•	•	•
2WTB6	0-2	•	•	•	•
2WTB7	0-2	•	•	•	•
2WTB7 (25-27) <sup>(6)</sup>	0-2	•	•	•	•
2WTB8	1-3	•	•	•	•
2WMW2	0-2	•	•	•	•
2WMW5	0-2	•	•	•	•
2WTB11 <sup>(7)</sup>	0-2	•	•	•	•

1 This table only lists surface soil samples collected during the Phase I RI

2 Polychlorinated Biphenyls

3 Target Analyte List (TAL) Metals plus boron and cyanide

4 • indicates samples analyzed at a fixed-base laboratory

5 090690-2WTB9 (4-6) is a field duplicate of 2WTB2 (0-2)

6 2WTB7 (25-27) is a field duplicate of 2WTB7 (0-2)

7 2WTB11 (0-2) is a field duplicate of 2WMW5 (0-2)

RI - Remedial Investigation

TABLE 1-2

SUMMARY OF SAMPLING AND ANALYTICAL PROGRAM FOR SEDIMENT - PHASE I AND II RI AND FFS  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 1 OF 2

Sample ID	Sample (feet below ground)	Analyses					
		Target Compound List (TCL)				TAL Metals <sup>(2)</sup>	Engineering <sup>(3)</sup>
		Volatiles	Semivolatile	Pesticides	PCB <sup>(1)</sup>		
<b>Phase I RI</b>							
112690-2WSD1	0-0.5	● <sup>(4)</sup>	●	●	●	●	
112690-2WSD10 <sup>(5)</sup>	0-0.5	●	●			●	
112690-2WSD2	0-0.5	●	●	●	●	●	
112690-2WSD3	0-0.5	●	●	●	●	●	
112690-2WSD4	0-0.5	●	●	●	●	●	
112690-2WSD5	0-0.5	●	●	●	●	●	
112690-2WSD6	0-0.5	●	●	●	●	●	
112690-2WSD7	0-0.5	●	●	●	●	●	
112690-2WSD8	0-0.5	●	●	●	●	●	
112690-2WSD9	0-0.5	●	●	●	●	●	
<b>Phase II RI</b>							
2WSD10	0-0.5			○ <sup>(6)</sup>			
2WSD11	0-0.5			○			
2WSD12	0-0.5			○			
2WSD13	0-0.5			●			
2WSD14	0-0.5			●/○			
2WSD15	0-0.5			○			
2WSD16	0-0.5			○			
2WSD17	0-0.5			○			
2WSD18	0-0.5			○			
2WSD19	0-0.5			○			
2WSD20	0-0.5			○			
2WSD21	0-0.5			○			
2WSD22	0-0.5			○			
2WSD27	0-0.5			○			
2WSD28	0-0.5			○			
2WSD29	0-0.5			○			
2WSD30	0-0.5			○			
2WSD31	0-0.5			○			
2WSD32	0-0.5			○			
2WSD33	0-0.5			○			
2WSD34	0-0.5			●/○			
2WSD35	0-0.5			○			
2WSD36	0-0.5			○			
2WSD37	0-0.5			○			
2WSD38	0-0.5			●/○			
2WSD39	0-0.5			●/○			● <sup>(7)</sup>
2WSD40	0-0.5			●/○			
2WSD41	0-0.5			●/○			
2WSD42	0-0.5			○			

TABLE 1-2

**SUMMARY OF SAMPLING AND ANALYTICAL PROGRAM FOR SEDIMENT - PHASE I AND II RI AND FFS  
SITE 2B - AREA A WETLAND  
NSB-NLON, GROTON, CONNECTICUT  
PAGE 2 OF 2**

Sample ID	Sample (feet below ground)	Analyses					
		Target Compound List (TCL)				TAL Metals <sup>(2)</sup>	Engineering <sup>(3)</sup>
		Volatiles	Semivolatile	Pesticides	PCB <sup>(1)</sup>		
<b>FFS<sup>(8)</sup></b>							
2WSD23	0-1			•	•		•
2WSD24	0-1			•	•		•
2WSD25	0-1			•	•		•
2WSD26	0-1			•	•		•
T1-A	0-1	•	•	•	•	• <sup>(9)</sup>	•
T1-B	0-1	•	•	•	•	•	•
T2-A	0-1	•	•	•	•	•	•
DUP-03 <sup>(10)</sup>	0-1	•	•	•	•	•	•
T2-B	0-1	•	•	•	•	•	•
T3-A	0-1	•	•	•	•	•	•
T3-B	0-1	•	•	•	•	•	•
T4-A	0-1	•	•	•	•	•	•
T4-B	0-1	•	•	•	•	•	•
T5-A	0-1	•	•	•	•	•	•
T5-B	0-1	•	•	•	•	•	•
T6-A	0-1	•	•	•	•	•	•
DUP-05 <sup>(11)</sup>	0-1	•	•	•	•	•	•
T6-B	0-1	•	•	•	•	•	•
T7-A	0-1	•	•	•	•	•	•
T7-B	0-1	•	•	•	•	•	•
T8-A	0-1	•	•	•	•	•	•
T8-B	0-1	•	•	•	•	•	•
T9-A	0-1	•	•	•	•	•	•
T9-B	0-1	•	•	•	•	•	•
T10-A	0-1	•	•	•	•	•	•
T10-B	0-1	•	•	•	•	•	•

1 Polychlorinated Biphenyls

2 Target Analyte List (TAL) metals plus boron and cyanide

3 Engineering Characteristics for sediment include grain size distribution, moisture content, and total organic carbon content.

4 • indicates samples analyzed at a fixed-base laboratory

5 112690-2WSD10 is a field duplicate of 112690-2WSD1

6 ○ indicates samples field screened with a portable gas chromatograph

7 The engineering characteristics also measured in this sample include specific gravity, organic content, cation exchange capacity, and pH.

8 Samples 2WSD23, 2WSD24, 2WSD25, and 2WSD26 were collected as part of the Area A Downstream/OBDA Focused Feasibility Study (FFS). The remaining samples were collected as part of the Area A Landfill FFS.

9 For the FFS, samples were analyzed for TAL metals plus boron and hardness.

10 DUP-03 is a field duplicate of T2-A

11 DUP-05 is a field duplicate of T6-A

TABLE 1-3

SUMMARY OF SAMPLING AND ANALYTICAL PROGRAM FOR SURFACE WATER - PHASE I AND II RI  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT

Sample ID	Sample Depth (feet below ground)	Analyses						
		Target Compound List (TCL)				TAL Metals <sup>(2)</sup>		Radiological <sup>(3)</sup>
		Volatiles	Semivolatiles	Pesticides	PCB <sup>(1)</sup>	Total	Dissolved	
<b>Phase I RI</b>								
121090-2WSW1	--	● <sup>(4)</sup>	●	●	●	●		●
121090-2WSW2	--	●	●	●	●	●		●
121090-2WSW3 <sup>(5)</sup>	--	●	●	●	●	●		●
<b>Phase II RI</b>								
2WSW1	Surface	●		●	●	● <sup>(6)</sup>	● <sup>(6)</sup>	
2WSW2	Surface	●	●	●	●	●	●	
2WSW6	Surface	●		●	●	●	●	
2WSW7	Surface	●		●	●	●	●	
2WSW8	Surface	●		●	●	●		
2WSW9	Surface	●		●	●	●		
2WSW10	Surface	●		●	●	●		
2WSW11	Surface	●		●	●	●	●	
2WSW12	Surface	●		●	●	●	●	

- 1 Polychlorinated biphenyls
- 2 Target Analyte List (TAL) Metals plus boron and hardness
- 3 Radiological analyses were gross alpha and gross beta analyses
- 4 ● indicates samples analyzed at a fixed-base laboratory
- 5 121090-2WSW3 is a field duplicate of 121090-2WSW2
- 6 During Phase II, the samples were analyzed for TAL metals plus boron and hardness

TABLE 1-4

**SUMMARY OF SAMPLING AND ANALYTICAL PROGRAM FOR TISSUE - PHASE I RI  
SITE 2B - AREA A WETLAND  
NSB-NLON, GROTON, CONNECTICUT**

Sample ID	Sample Type	Analyses			
		Target Compound List (TCL)		TAL Metals <sup>(2)</sup>	Percent Body Lipids
		Pesticides	PCB <sup>(1)</sup>		
<b>AVIAN<sup>(3)</sup></b>					
90 MBO 01	Tissue	• <sup>(4)</sup>	•	•	•
90 MBO 02	Tissue	•	•	•	•
90 MBO 03	Tissue	•	•	•	•
90 MBO 04	Tissue	•	•	•	•
90 MBO 05	Tissue	•	•	•	•
90 MBO 06	Tissue	•	•	•	•
90 MBO 07	Tissue	•	•	•	•
90 MBO 08	Tissue	•	•	•	•
90 MBO 09	Tissue	•	•	•	•
90 MBO 10	Tissue	•	•	•	•
90 MBO 11	Tissue	•	•	•	•
90 MBO 12	Tissue	•	•	•	•
90 MBO 13	Tissue	•	•	•	•
90 MBO 14	Tissue	•	•	•	•
<b>AVIAN - CONTROL</b>					
90 MBO 15	Tissue	•	•	•	•
90 MBO 16	Tissue	•	•	•	•
90 MBO 17	Tissue	•	•	•	•
<b>AMPHIBIAN<sup>(5)</sup></b>					
Pond 1A	Tissue	•	•	•	•
Pond 1A	Liver	•	•		•
Pond 1B	Tissue	•	•	•	•
Pond 1B	Liver	•	•		•
Pond 1C	Tissue	•	•	•	•
Pond 1C	Liver	•	•		•
Pond 1D	Tissue	•	•	•	•
Pond 1D	Liver	•	•		•
<b>AMPHIBIAN - CONTROL<sup>(6)</sup></b>					
90 MBO 18	Tissue	•	•	•	•
90 MBO 18	Liver	•	•		•

1 Polychlorinated Biphenyls

2 Target Analyte List (TAL) metals plus cyanide.

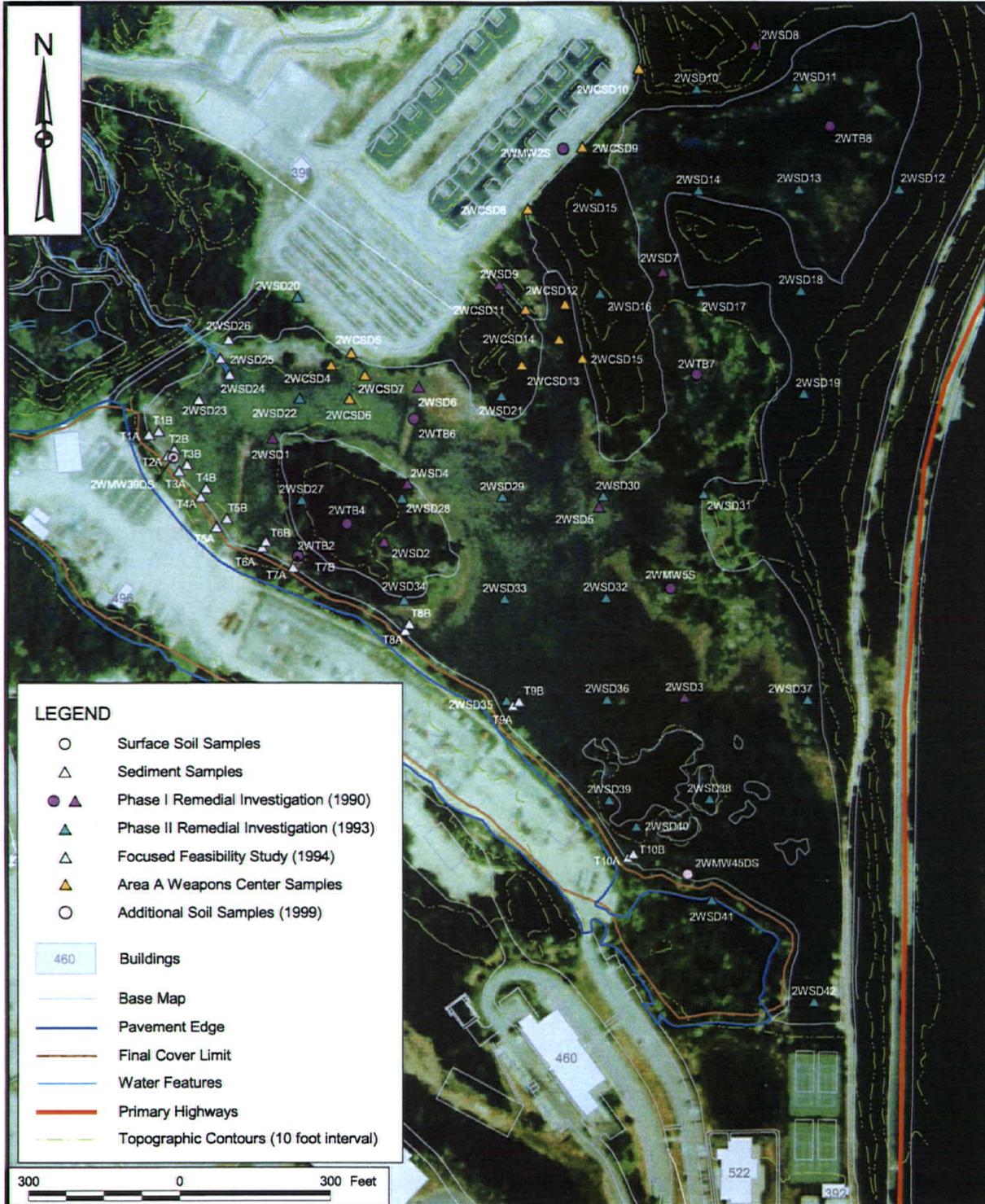
3 Gray Catbird fledglings were trapped in both Area A Downstream and Area A Wetland. Information was not available to determine from which of the sites the fledglings were collected.

4 I indicates samples analyzed at a fixed-based laboratory.

5 Frogs.

6 Amphibian Control samples are applicable to Area A Downstream and Area A Wetland.

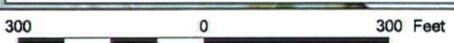




**LEGEND**

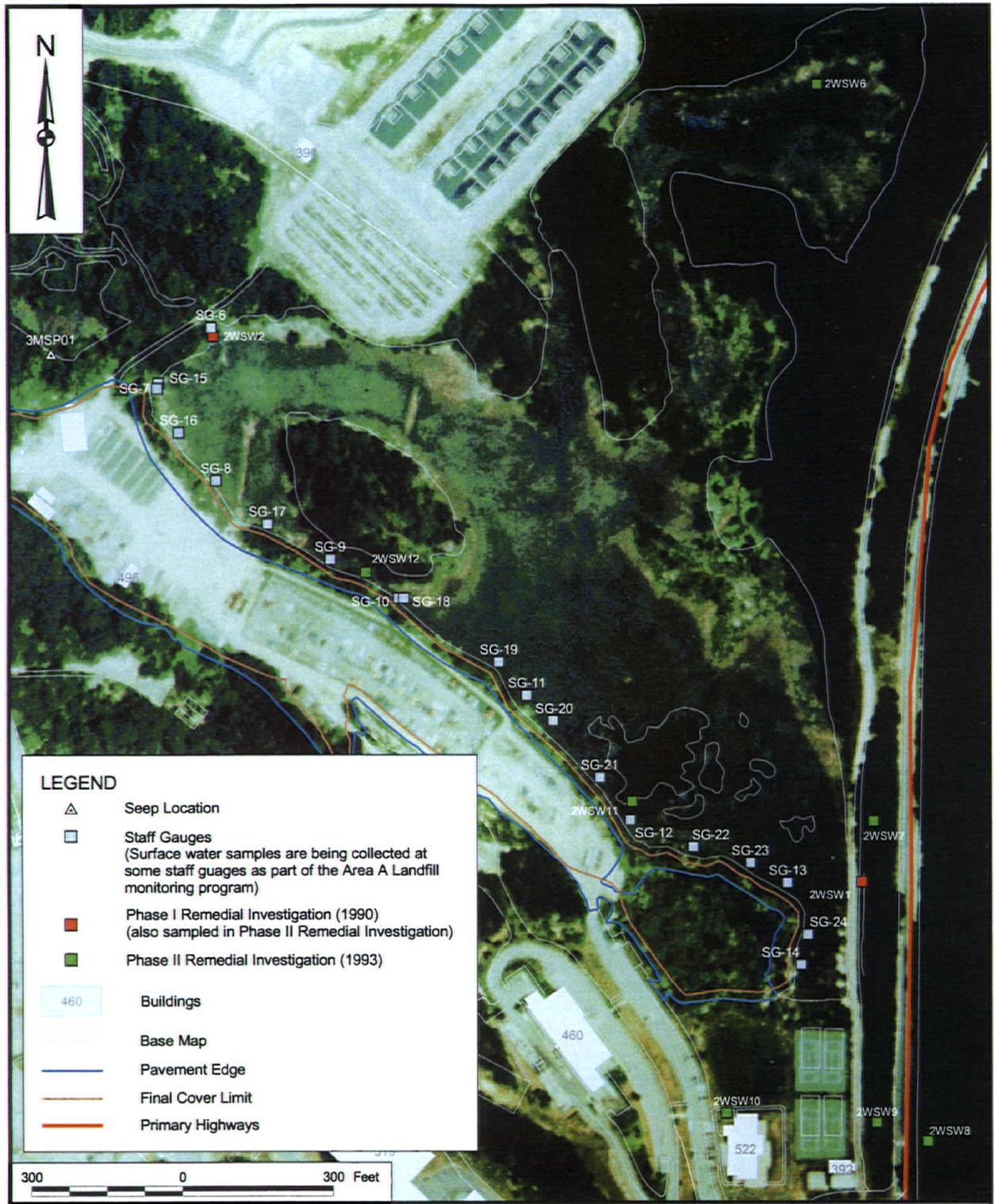
- Surface Soil Samples
- △ Sediment Samples
- ▲ Phase I Remedial Investigation (1990)
- ▲ Phase II Remedial Investigation (1993)
- ▲ Focused Feasibility Study (1994)
- ▲ Area A Weapons Center Samples
- Additional Soil Samples (1999)

- 460 Buildings
- Base Map
- Pavement Edge
- Final Cover Limit
- Water Features
- Primary Highways
- Topographic Contours (10 foot interval)



DRAWN BY J. ENGLISH CHECKED BY A. BERNHARDT COST/SCHEDULE-AREA SCALE AS NOTED	DATE 8/22/07 DATE 8/10/07 DATE DATE DATE	Tetra Tech NUS, Inc. HISTORICAL SOIL & SEDIMENT (SD) SAMPLING LOCATIONS AREA A WETLAND NAVAL SUBMARINE BASE NEW LONDON GROTON, CONNECTICUT	CONTRACT NUMBER CTO 0439 APPROVED BY APPROVED BY DRAWING NO.	OWNER NO. DATE DATE DATE FIGURE 1-2 REV 0
---	--	--	--	---

P:\GIS\NLON\APR\AREA A SITE LOCATION.APR AREA A SOIL AND SEDIMENT SAMPLING LOCATIONS LAYOUT 8/10/07 JEE



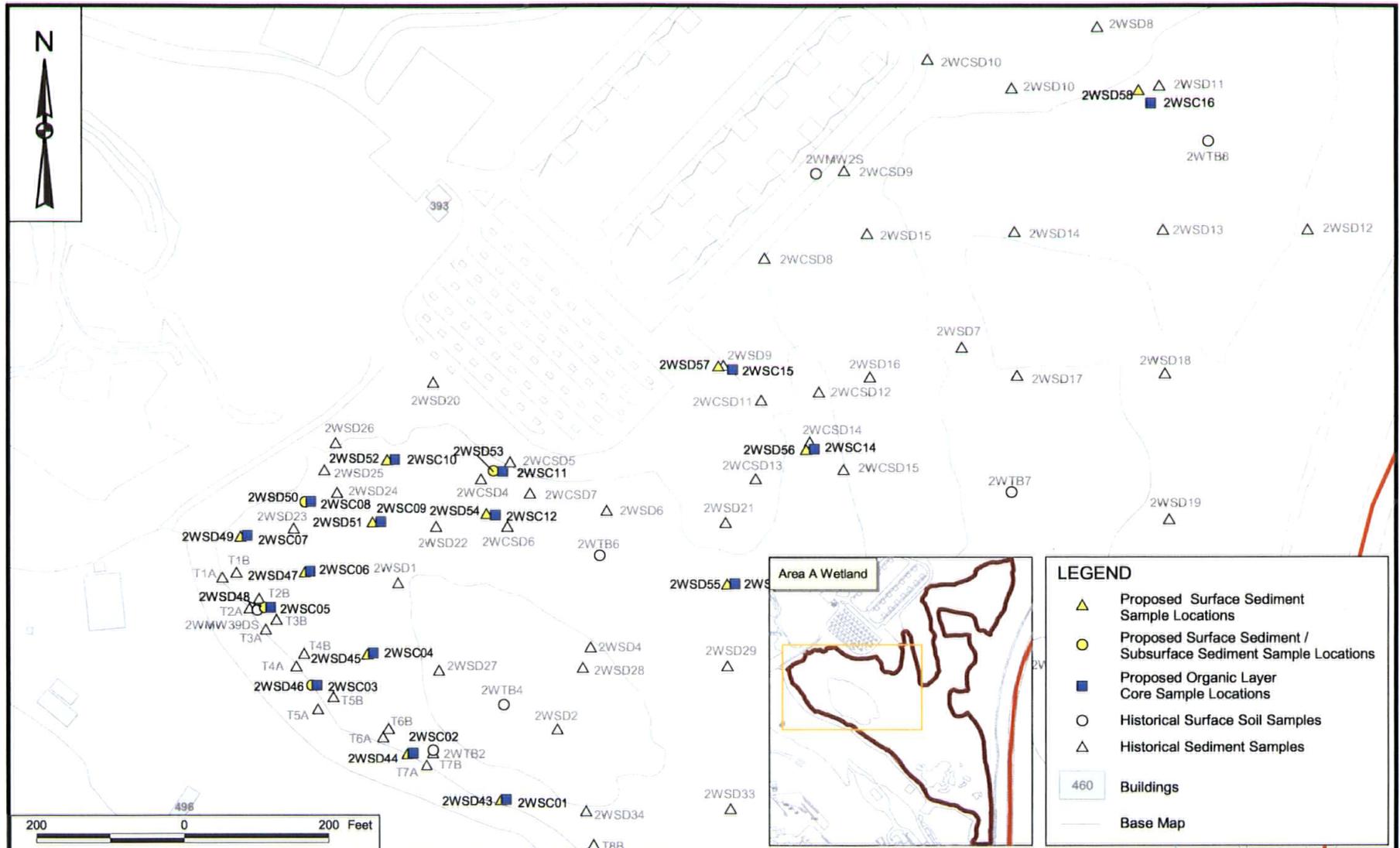
**LEGEND**

- △ Seep Location
- Staff Gauges  
(Surface water samples are being collected at some staff gauges as part of the Area A Landfill monitoring program)
- Phase I Remedial Investigation (1990)  
(also sampled in Phase II Remedial Investigation)
- Phase II Remedial Investigation (1993)
- 460 Buildings
- Base Map
- Pavement Edge
- Final Cover Limit
- Primary Highways



DRAWN BY J. ENGLISH CHECKED BY A. BERNHARDT COST/SCHEDULE-AREA SCALE AS NOTED	DATE 7/08/07 DATE 8/07/07 DATE DATE DATE	Tetra Tech NUS, Inc. HISTORICAL SURFACE WATER SAMPLING LOCATIONS AREA A WETLAND NAVAL SUBMARINE BASE NEW LONDON GROTON, CONNECTICUT	CONTRACT NUMBER CTO 0439 APPROVED BY APPROVED BY DRAWING NO. FIGURE 1-3	OWNER NO. DATE DATE REV 0
---	--	---	--	---------------------------------------

P:\GIS\INLON\APR\AREA A SITE LOCATION\APR AREA A SURFACE WATER SAMPLING LOCATIONS LAYOUT 8/07/07 JEE



DRAWN BY J. ENGLISH DATE 7/13/07			CONTRACT NUMBER CTO 0439		OWNER NO. 				
CHECKED BY A. BERNHARDT DATE 10/03/07			APPROVED BY 		DATE 				
COST/SCHEDULE-AREA 		PROPOSED SAMPLING LOCATIONS FOR PHASE III INVESTIGATION AREA A WETLAND NAVAL SUBMARINE BASE NEW LONDON GROTON, CONNECTICUT				APPROVED BY 		DATE 	
SCALE AS NOTED						DRAWING NO. FIGURE 1-4		REV 0	

## 2.0 MEASUREMENT/DATA ACQUISITION

### 2.1 SAMPLING TASKS

This section of the QAPP addresses the components of the sampling system, including sampling process design and rationale, procedures, and requirements.

#### 2.1.1 Sampling Process Design and Rationale

The overall sampling design and rationale for collecting various samples is presented in QAPP Worksheet #17. Figure 1-4 shows the planned sampling locations for surface sediment, subsurface sediment, and core locations.

A list of sample IDs, depths, Standard Operating Procedures (SOPs), and a summary of the rationale for sampling tasks is provided in QAPP Worksheet #18.

Analytical requirements for sample containers and preservation are summarized in QAPP Worksheet #19, also referenced in Section 2.1.2.2

The design and rationale of the proposed Phase III Investigation is presented in QAPP Worksheet #17. Tasks include collection of surface and subsurface sediment for chemical analysis for the updated RI/FS.

#### 2.1.2 Sampling Procedures and Requirements

##### 2.1.2.1 Sample Collection Procedures

A summary of sediment and soil sample site locations, sample IDs, depths, SOPs, and summary of rationale for sampling tasks is provided in Worksheet #18. For the sampling event, sample identification will be sequential for all media with a designator for Area A Wetland followed by the medium and unique sample location identifier. All TtNUS SOPs relevant to the investigation are listed on QAPP Worksheet #21 and the SOPs are included in Appendix A. Sediment samples that are collected for chemical analysis should have a minimum of 30 percent solids.

Based on the boring logs prepared during the construction of some of the monitoring wells in the Area A Wetland (which included soil sampling/characterization using split spoons), some of the locations had poor sample recovery. It was not clear if the recovery was poor because the sediment very soft or because *Phragmites* stalks prevented the soil from entering the split spoon. The thickness and depth of the organic layer above the dredge spoils will be visually determined by digging holes using a sediment

core or hand auger, and recording the observations. In addition, at approximately half of the locations, samples will be collected from the organic layer and the dredge spoils layer to determine if there is a difference in TOC concentrations in the sediment profile.

#### **2.1.2.2 Sample Containers, Volume, and Preservation**

Sample-specific information on containers, volume, and preservation requirements is provided on QAPP Worksheet #19.

#### **2.1.2.3 Field Quality Control Samples**

The collection of QC samples will include blind duplicates, matrix spike, field blank, and equipment blank samples. A summary of the frequency of QC sampling is included on QAPP Worksheet #20.

#### **2.1.2.4 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 laboratories. Sample equipment decontamination procedures are presented in SOP SA-7.1 in Appendix A.

#### **2.1.2.5 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 Section 2.1.2.4. 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. It is not expected that any electronic equipment needing calibration will be used during this field investigation.

#### **2.1.2.6 Field Documentation Procedures**

Field documentation will be performed in accordance with SOP SA-6.3 presented in Appendix A.

## 2.2 ANALYTICAL TASKS

This section provides information with regard to the analytical SOPs, calibration procedures, instrument/equipment maintenance, testing, and inspection procedures for the selected laboratory(s). Katahdin Analytical Services, Inc. will be used for the chemical analysis of the sediment and soil samples. Katahdin Analytical Services, Inc. is NFESC-approved and NELAP-accredited and certificates of accreditation are available upon request from Katahdin Analytical Services, Inc.

### 2.2.1 Analytical SOPs

All relevant analytical SOPs are summarized in QAPP Worksheet #23. These laboratory SOPs are provided in Appendix A.

### 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 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 QAPP Worksheet #25. These procedures are designed to ensure that all necessary equipment is continually available 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 required for analytical work will be available when needed and will be free of target compounds and any analytical interferences. Laboratory SOPs provided in Appendix A govern the inspection and acceptance procedures.

## 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 sampling tasks. Detailed and accurate documentation is necessary in order to ensure data integrity, authenticity, and defensibility.

### **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 used 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. Representative field forms are included in Appendix A at the end of the appropriate sampling SOP.

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

### **2.3.2 Sample Handling and Tracking System**

The following subsections outline the procedures that will be used by field and laboratory personnel to document sample collection activities during the soils and sediment sampling event. Detailed and accurate documentation is necessary in order to ensure data integrity.

#### **2.3.2.1 Sample Handling**

Sample handling is described in QAPP Worksheet #26.

### 2.3.2.2 Sample Delivery

The shipment of samples to the laboratory will be made by a shipping courier service (e.g., Federal Express), unless the laboratory is close enough to the site to provide a pickup service. After samples have been collected, they will be sent to the laboratory within 24 to 72 hours depending on the analyte holding time. 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 will each retain a copy of the form every 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 analytical report. Sample custody is described in QAPP Worksheet #27.

The 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 shipping courier. When transferring samples, the individuals relinquishing and receiving the samples will each sign the chain-of-custody form, and the date and time will be recorded. This documents the sample custody transfer from the sampler to the shipping courier, and finally, to the laboratory. Upon arrival at the laboratory, internal sample custody procedures will be followed as defined in the laboratory SOPs included in Appendix A.

## 2.4 QUALITY CONTROL SAMPLES

This section describes the QC samples that will be collected as part of the soil and sediment sampling event.

### 2.4.1 Sampling Quality Control Samples

QC samples will be collected or generated during environmental sampling activities and will include field duplicates for soils and sediment, where appropriate. A summary of QA/QC samples requirements are provided in QAPP Worksheet #20. The four types of field QC samples are defined as follows:

Field Duplicates - Field duplicates are used to assess the overall precision of the sampling and analysis program. Field duplicates will be collected at a frequency of 10 percent per sampling matrix. Duplicates are submitted for laboratory analyses for the same analytical parameters as the associated environmental samples.

Equipment Rinsate Blanks - Equipment rinsate blanks are obtained under representative field conditions by running analyte-free water through or over decontaminated sample collection equipment (e.g., hand tools, stainless steel bowls, 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 used on the project. Equipment rinsate blanks will be analyzed for the same analytical parameters as the associated environmental samples and will be collected in the appropriate sample containers.

Source Water Blank - 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 (i.e., 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 QC samples to be used during the sampling event are provided in QAPP Worksheet #28.

## 2.5 DATA MANAGEMENT TASKS

This section describes how project information will be managed, organized, and maintained for efficient use by project personnel. The information management process is outlined from 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 the project files is provided in QAPP Worksheet #29.

### 2.5.2 Data Package Deliverables

#### 2.5.2.1 Sample Collection and Field Measurement Data Package Deliverables

Sample collection data are as required by the sample collection SOPs of Appendix A. In addition, calibration data will also be provided in accordance with the SOPs of Appendix A although no field calibration of field equipment is expected.

#### 2.5.2.2 On-Site Analysis Data Package Deliverables

This project does not involve on-site analyses. Therefore, this section is not applicable.

#### 2.5.2.3 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, titled Analytical Services. A copy of the statement of work for analytical services is provided as Appendix E.

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 NIRIS-compatible electronic data deliverable (EDD). Data packages will be contract laboratory program (CLP)-equivalent (i.e. they will contain CLP-equivalent summary forms and raw data). 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 Appendix A of this QAPP.

### **2.5.4 Data Handling and Management**

The data-handling procedures to be followed by the laboratory 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. These forms are included in the applicable SOPs of Appendix A of this QAPP.

The overall field data flow is as follows:

1. Field personnel use this QAPP to identify sampling locations.
2. As applicable, sampling locations are evaluated to ensure they are safe for personnel to collect samples, and the evaluations are documented in accordance with the SOPs of Appendix A or the health and safety plan.
3. 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 the SOPs of Appendix A.
4. Samples are collected and documented and sampling equipment is decontaminated in accordance with the SOPs of Appendix A.
5. Samples are shipped to the laboratory for analysis and shipping documentation, including chain of custody records, are compiled for future transfer to the project manager.
6. 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.

7. Field conditions are recorded throughout the field work.
8. 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 and inspected, and the condition of the samples is logged.
2. Analytical equipment is calibrated for analysis, and the 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 (LIMS).
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. Upon completion of validation, 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 GIS system 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 PM will ensure that sample jar labels are printed before field sampling, if necessary. The FOL and PM, or PM designee, will review the labels for completeness of information and adherence to work plan requirements, as well as for accuracy. The PM will coordinate with the analytical laboratory to ensure that they are aware of the number and types 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 PM or designee via facsimile at the end of each day. The PM 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 PM 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 PM will ensure that the laboratory has 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 geographical information system (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 PM 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 2B - AREA A WETLAND  
NSB-NLON, GROTON, CONNECTICUT  
PAGE 1 OF 4**

DATA PACKAGE ELEMENTS	Semi-Volatiles /PAHs	Pesticides / PCBs	Metals	Miscellaneous <sup>(1)</sup>
♦ NARRATIVE (Org. Narrative, Inorg. Cover Page)	X	X	X	X
♦ SHIPPING/RECEIVING DOCUMENTS AND INTERNAL LABORATORY COC RECORDS:				
- Airbills	X	X	X	X
- Chain-of-Custody Records/Forms (Traffic Report)	X	X	X	X
- Sample Log-In Sheet (Org. and Inorg. DC-1 Form)	X	X	X	X
- Miscellaneous Shipping/Receiving Records	X	X	X	X
- Internal Lab. Sample Transfer Records and Tracking Sheets	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
- Tentatively Identified Compounds Tabulate Summary Form (Org. Form I TIC)				
- Reconstructed Total Ion Chromatogram (RIC) for each sample	X			
- Raw spectra of target compound and background subtracted spectrum of target compound for each sample	X			
- Mass spectra of all reported TICs/three best library matches for each sample				
- Chromatograms from both columns for each sample		X (pesticides)		
- GC Integration report or data system printouts and calibration plots for each sample	X	X		
- Pesticide/PCB Identification Tabulated Summary Form (Org. Form X)		X		
- For Pest/PCB or Dioxin/Furan results confirmed by GC/MS, copies of raw spectra and background subtracted spectrum of target compounds		X		
- GPC sample chromatograms (if necessary)	X	X		

TABLE 2-1

**LABORATORY DATA PACKAGE ELEMENTS  
SITE 2B - AREA A WETLAND  
NSB-NLON, GROTON, CONNECTICUT  
PAGE 2 OF 4**

DATA PACKAGE ELEMENTS	Semi-Volatiles /PAHs	Pesticides / PCBs	Metals	Miscellaneous <sup>(1)</sup>
- Sample preparation/extraction/digestion log (Inorg. Form XIII) and logbook pages	X	X	X	
- Sample analysis run log (Inorg. Form XIV) and logbook pages	X	X	X	
- ICP Raw Data			X	
- Furnace AA Raw Data				
- Mercury Raw Data			X	
- Cyanide Raw Data				
- Other Analytical Raw Data	X	X	X	X
<b>◆ STANDARDS DATA:</b>				
- Method Detection Limit Study Tabulated Summary Form	X	X	X	
- Initial Calibration Tabulated Summary Form (Org. Form VI, Inorg. Form IIA)	X	X	X	
- Continuing Calibration Tabulated Summary Form (Org. Form VII, Inorg. Form IIA)	X	X	X	
- RICs and quantitation reports for all GC/MS Standards	X			
- Pesticides Analyte Resolution tabulated Summary Form (Org. Form VI, Pest-4)		X		
- Pesticides Calibration Verification Tabulated Summary Form (Org. Form VII, Pest-1 and Pest-2)		X		
- Pesticide Analytical Sequence Tabulated Summary Form (Org. Form VIII-Pest)		X		
- GC Chromatograms and data system printouts for all GC standards		X		
- For Pesticides/Aroclors confirmed by GC/MS, copies of spectra for standards data		X		
- GPC Calibration Tabulated Summary Form (Org. Form IX, Pest-2)		X		
- Florisil Cartridge Check Tabulated Summary Form (Org. Form IX, Pest-1)		X		
- Instrument Detection Limits Tabulated Summary Form (Inorg. Form X)			X	
- ICP Interement Correction Factors Tabulated Summary Form (Inorg. Form XIA and XIB)			X	

TABLE 2-1

**LABORATORY DATA PACKAGE ELEMENTS  
SITE 2B - AREA A WETLAND  
NSB-NLON, GROTON, CONNECTICUT  
PAGE 3 OF 4**

DATA PACKAGE ELEMENTS	Semi-Volatiles /PAHs	Pesticides / PCBs	Metals	Miscellaneous <sup>(1)</sup>
- 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	
◆ <b>QC DATA:</b>				
- Tuning and Mass Calibration Tabulated Summary Form (Org. Form V)	X			
- Window defining mixture				
- Chromatographic resolution				
- Surrogate Percent Recovery Tabulated Summary Form (Org. Form II)	X	X		
- MS/MSD Recovery Tabulated Summary Form (Org. Form III)	X	X		
- Method Blank Tabulated Summary Form (Org. Form IV and Inorg. Form III)	X	X	X	
- Internal Standard Area and RT Tabulated Summary Form (Org. Form VIII)	X			
- Labeled Compound Recovery Summary Form				
- QC Raw Data – RICs, Chromatograms, Quan Reports, Integration Reports, Mass Spectra, etc.	X	X		
- Spike Sample Recovery Tabulated Summary Form (Inorg. Form IV)			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	
- 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	
- QC sample preparation logbook pages	X	X	X	

TABLE 2-1

LABORATORY DATA PACKAGE ELEMENTS  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 4 OF 4

DATA PACKAGE ELEMENTS	Semi-Volatiles /PAHs	Pesticides / PCBs	Metals	Miscellaneous <sup>(1)</sup>
- Toxicity Equivalence Factor				
- Compound Identification Criteria				
♦ <b>MISCELLANEOUS DATA:</b>				
- Original preparation and analysis forms or copies of preparation and analysis logbook pages	X	X	X	X
- Screening records	X	X	X	X
- All instrument output, including strip charts from screening activities	X	X	X	X
- Preparation Logs Raw Data	X	X	X	X
- Percent Solids Determination Log	X	X	X	X
- Other Records (ex. Telephone Communication Log)	X	X	X	X

(1) Miscellaneous data package will include information as applicable to the method.

### 3.0 ASSESSMENT AND OVERSIGHT

#### 3.1 ASSESSMENT AND RESPONSE ACTIONS

Assessment activities ensure that the data quality is adequate for the data's intended use 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 for this investigation are system audits and field audits and are identified in 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 PM 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 PM.
- The PM 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, external, or EPA.
- 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. 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 QA manager, will be responsible for developing and initiating appropriate corrective action through the FOL and verifying that the corrective action has been effective. Corrective actions may include the following: resampling and/or reanalyzing a sample or amending or adjusting project procedures. If warranted by the severity of the problem (for example, if a major change in the approved plan is required), the U.S. Navy will be notified in writing and the U.S. 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 corrected. 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 manager.

For quality assurance issues involving the analytical laboratory 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 appropriate personnel to be consistently aware of salient project activities and to provide opportunities for 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 in QAPP Worksheet #33 is summarized in the following sections.

#### **Field Status Reports**

The FOL will give verbal status reports to the PM 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 PM.

#### **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 updated RI/FS report.

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 updated RI/FS report. 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. Results and trends of PT sample analyses performed by all laboratories (per analytical group, matrix, and concentration level)
- Description and findings of assessments.
- Required corrective actions and effectiveness of corrective actions
- Limitations of the data usability

### **3.3 FINAL PROJECT REPORT**

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

## 4.0 DATA REVIEW

### 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. 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 (i.e., 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. One hundred percent of the environmental samples will be validated.

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 a completeness verification and will then perform data validation according to the most recent 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.
- Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM) (January 2006), and
- Test Methods for Evaluating Solid Waste: Physical/Chemical Methods (SW-846), 3<sup>rd</sup> Edition, up to and including Update III (1986).

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 noncompliances will be generated. Noncompliances, which may result in data qualification, 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 and is suitable for its intended use. 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 and laboratory data verification. Verification inputs as per QAPP Worksheet #34 will be checked by the DVM.

#### 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 USEPA Region 1 guidance. Step IIa validation procedures (i.e., compliance with methods, procedures and contracts) are discussed in Section 4.2.2.1. Step IIb validation procedures (i.e., comparison of analytical results to the MPCs documented in this QAPP) are discussed in Section 4.2.2.2.

##### 4.2.2.1 **Step IIa: Validation Activities**

Step IIa validation activities are documented in QAPP Worksheet #35. Data validation will be completed to ensure that the data are of evidentiary quality. Particular emphasis will be placed on holding time compliance, instrument calibration, laboratory control samples, matrix spike recoveries, and laboratory and field quality control blanks, although all required elements of the validation process will be considered.

One hundred percent of the laboratory data from chemical analyses will be validated. Validation of analytical data will be conducted by TtNUS. Final review and approval of validation deliverables will be completed for the DVM. All parameters will be reviewed using applicable sections of the aforementioned guidelines and the laboratory SOPs. Each analysis will be validated in accordance with 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 the PM and the U.S. Navy.

##### 4.2.2.2 **Step IIb: Validation Activities**

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

#### 4.2.3 Step III: Data Usability Assessment

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

After 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 statistical analyses will be performed to estimate several of the data set characteristics. The statistical evaluations will include simple summary statistics for target analytes, such as the maximum concentration, minimum concentration, number of samples exhibiting no detectable analyte, the number of samples exhibiting detectable analytes, and the proportion of samples with detectable and undetectable analytes. 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 "UR" and "R").
- Evaluate project assumptions.
- Characterize data set distributions (e.g., Shapiro-Wilk W test) if enough data are available.
- 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 percent 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 COC concentration detected at that sample location.

Statistical tests for outliers will be conducted using standard 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.

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 PM. 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 soils and sediments, one matrix group – solids – may be used when discussing biases. This grouping also causes 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.

#### 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 a corrective action will be devised as necessary. In general, RPD values (see Section 1.5.2.1) from laboratory and field duplicates will be used for this evaluation.

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.

#### 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 1.5.2.2) 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 percent will generally be considered acceptable. Biases between 40 and 75 percent or 125 to 160 percent will be considered to be moderate. Biases between 10 and 40 percent or 160 to 190 percent will generally be considered to be severe. Biases less than 10 percent or greater than 190 percent 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. Because analytical performance is generally similar for groundwater and surface water and for soils and sediments, two

matrix groups – solids and aqueous – may be used when discussing biases. These groupings also cause the number of samples within a group to be greater than when the matrices are discussed separately.

#### 4.2.3.1.3 Representativeness

Representativeness (see Section 1.5.2.3) will be evaluated by reviewing field logs and analytical results to verify that project requirements were satisfied 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. Representativeness 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 phase and suspended solids of a groundwater or surface water sample.

#### 4.2.3.1.4 Comparability

Comparability (see Section 1.5.2.4) 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.

#### 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 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.

## REFERENCES

Brown and Root Environmental, 1997a. Phase II Remedial Investigation Report for Naval Submarine Base - New London, Groton, Connecticut. Wayne, Pennsylvania. March.

Department of Defense (DOD), 2006. Department of Defense Quality Systems Manual (DOD QSM). January.

DOD, Department of Energy, U.S. Environmental Protection Agency (USEPA), 2005. Interagency Data Quality Task Force Uniform Federal Policy Quality Assurance Project Plan Manual. March.

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

Tetra Tech NUS, Inc. (TtNUS), 2007. Health and Safety Plan.

USEPA, 1989. Part 4 Inorganic Data Validation Functional Guidelines. February.

USEPA, 1996. Part 2 Volatile/Semivolatile Data Validation Functional Guidelines. December.

USEPA, 1996. Part 3 Pesticides/Polychlorinated Biphenyls Data Validation Functional Guidelines. December.

USEPA, 1999. Region I, USEPA-New England Quality Assurance Project Plan Manual. USEPA-New England Region I Quality Assurance Unit staff, Office of Environmental Measurement and Evaluation, Final.

USEPA, 2006. Guidance on Systematic Planning using the Data Quality Objectives Process. EPA QA/G-4, EPA/240/B-06/001. USEPA Office of Environmental Information, Washington DC. February.

**APPENDIX A**

**FIELD AND LABORATORY SOPS ON CD-ROM**

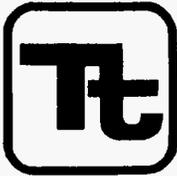
## LIST OF SOPs ON CD-ROM

### Field SOPs

- CT-04 Sample Nomenclature
- SA-6.3 Field Documentation
- SA-7.1 Decontamination of Field Equipment
- SA-6.1 Non-Radiological Sample Handling
- SA-1.2 Surface Water and Sediment Handling
- SA-1.3 Soil Sampling

### Laboratory SOPs

- CA-500 Preparation of Sediment/Soil Samples by Sonication Using Method 3550 for Subsequent Pesticides/PCBs Analysis
- CA-524 Preparation of Sediment/Soil Samples by Soxhlet Extraction Using Method 3540 for Pesticides/PCBs Analysis
- CA-741 Determination of Total Organic Carbon in Solids Using the EPA Region II Lloyd Kahn Method
- CA-512 Preparation of Sediment/Soil Samples by Sonication Using Method 3550 for Subsequent Extractable Semi-Volatile Analysis
- SD-902 Sample Receipt and Internal Control
- CA-526 Preparation of Sediment/Soil Samples by Sonication Using Method 3540 for Subsequent Extractable Semi-Volatile Analysis
- CA-213 Analysis of Semivolatile Organic Compounds by: SW 846 Method 8270 - Modified for Selected Ion Monitoring (SIM)
- CA-302 Analysis of Pesticides by Gas Chromatography/Electron Capture Detector (GC/ECD): SW-846 Method 8081
- CA-611 Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471
- CA-605 Acid Digestion of Solid Samples by USEPA Method 3050 for Metals Analysis by ICP-AES and GFAA
- CA-608 Trace Metals Analysis by ICP-AES Using USEPA Method 6010
- CA-604 Acid Digestion of Aqueous Samples by USEPA Method 3010 for ICP Analysis of Total or Dissolved Metals
- CA-329 Analysis of PCBs as Total Aroclors by Gas Chromatography/Electron Capture Detector (GC/ECD): SW-846 Method 842
- CA-615 Digestion and Analysis of Aqueous Samples for Mercury by USEPA Method 7470
- CA-502 Preparation of Aqueous Samples for Extractable Semivolatile Analysis Using the Extractable Portion of EPA Methods 625, 3510, and 3520
- CA-515 Preparation of Aqueous Samples for Pesticides/PCBs Analysis Using the Extractable Portion of EPA Methods 3510, 3520, and 3535A
- CA-70 pH Concentration Measurements in Soil Matrices - SW 846 Method 9045



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

Number	CT-04	Page	1 of 6
Effective Date	09/03	Revision	1
Applicability	Tetra Tech NUS, Inc.		
Prepared	Risk Assessment Department		
Approved	D. Senovich <i>ds</i>		

Subject  
SAMPLE NOMENCLATURE

## TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 PURPOSE.....	2
2.0 SCOPE.....	2
3.0 GLOSSARY .....	2
4.0 RESPONSIBILITIES.....	2
5.0 PROCEDURES.....	2
5.1 INTRODUCTION.....	2
5.2 SAMPLE IDENTIFICATION FIELD REQUIREMENTS.....	3
5.3 EXAMPLE SAMPLE FIELD DESIGNATIONS .....	4
5.4 EXAMPLES OF SAMPLE NOMENCLATURE .....	5
5.5 FIELD QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) SAMPLE NOMENCLATURE).....	6
5.6 EXAMPLES OF FIELD QA/QC SAMPLE NOMENCLATURE .....	6
6.0 DEVIATIONS .....	6

Subject  SAMPLE NOMENCLATURE	Number CT-04	Page 2 of 6
	Revision 1	Effective Date 09/03

## 1.0 PURPOSE

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

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

## 2.0 SCOPE

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

## 3.0 GLOSSARY

None.

## 4.0 RESPONSIBILITIES

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

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

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

## 5.0 PROCEDURES

### 5.1 Introduction

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

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

Subject  SAMPLE NOMENCLATURE	Number CT-04	Page 3 of 6
	Revision 1	Effective Date 09/03

Additional segments may be added as needed. For example:

(1) Soil and Sediment Sample ID

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

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

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

(3) Biota Sample ID

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

## 5.2 Sample Identification Field Requirements

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

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

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

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

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

The sample depth field is used to note the depth below ground surface (bgs) at which a soil or sediment sample is collected. The first two numbers of the four-number code specify the top interval, and the third and fourth specify the bottom interval in feet bgs of the sample. If the sample depth is equal to or greater than 100, then only the top interval would be represented and the sampling depth would be truncated to

Subject  SAMPLE NOMENCLATURE	Number CT-04	Page 4 of 6
	Revision 1	Effective Date 09/03

three-characters. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet, boring log, logbook, etc.

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

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

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

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

### 5.3 Example Sample Field Designations

Examples of each of the fields are as follows:

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

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

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

Sample Type - Examples of sample types are as follows:

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

Subject  SAMPLE NOMENCLATURE	Number CT-04	Page 5 of 6
	Revision 1	Effective Date 09/03

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

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

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

Species Identifier - Examples of species identifier are as follows:

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

#### 5.4 Examples of Sample Nomenclature

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

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

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

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

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

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

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

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

Subject  SAMPLE NOMENCLATURE	Number CT-04	Page 6 of 6
	Revision 1	Effective Date 09/03

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

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

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

The QC types are identified as:

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

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

### 5.6 Examples of Field QA/QC Sample Nomenclature

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

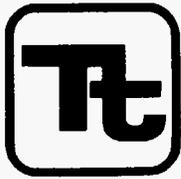
The third duplicate of the day taken of a subsurface soil sample collected on November 17, 2003 would be designated as FD11170303.

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

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

### 6.0 **DEVIATIONS**

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



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

Number SA-6.3	Page 1 of 12
Effective Date 09/03	Revision 2
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved D. Senovich <i>ds</i>	

Subject  
FIELD DOCUMENTATION

## TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 PURPOSE.....	2
2.0 SCOPE.....	2
3.0 GLOSSARY .....	2
4.0 RESPONSIBILITIES.....	2
5.0 PROCEDURES .....	2
5.1 SITE LOGBOOK .....	2
5.1.1 General.....	2
5.1.2 Photographs.....	3
5.2 FIELD NOTEBOOKS .....	3
5.3 FIELD FORMS .....	4
5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results..	4
5.3.2 Hydrogeological and Geotechnical Forms .....	5
5.3.3 Equipment Calibration and Maintenance Form .....	6
5.4 FIELD REPORTS.....	6
5.4.1 Daily Activities Report.....	6
5.4.2 Weekly Status Reports.....	7
6.0 LISTING OF TETRA TECH NUS FIELD FORMS FOUND ON THE TTNUS INTRANET SITE. <u><a href="http://intranet.ttnus.com">HTTP://INTRANET.TTNUS.COM</a> CLICK ON FIELD LOG SHEETS.....</u>	7

### ATTACHMENTS

A	TYPICAL SITE LOGBOOK ENTRY .....	9
B	SAMPLE LABEL.....	10
C	CHAIN-OF-CUSTODY RECORD FORM.....	11
D	CHAIN-OF-CUSTODY SEAL .....	12

Subject  FIELD DOCUMENTATION	Number SA-6.3	Page 2 of 12
	Revision 2	Effective Date 09/03

## 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

Subject  FIELD DOCUMENTATION	Number SA-6.3	Page 3 of 12
	Revision 2	Effective Date 09/03

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.

Subject  FIELD DOCUMENTATION	Number SA-6.3	Page 4 of 12
	Revision 2	Effective Date 09/03

### 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.

Subject  FIELD DOCUMENTATION	Number SA-6.3	Page 5 of 12
	Revision 2	Effective Date 09/03

#### 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.

Subject  FIELD DOCUMENTATION	Number SA-6.3	Page 6 of 12
	Revision 2	Effective Date 09/03

#### 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 collected, 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.

Subject  FIELD DOCUMENTATION	Number SA-6.3	Page 7 of 12
	Revision 2	Effective Date 09/03

#### 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

Subject  FIELD DOCUMENTATION	Number SA-6.3	Page 8 of 12
	Revision 2	Effective Date 09/03

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

Subject  FIELD DOCUMENTATION	Number SA-6.3	Page 9 of 12
	Revision 2	Effective Date 09/03

**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 jenny 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

Subject  FIELD DOCUMENTATION	Number SA-6.3	Page 10 of 12
	Revision 2	Effective Date 09/03

**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:	

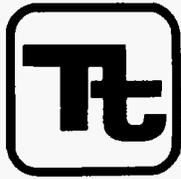


Subject  FIELD DOCUMENTATION	Number SA-6.3	Page 12 of 12
	Revision 2	Effective Date 09/03

ATTACHMENT D

CHAIN-OF-CUSTODY SEAL

<b>Signature</b> <hr/> <b>Date</b> <hr/> <b>CUSTODY SEAL</b>		<b>CUSTODY SEAL</b> <hr/> <b>Date</b> <hr/> <b>Signature</b>
--	--	--



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

Number	SA-7.1	Page	1 of 8
Effective Date	09/03	Revision	3
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	D. Senovich <i>ds</i>		

Subject DECONTAMINATION OF FIELD EQUIPMENT

## TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 PURPOSE.....	2
2.0 SCOPE.....	2
3.0 GLOSSARY .....	2
4.0 RESPONSIBILITIES.....	3
5.0 PROCEDURES.....	3
5.1 DECONTAMINATION DESIGN/CONSTRUCTIONS CONSIDERATIONS .....	3
5.1.1 Temporary Decontamination Pads.....	3
5.1.2 Decontamination Activities at Drill Rigs/DPT Units .....	4
5.1.3 Decontamination Activities at Remote Sample Locations.....	5
5.2 EQUIPMENT DECONTAMINATION PROCEDURES .....	5
5.2.1 Monitoring Well Sampling Equipment .....	5
5.2.2 Down-Hole Drilling Equipment .....	6
5.2.3 Soil/Sediment Sampling Equipment.....	6
5.3 CONTACT WASTE/MATERIALS .....	7
5.3.1 Decontamination Solutions.....	7
5.4 DECONTAMINATION EVALUATION .....	7

Subject DECONTAMINATION OF FIELD EQUIPMENT	Number SA-7.1	Page 2 of 8
	Revision 3	Effective Date 09/03

## 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.

Subject DECONTAMINATION OF FIELD EQUIPMENT	Number SA-7.1	Page 3 of 8
	Revision 3	Effective Date 09/03

#### 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

Subject DECONTAMINATION OF FIELD EQUIPMENT	Number SA-7.1	Page 4 of 8
	Revision 3	Effective Date 09/03

- 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.

Subject DECONTAMINATION OF FIELD EQUIPMENT	Number SA-7.1	Page 5 of 8
	Revision 3	Effective Date 09/03

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.

---

\* - 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.

Subject DECONTAMINATION OF FIELD EQUIPMENT	Number SA-7.1	Page 6 of 8
	Revision 3	Effective Date 09/03

- 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.

Subject DECONTAMINATION OF FIELD EQUIPMENT	Number SA-7.1	Page 7 of 8
	Revision 3	Effective Date 09/03

- 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.

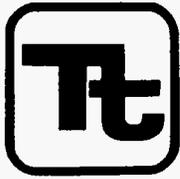
Subject DECONTAMINATION OF FIELD EQUIPMENT	Number SA-7.1	Page 8 of 8
	Revision 3	Effective Date 09/03

- 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.



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

Number	SA-6.1	Page	1 of 11
Effective Date	02/04	Revision	3
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	D. Senovich <i>[Signature]</i>		

Subject  
NON-RADIOLOGICAL SAMPLE HANDLING

## TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 PURPOSE.....	2
2.0 SCOPE.....	2
3.0 GLOSSARY .....	2
4.0 RESPONSIBILITIES.....	3
5.0 PROCEDURES.....	3
5.1 SAMPLE CONTAINERS.....	3
5.2 SAMPLE PRESERVATION.....	3
5.2.1 Overview .....	4
5.2.2 Preparation and Addition of Reagents .....	4
5.3 FIELD FILTRATION.....	5
5.4 SAMPLE PACKAGING AND SHIPPING.....	6
5.4.1 Environmental Samples .....	6
6.0 REFERENCES.....	7
 <u>ATTACHMENTS</u>	
A GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS.....	8
B ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES.....	9

Subject NON-RADIOLOGICAL SAMPLE HANDLING	Number SA-6.1	Page 2 of 11
	Revision 3	Effective Date 02/04

## 1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide information on sample preservation, packaging, and shipping procedures to be used in handling environmental samples submitted for chemical constituent, biological, or geotechnical analysis. Sample chain-of-custody procedures and other aspects of field documentation are addressed in SOP SA-6.3. Sample identification is addressed in SOP CT-04.

## 2.0 SCOPE

This procedure describes the appropriate containers to be used for samples depending on the analyses to be performed, and the steps necessary to preserve the samples when shipped off site for chemical analysis.

## 3.0 GLOSSARY

Hazardous Material - A substance or material which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. Under 49 CFR, the term includes hazardous substances, hazardous wastes, marine pollutants, and elevated temperature materials, as well as materials designated as hazardous under the provisions of §172.101 and §172.102 and materials that meet the defining criteria for hazard classes and divisions in Part 173. With slight modifications, IATA has adopted DOT "hazardous materials" as IATA "Dangerous Goods."

Hazardous Waste - Any substance listed in 40 CFR, Subpart D (y261.30 et seq.), or otherwise characterized as ignitable, corrosive, reactive, or toxic (as defined by Toxicity Characteristic Leaching Procedure, TCLP, analysis) as specified under 40 CFR, Subpart C (y261.20 et seq.), that would be subject to manifest requirements specified in 40 CFR 262. Such substances are defined and regulated by EPA.

Marking - A descriptive name, identification number, instructions, cautions, weight, specification or UN marks, or combination thereof required on outer packaging of hazardous materials.

n.o.i - Not otherwise indicated (may be used interchangeably with n.o.s.).

n.o.s. - Not otherwise specified.

Packaging - A receptacle and any other components or materials necessary for compliance with the minimum packaging requirements of 49 CFR 174, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank-car tanks to perform a containment function in conformance with the minimum packaging requirements of 49 CFR 173.24(a) & (b).

Placard - Color-coded, pictorial sign which depicts the hazard class symbol and name and which is placed on the side of a vehicle transporting certain hazardous materials.

### Common Preservatives:

- Hydrochloric Acid - HCl
- Sulfuric Acid - H<sub>2</sub>SO<sub>4</sub>
- Nitric Acid - HNO<sub>3</sub>
- Sodium Hydroxide - NaOH

Subject NON-RADIOLOGICAL SAMPLE HANDLING	Number SA-6.1	Page 3 of 11
	Revision 3	Effective Date 02/04

#### Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>

Normality (N) - Concentration of a solution expressed as equivalent per liter, an equivalent being the amount of a substance containing 1 gram-atom of replaceable hydrogen or its equivalent.

Reportable Quantity (RQ) - For the purposes of this SOP, means the quantity specified in column 3 of the Appendix to DOT 49 CFR §172.101 for any material identified in column 1 of the appendix. A spill greater than the amount specified must be reported to the National Response Center.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the location and time of collection.

#### **4.0 RESPONSIBILITIES**

Field Operations Leader - Directly responsible for the bottling, preservation, labeling, packaging, shipping, and custody of samples up to and including release to the shipper.

Field Samplers - Responsible for initiating the Chain-of-Custody Record (per SOP SA-6.3), implementing the packaging and shipping requirements, and maintaining custody of samples until they are relinquished to another custodian or to the shipper.

#### **5.0 PROCEDURES**

Sample identification, labeling, documentation, and chain-of-custody are addressed by SOP SA-6.3.

##### **5.1 Sample Containers**

Different types of chemicals react differently with sample containers made of various materials. For example, trace metals adsorb more strongly to glass than to plastic, whereas many organic chemicals may dissolve various types of plastic containers. Attachments A and B show proper containers (as well as other information) per 40 CFR 136. In general, the sample container shall allow approximately 5-10 percent air space ("ullage") to allow for expansion/vaporization if the sample warms during transport. However, for collection of volatile organic compounds, head space shall be omitted. The analytical laboratory will generally provide certified-clean containers for samples to be analyzed for chemical constituents. Shelby tubes or other sample containers are generally provided by the driller for samples requiring geotechnical analysis. Sufficient lead time shall be allowed for a delivery of sample container orders. Therefore, it is critical to use the correct container to maintain the integrity of the sample prior to analysis.

Once opened, the container must be used at once for storage of a particular sample. Unused but opened containers are to be considered contaminated and must be discarded. Because of the potential for introduction of contamination, they cannot be reclosed and saved for later use. Likewise, any unused containers which appear contaminated upon receipt, or which are found to have loose caps or a missing Teflon liner (if required for the container), shall be discarded.

##### **5.2 Sample Preservation**

Many water and soil samples are unstable and therefore require preservation to prevent changes in either the concentration or the physical condition of the constituent(s) requiring analysis. Although complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological

Subject NON-RADIOLOGICAL SAMPLE HANDLING	Number SA-6.1	Page 4 of 11
	Revision 3	Effective Date 02/04

changes that inevitably take place after the sample is collected. Preservation techniques are usually limited to pH control, chemical addition(s), and refrigeration/ freezing (certain biological samples only).

### 5.2.1 Overview

The preservation techniques to be used for various analytes are listed in Attachments A and B. Reagents required for sample preservation will either be added to the sample containers by the laboratory prior to their shipment to the field or be added in the field (in a clean environment). Only high purity reagents shall be used for preservation. In general, aqueous samples of low-concentration organics (or soil samples of low- or medium-concentration organics) are cooled to 4°C. Medium-concentration aqueous samples, high-hazard organic samples, and some gas samples are typically not preserved. Low-concentration aqueous samples for metals are acidified with HNO<sub>3</sub>, whereas medium-concentration and high-hazard aqueous metal samples are not preserved. Low- or medium-concentration soil samples for metals are cooled to 4°C, whereas high-hazard samples are not cooled.

The following subsections describe the procedures for preparing and adding chemical preservatives. Attachments A and B indicate the specific analytes which require these preservatives.

The FOL is responsible for ensuring that an accurate Chemical Inventory is created and maintained for all hazardous chemicals brought to the work site (see Section 5 of the TtNUS Health and Safety Guidance Manual). Furthermore, the FOL must ensure that a corresponding Material Safety Data Sheet (MSDS) is collected for every substance entered on the site Chemical Inventory, and that all persons using/handling/ disposing of these substances review the appropriate MSDS for substances they will work with. The Chemical Inventory and the MSDSs must be maintained at each work site in a location and manner where they are readily-accessible to all personnel.

### 5.2.2 Preparation and Addition of Reagents

Addition of the following acids or bases may be specified for sample preservation; these reagents shall be analytical reagent (AR) grade or purer and shall be diluted to the required concentration with deionized water before field sampling commences. To avoid uncontrolled reactions, be sure to Add Acid to water (not vice versa). A dilutions guide is provided below.

Acid/Base	Dilution	Concentration	Estimated Amount Required for Preservation
Hydrochloric Acid (HCl)	1 part concentrated HCl: 1 part double-distilled, deionized water	6N	5-10 mL
Sulfuric Acid (H <sub>2</sub> SO <sub>4</sub> )	1 part concentrated H <sub>2</sub> SO <sub>4</sub> : 1 part double-distilled, deionized water	18N	2 - 5 mL
Nitric Acid (HNO <sub>3</sub> )	Undiluted concentrated HNO <sub>3</sub>	16N	2 - 5 mL
Sodium Hydroxide (NaOH)	400 grams solid NaOH dissolved in 870 mL double-distilled, deionized water; yields 1 liter of solution	10N	2 mL

The amounts required for preservation shown in the above table assumes proper preparation of the preservative and addition of the preservative to one liter of aqueous sample. This assumes that the sample is initially at pH 7, is poorly buffered, and does not contain particulate matter; as these conditions vary, more preservative may be required. Consequently, the final sample pH must be checked using narrow-range pH paper, as described in the generalized procedure detailed below:

Subject NON-RADIOLOGICAL SAMPLE HANDLING	Number SA-6.1	Page 5 of 11
	Revision 3	Effective Date 02/04

- Pour off 5-10 mL of sample into a dedicated, clean container. Use some of this sample to check the initial sample pH using wide range (0-14) pH paper. Never dip the pH paper into the sample; always apply a drop of sample to the pH paper using a clean stirring rod or pipette.
- Add about one-half of the estimated preservative required to the original sample bottle. Cap and invert gently several times to mix. Check pH (as described above) using medium range pH paper (pH 0-6 or pH 7.5-14, as applicable).
- Cap sample bottle and seal securely.

Additional considerations are discussed below:

- To test if ascorbic acid must be used to remove oxidizing agents present in the sample before it can be properly preserved, place a drop of sample on KI-starch paper. A blue color indicates the need for ascorbic acid addition.

If required, add a few crystals of ascorbic acid to the sample and retest with the KI-starch paper. Repeat until a drop of sample produces no color on the KI-starch paper. Then add an additional 0.6 grams of ascorbic acid per each liter of sample volume.

Continue with proper base preservation of the sample as described above.

- Samples for sulfide analysis must be treated by the addition of 4 drops (0.2 mL) of 2N zinc acetate solution per 100 ml of sample.

The 2N zinc acetate solution is made by dissolving 220 grams of zinc acetate in 870 mL of double-distilled, deionized water to make 1 liter of solution.

The sample pH is then raised to 9 using the NaOH preservative.

- Sodium thiosulfate must be added to remove residual chlorine from a sample. To test the sample for residual chlorine use a field test kit specially made for this purpose.

If residual chlorine is present, add 0.08 grams of sodium thiosulfate per liter of sample to remove the residual chlorine.

Continue with proper acidification of the sample as described above.

For biological samples, 10% buffered formalin or isopropanol may also be required for preservation. Questions regarding preservation requirements should be resolved through communication with the laboratory before sampling begins.

### 5.3 Field Filtration

At times, field-filtration may be required to provide for the analysis of dissolved chemical constituents. Field-filtration must be performed prior to the preservation of samples as described above. General procedures for field filtration are described below:

- The sample shall be filtered through a non-metallic, 0.45-micron membrane filter, immediately after collection. The filtration system shall consist of dedicated filter canister, dedicated tubing, and a peristaltic pump with pressure or vacuum pumping squeeze action (since the sample is filtered by mechanical peristalsis, the sample travels only through the tubing).

Subject NON-RADIOLOGICAL SAMPLE HANDLING	Number SA-6.1	Page 6 of 11
	Revision 3	Effective Date 02/04

- To perform filtration, thread the tubing through the peristaltic pump head. Attach the filter canister to the discharge end of the silicon tubing (note flow direction arrow); attach the aqueous sample container to the intake end of the silicon tubing. Turn the peristaltic pump on and perform filtration. Run approximately 100 ml of sample through the filter and discard prior to sample collection.
- Continue by preserving the filtrate (contained in the filter canister), as applicable and generally described above.

#### **5.4 Sample Packaging and Shipping**

Only employees who have successfully completed the TtNUS "Shipping Hazardous Materials" training course are authorized to package and ship hazardous substances. These trained individuals are responsible for performing shipping duties in accordance with this training.

Samples collected for shipment from a site shall be classified as either environmental or hazardous material samples. Samples from drums containing materials other than Investigative Derived Waste (IDW) and samples obtained from waste piles or bulk storage tanks are generally shipped as hazardous materials. A distinction must be made between the two types of samples in order to:

- Determine appropriate procedures for transportation of samples (if there is any doubt, a sample shall be considered hazardous and shipped accordingly.)
- Protect the health and safety of transport and laboratory personnel receiving the samples (special precautions are used by the shipper and at laboratories when hazardous materials are received.)

Detailed procedures for packaging environmental samples are outlined in the remainder of this section.

##### **5.4.1 Environmental Samples**

Environmental samples are packaged as follows:

- Place properly identified sample container, with lid securely fastened, in a plastic bag (e.g. Ziploc baggie), and seal the bag.
- Place sample in a cooler constructed of sturdy material which has been lined with a large, plastic bag (e.g. "garbage" bag). Drain plugs on coolers must be taped shut.
- Pack with enough cushioning materials such as bubble wrap (shoulders of bottles must be iced if required) to minimize the possibility of the container breaking.
- If cooling is required (see Attachments A and B), place ice around sample container shoulders, and on top of packing material (minimum of 8 pounds of ice for a medium-size cooler).
- Seal (i.e., tape or tie top in knot) large liner bag.
- The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing the vials for VOC analysis. The COC form should then state how many coolers are included with that shipment.
- Close and seal outside of cooler as described in SOP SA-6.3. Signed custody seals must be used.

Subject NON-RADIOLOGICAL SAMPLE HANDLING	Number SA-6.1	Page 7 of 11
	Revision 3	Effective Date 02/04

Coolers must be marked as containing "Environmental Samples." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT marking or labeling is required; there are no DOT restrictions on mode of transportation.

## 6.0 REFERENCES

American Public Health Association, 1981. Standard Methods for the Examination of Water and Wastewater, 15th Edition. APHA, Washington, D.C.

International Air Transport Association (latest issue). Dangerous Goods Regulations, Montreal, Quebec, Canada.

U.S. Department of Transportation (latest issue). Hazardous Materials Regulations, 49 CFR 171-177.

U.S. EPA, 1984. "Guidelines Establishing Test Procedures for the Analysis of Pollutants under Clean Water Act." Federal Register, Volume 49 (209), October 26, 1984, p. 43234.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020, U.S. EPA-EMSL, Cincinnati, Ohio.

Subject <b>NON-RADIOLOGICAL SAMPLE HANDLING</b>	Number <b>SA-6.1</b>	Page <b>8 of 11</b>
	Revision <b>3</b>	Effective Date <b>02/04</b>

### ATTACHMENT A

#### GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

Sample Type and Concentration	Container <sup>(1)</sup>	Sample Size	Preservation <sup>(2)</sup>	Holding Time <sup>(2)</sup>
-------------------------------	--------------------------	-------------	-----------------------------	-----------------------------

#### WATER

Organics (GC&GC/MS)	VOC	Low	Borosilicate glass	2 x 40 mL	Cool to 4°C HCl to ≤ 2	14 days <sup>(9)</sup>
	Extractables SVOCs and pesticide/PCBs)	(Low	Amber glass	2x2 L or 4x1 L	Cool to 4°C	7 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticide/PCBs)	(Medium	Amber glass	2x2 L or 4x1 L	None	7 days to extraction; 40 days after extraction
Inorganics	Metals	Low	High-density polyethylene	1 L	HNO <sub>3</sub> to pH ≤ 2	6 months (Hg-28 days)
		Medium	Wide-mouth glass	16 oz.	None	6 months
	Cyanide	Low	High-density polyethylene	1 L	NaOH to pH>12	14 days
	Cyanide	Medium	Wide-mouth glass	16 oz.	None	14 days
Organic/ Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	14 days

#### SOIL

Organics (GC&GC/MS)	VOC		EnCore Sampler	(3) 5 g Samplers	Cool to 4°C	48 hours to lab preservation
	Extractables SVOCs and pesticides/PCBs)	(Low	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticides/PCBs)	(Medium	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
Inorganics	Low/Medium		Wide-mouth glass	8 oz.	Cool to 4°C	6 months (Hg - 28 days) Cyanide (14 days)
Organic/Inorga nic	High Hazard		Wide-mouth glass	8 oz.	None	NA
Dioxin/Furan	All		Wide-mouth glass	4 oz.	None	35 days until extraction; 40 days after extraction
TCLP	All		Wide-mouth glass	8 oz.	None	7 days until preparation; analysis as per fraction

#### AIR

Volatile Organics	Low/Medium		Charcoal tube -- 7 cm long, 6 mm OD, 4 mm ID	100 L air	Cool to 4°C	5 days recommended
----------------------	------------	--	---	-----------	-------------	--------------------

1 All glass containers should have Teflon cap liners or septa.

2 See Attachment E. Preservation and maximum holding time allowances per 40 CFR 136.

Subject <b>NON-RADIOLOGICAL SAMPLE HANDLING</b>	Number <b>SA-6.1</b>	Page <b>9 of 11</b>
	Revision <b>3</b>	Effective Date <b>02/04</b>

**ATTACHMENT B**

**ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES,  
AND HOLDING TIMES**

Parameter Number/Name	Container <sup>(1)</sup>	Preservation <sup>(2)(3)</sup>	Maximum Holding Time <sup>(4)</sup>
-----------------------	--------------------------	--------------------------------	-------------------------------------

**INORGANIC TESTS:**

Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days
Ammonia - Nitrogen	P, G	Cool, 4°C; H <sub>2</sub> SO <sub>4</sub> to pH 2	28 days
Biochemical Oxygen Demand (BOD)	P, G	Cool, 4°C	48 hours
Bromide	P, G	None required	28 days
Chemical Oxygen Demand (COD)	P, G	Cool, 4°C; H <sub>2</sub> SO <sub>4</sub> to pH 2	28 days
Chloride	P, G	None required	28 days
Chlorine, Total Residual	P, G	None required	Analyze immediately
Color	P, G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P, G	Cool, 4°C; NaOH to pH 12; 0.6 g ascorbic acid <sup>(5)</sup>	14 days <sup>(6)</sup>
Fluoride	P	None required	28 days
Hardness	P, G	HNO <sub>3</sub> to pH 2; H <sub>2</sub> SO <sub>4</sub> to pH 2	6 months
Total Kjeldahl and Organic Nitrogen	P, G	Cool, 4°C; H <sub>2</sub> SO <sub>4</sub> to pH 2	28 days
Nitrate - Nitrogen	P, G	None required	48 hours
Nitrate-Nitrite - Nitrogen	P, G	Cool, 4°C; H <sub>2</sub> SO <sub>4</sub> to pH 2	28 days
Nitrite - Nitrogen	P, G	Cool, 4°C	48 hours
Oil & Grease	G	Cool, 4°C; H <sub>2</sub> SO <sub>4</sub> to pH 2	28 days
Total Organic Carbon (TOC)	P, G	Cool, 4°C; HCl or H <sub>2</sub> SO <sub>4</sub> to pH 2	28 days
Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
Oxygen, Dissolved-Probe	G Bottle & top	None required	Analyze immediately
Oxygen, Dissolved-Winkler	G Bottle & top	Fix on site and store in dark	8 hours
Phenols	G	Cool, 4°C; H <sub>2</sub> SO <sub>4</sub> to pH 2	28 days
Phosphorus, Total	P, G	Cool, 4°C; H <sub>2</sub> SO <sub>4</sub> to pH 2	28 days
Residue, Total	P, G	Cool, 4°C	7 days
Residue, Filterable (TDS)	P, G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days
Residue, Settleable	P, G	Cool, 4°C	48 hours
Residue, Volatile (Ash Content)	P, G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific Conductance	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days

Subject NON-RADIOLOGICAL SAMPLE HANDLING	Number SA-6.1	Page 10 of 11
	Revision 3	Effective Date 02/04

**ATTACHMENT B  
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES,  
AND HOLDING TIMES  
PAGE TWO**

Parameter Number/Name	Container <sup>(1)</sup>	Preservation <sup>(2)(3)</sup>	Maximum Holding Time <sup>(4)</sup>
-----------------------	--------------------------	--------------------------------	-------------------------------------

**INORGANIC TESTS (Cont'd):**

Sulfide	P, G	Cool, 4°C; add zinc acetate plus sodium hydroxide to pH 9	7 days
Sulfite	P, G	None required	Analyze immediately
Turbidity	P, G	Cool, 4°C	48 hours

**METALS:<sup>(7)</sup>**

Chromium VI (Hexachrome)	P, G	Cool, 4°C	24 hours
Mercury (Hg)	P, G	HNO <sub>3</sub> to pH 2	28 days
Metals, except Chromium VI and Mercury	P, G	HNO <sub>3</sub> to pH 2	6 months

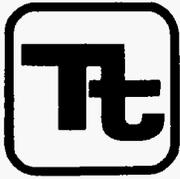
**ORGANIC TESTS:<sup>(8)</sup>**

Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup>	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup> HCl to pH 2 <sup>(9)</sup>	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4°C; 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup> adjust pH to 4-5 <sup>(10)</sup>	14 days
Phenols <sup>(11)</sup>	G, Teflon-lined cap	Cool, 4°C; 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup>	7 days until extraction; 40 days after extraction
Benzidines <sup>(11), (12)</sup>	G, Teflon-lined cap	Cool, 4°C; 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup>	7 days until extraction <sup>(13)</sup>
Phthalate esters <sup>(11)</sup>	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitrosamines <sup>(11), (14)</sup>	G, Teflon-lined cap	Cool, 4°C; store in dark; 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup>	7 days until extraction; 40 days after extraction
PCBs <sup>(11)</sup>	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitroaromatics & Isophorone <sup>(11)</sup>	G, Teflon-lined cap	Cool, 4°C; 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup> ; store in dark	7 days until extraction; 40 days after extraction
Polynuclear Aromatic Hydrocarbons (PAHs) <sup>(11), (14)</sup>	G, Teflon-lined cap	Cool, 4°C; 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup> ; store in dark	7 days until extraction; 40 days after extraction
Haloethers <sup>(11)</sup>	G, Teflon-lined cap	Cool, 4°C; 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup>	7 days until extraction; 40 days after extraction
Dioxin/Furan (TCDD/TCDF) <sup>(11)</sup>	G, Teflon-lined cap	Cool, 4°C; 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup>	7 days until extraction; 40 days after extraction

Subject NON-RADIOLOGICAL SAMPLE HANDLING	Number SA-6.1	Page 11 of 11
	Revision 3	Effective Date 02/04

**ATTACHMENT B  
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES,  
AND HOLDING TIMES  
PAGE THREE**

- (1) Polyethylene (P): generally 500 ml or Glass (G): generally 1L.
- (2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
- (3) When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172).
- (4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer periods, and has received a variance from the Regional Administrator.
- (5) Should only be used in the presence of residual chlorine.
- (6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments are made to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
- (7) Samples should be filtered immediately on site before adding preservative for dissolved metals.
- (8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
- (9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.
- (10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
- (11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).
- (12) If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.
- (13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
- (14) For the analysis of diphenylnitrosamine, add 0.008% Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> and adjust pH to 7-10 with NaOH within 24 hours of sampling.
- (15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

Number	SA-1.2	Page	1 of 12
Effective Date	09/03	Revision	5
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	D. Senovich <i>ds</i>		

Subject  
SURFACE WATER AND SEDIMENT SAMPLING

## TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 PURPOSE.....	2
2.0 SCOPE.....	2
3.0 GLOSSARY .....	2
4.0 RESPONSIBILITIES.....	2
5.0 PROCEDURES.....	2
5.1 INTRODUCTION.....	2
5.2 DEFINING THE SAMPLING PROGRAM.....	3
5.2.1 Sampling Program Objectives.....	3
5.2.2 Location of Sampling Stations.....	3
5.2.3 Frequency of Sampling .....	4
5.3 SURFACE WATER SAMPLE COLLECTION .....	4
5.3.1 Streams, Rivers, Outfalls and Drainage Features (Ditches, Culverts).....	4
5.3.2 Lakes, Ponds and Reservoirs .....	5
5.3.3 Estuaries .....	5
5.3.4 Surface Water Sampling Equipment.....	6
5.3.5 Surface Water Sampling Techniques .....	7
5.4 ONSITE WATER QUALITY TESTING.....	8
5.5 SEDIMENT SAMPLING .....	8
5.5.1 General.....	8
5.5.2 Sampling Equipment and Techniques .....	9
6.0 REFERENCES.....	10
 <u>ATTACHMENTS</u>	
A SURFACE WATER SAMPLE LOG SHEET.....	11
B SOIL & SEDIMENT SAMPLE LOG SHEET .....	12

Subject SURFACE WATER AND SEDIMENT SAMPLING	Number SA-1.2	Page 2 of 12
	Revision 5	Effective Date 09/03

## 1.0 PURPOSE

This procedure describes methods and equipment commonly used for collecting environmental samples of surface water and aquatic sediment for either onsite examination and chemical testing, or for subsequent laboratory analysis.

## 2.0 SCOPE

The information presented in this guideline is generally 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

Environmental Sample - a sample containing (or suspected to contain) low-level concentrations of contaminants, which does not require special handling or transport considerations as detailed in SOP SA-6.1.

Hazardous Waste Sample - a sample containing (or suspected to contain) higher concentrations of contaminants thus requiring special handling and/or transport considerations per SOP SA-6.1.

## 4.0 RESPONSIBILITIES

Project Manager - The Project Manager has the overall responsibility for seeing that all surface water and sediment sampling activities are properly conducted by appropriately trained personnel.

Field Operations Leader - The Field Operations Leader (FOL) is responsible for the supervision of onsite water quality analyses, ensuring proper sample collection, handling, and the completion and accuracy of all field documentation, and making sure that custody of all samples obtained is maintained according to proper procedures.

## 5.0 PROCEDURES

### 5.1 Introduction

Collecting a representative sample from surface water or sediments is difficult because of water movement, stratification, or patchiness. To collect representative samples, one must standardize sampling bias related to site selection, sampling frequency, sample collection, sampling devices, and sample handling, preservation, and identification.

Representativeness is 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 taken. 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.

Subject SURFACE WATER AND SEDIMENT SAMPLING	Number SA-1.2	Page 3 of 12
	Revision 5	Effective Date 09/03

## 5.2 Defining the Sampling Program

Many factors must be considered in developing a sampling program for surface water or sediments including study objectives, accessibility, site topography, physical characteristics of the water body (such as flow and mixing), point and diffuse sources of contamination, and personnel and equipment available to conduct the study. For waterborne constituents, dispersion depends on the vertical and lateral mixing within the body of water. For sediments, dispersion depends on bottom current or flow characteristics, sediment characteristics (density, size) and geochemical properties (which affect adsorption/desorption). The hydrogeologist developing the sampling plan must therefore know not only the mixing characteristics of streams and lakes, but also must understand the role of fluvial-sediment transport, deposition, and chemical sorption.

### 5.2.1 Sampling Program Objectives

The objective of surface water sampling is to determine the surface water quality entering, leaving or remaining within the site. 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., spills). The major pathways for surface water contamination (not including airborne deposition) are overland runoff, leachate influx to the waterbody, 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) which encompass 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 location 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 are: (1) move the site far enough downstream to allow for adequate mixing, or (2) collect integrated samples in a cross section. Also, nonhomogeneous 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 the 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.

### 5.2.2 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

Subject <b>SURFACE WATER AND SEDIMENT SAMPLING</b>	Number <b>SA-1.2</b>	Page 4 of 12
	Revision <b>5</b>	Effective Date <b>09/03</b>

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 and reservoirs, as well as those on 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.

Sampling in marshes or tidal areas may require the use of an all-terrain vehicle (ATV). The same precautions mentioned above with regard to sediment disturbance apply.

Under ideal and uniform contaminant dispersion conditions in a flowing stream, the same concentrations of each 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 in order to ensure, as nearly as possible, that samples are taken where uniform flow or deposition and good mixing conditions exist.

The availability of streamflow and sediment discharge records can be an important consideration in choosing sampling sites in streams. Streamflow 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 streamflow data by direct or indirect methods.

**5.2.3 Frequency of Sampling**

The sampling frequency and the objectives of the sampling event will be defined by the project plan 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 the 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 shall 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 shall 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.

**5.3 Surface Water Sample Collection**

**5.3.1 Streams, Rivers, Outfalls and Drainage Features (Ditches, Culverts)**

Methods for sampling streams, rivers, outfalls, and drainage features 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.

Subject <b>SURFACE WATER AND SEDIMENT SAMPLING</b>	Number <b>SA-1.2</b>	Page <b>5 of 12</b>
	Revision <b>5</b>	Effective Date <b>09/03</b>

Generally, the number and type of samples to be taken depend on the river's width, depth, 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 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 plan documents.

### **5.3.2 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, the 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. These verticals are often taken 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 which is exposed to the atmosphere. The hypolimnion is the lower, "confined" layer which 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 verticals 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 since 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, dissolved oxygen, some cations and anions, and light penetration.

### **5.3.3 Estuaries**

Estuarine areas are by definition, zones where inland freshwaters (both surface and ground) mix with oceanic saline waters. Estuaries are generally categorized into three types dependent upon freshwater inflow and mixing properties. Knowledge of the estuary type is necessary to determine sampling locations. Each type of estuarine area is described below:

Subject SURFACE WATER AND SEDIMENT SAMPLING	Number SA-1.2	Page 6 of 12
	Revision 5	Effective Date 09/03

- 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 sheetflow areas. Being well mixed, the sampling locations are not critical in this type of estuary.
- 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 upon the tidal phases, 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 dissolved oxygen and temperature profiles.

#### 5.3.4 Surface Water Sampling Equipment

The selection of sampling equipment depends on the site conditions and sample type to be acquired. The most frequently used samplers are:

- Open tube.
- Dip sampler.
- Weighted bottle.
- Hand pump.
- Kemmerer.
- Depth-Integrating Sampler.

The dip sampler and the weighted bottle sampler are used most often, and detailed discussions for these devices only (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. Nonreactive/noncontaminating properties - Teflon-coated, glass, stainless-steel or PVC sample chambers are preferred (in that order).

As specified above, each sample (grab or each aliquot collected for compositing) shall be measured for but not limited to:

- Specific conductance.
- Temperature.
- pH.
- Dissolved oxygen (optional).

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

Subject SURFACE WATER AND SEDIMENT SAMPLING	Number SA-1.2	Page 7 of 12
	Revision 5	Effective Date 09/03

notebook or on sample logsheets (see Attachment A). These analyses will provide information on water mixing/stratification and potential contamination.

#### Dip Sampling

Water is often sampled by filling a container either attached to a pole or held directly, from just beneath the surface of the water (a dip or grab sample). Constituents measured in grab samples are only indicative of conditions near the surface of the water and may not be a true representation of the total concentration that is distributed throughout the water column and in the cross section. Therefore, whenever possible, it is recommended to augment dip samples with samples that represent both dissolved and suspended constituents and both vertical and horizontal distributions.

#### Weighted Bottle Sampling

A grab sample can also be taken 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 a stopped glass or plastic bottle, a weight and/or holding device, and lines to open the stopper and lower or raise the bottle. The procedure for sampling with this device is:

- Gently lower the sampler to the desired depth so as not to remove the stopper prematurely (watch for bubbles).
- Pull out the stopper with a sharp jerk of the stopper line.
- Allow the bottle to fill completely, as evidenced by the absence of air bubbles.
- Raise the sampler and cap the bottle.
- Decontaminate the outside of the bottle. This bottle can be used as the sample container as long as the bottle is an approved container type.

#### Kemmerer

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 being 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.

### **5.3.5 Surface Water Sampling Techniques**

Most samples taken during site investigations are grab samples. Typically, surface water sampling involves immersing the sample container in the body of water; however, the following suggestions are made to help ensure that the samples obtained are representative of site conditions:

Subject <b>SURFACE WATER AND SEDIMENT SAMPLING</b>	Number <b>SA-1.2</b>	Page <b>8 of 12</b>
	Revision <b>5</b>	Effective Date <b>09/03</b>

- The most representative samples are obtained from mid-channel at a 0.6 foot stream depth in a well-mixed stream.
- Even though the containers used to obtain the samples are previously laboratory cleaned, it is suggested that the sample container be rinsed at least once with the water to be sampled before the sample is taken. This is not applicable when sample containers are provided "pre-preserved."
- For sampling moving water, it is suggested that the farthest downstream sample be obtained first, and that subsequent samples be taken as one works upstream. In general, work from zones suspected of low contamination to zones of high contamination.
- To sample a pond or other standing body of water, the surface area may be divided into grids. A series of samples taken from each grid node is combined into one sample, or several grid nodes are selected at random.
- Care should be taken to avoid excessive agitation of the water, as loss of volatile constituents could result.
- When obtaining samples in 40 mL septum vials for volatile organics analysis, it is important 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. The vial can be turned upside down to check for air bubbles.
- Do not sample at the surface, unless sampling specifically for a known constituent which is immiscible and on top of the water. Instead, the sample container should be inverted, lowered to the approximate depth, and held at about a 45-degree angle with the mouth of the bottle facing upstream. When sample containers are provided "pre-preserved," use a dedicated, clean, un-preserved bottle for sampling and transfer to an appropriately-preserved container.

#### **5.4        Onsite Water Quality Testing**

Onsite water quality testing shall be conducted as described in SOP SA-1.1.

#### **5.5        Sediment Sampling**

##### **5.5.1      General**

Sediment samples are usually collected at the same verticals at which water samples were collected. If only one sediment sample is to be collected, the sampling location shall be approximately at the center of the water body.

Generally, the coarser grained sediments are deposited near the headwaters of the reservoir. Bed sediments near the center of a water body will be composed of fine-grained materials which 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 while areas likely to show net erosion (i.e., high-velocity, turbulent areas) and suspension of fine solid materials, shall be avoided.

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

Subject SURFACE WATER AND SEDIMENT SAMPLING	Number SA-1.2	Page 9 of 12
	Revision 5	Effective Date 09/03

water column concentrations are below detection limits). Therefore, it is important to minimize the loss of low-density "fines" during any sampling process.

All relevant information pertaining to sediment sampling shall be documented as applicably described in SOP SA-6.3 and Attachment B.

### 5.5.2 Sampling Equipment and Techniques

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.

The following health and safety provisions apply when working on/over/near water.

Size of Work Team:

- 1) Never less than 2 persons [who are wearing USCG approved Personal Flotation Devices (PFDs)]
- 2) A minimum of 3 persons if any of the following conditions are anticipated or observed:
  - Depth is greater than 3 feet
  - Involves a waterway that is turbulent or swift
  - 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 would 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 change

The third person in the above condition must be equipped and prepared to render emergency support [e.g., lifeline, tethered PFD (life saver), skiff, means to contact external emergency response support, etc.]

The following samplers may be used to collect bottom materials:

- Scoop sampler.
- Dredge samplers.

Each type of sampler is discussed subsequently.

#### 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 it can be waded, the easiest and best way to collect a sediment sample is to use a scoop sampler. This reduces the potential for cross-contamination. This method is accomplished by reaching over or wading into the water body and, while facing upstream (into the current), scooping the sampler along the bottom in an upstream direction. It is very difficult not to disturb fine-grained materials of the sediment-water interface when using this method.

Subject SURFACE WATER AND SEDIMENT SAMPLING	Number SA-1.2	Page 10 of 12
	Revision 5	Effective Date 09/03

### Dredges

Dredges are generally used to sample sediments which cannot easily be obtained using coring devices (i.e., 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. There are three major types of dredges: Peterson, Eckman and Ponar dredges.

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.

### **6.0 REFERENCES**

American Public Health Association, 1980. Standard Methods for the Examination of Water and Wastewater, 15th Edition, APHA, Washington, D.C.

Feltz, H. R., 1980. Significance of Bottom Material Data in Evaluating Water Quality in Contaminants and Sediments. Ann Arbor, Michigan, Ann Arbor Science Publishers, Inc., V. 1, p. 271-287.

Kittrell, F. W., 1969. A Practical Guide to Water Quality Studies of Streams. U.S. Federal Water Pollution Control Administration, Washington, D.C., 135 p.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020.

U.S. EPA, 1980. Standard Operating Procedures and Quality Assurance Manual. Water Surveillance Branch, USEPA Surveillance and Analytical Division, Athens, Georgia.

U.S. Geological Survey, 1977. National Handbook of Recommended Methods for Water-Data Acquisition. Office of Water Data Coordination, USGS, Reston, Virginia.



Subject SURFACE WATER AND SEDIMENT SAMPLING	Number SA-1.2	Page 12 of 12
	Revision 5	Effective Date 09/03

**ATTACHMENT B  
SOIL & SEDIMENT SAMPLE LOG SHEET**



Tetra Tech NUS, Inc.

**SOIL & SEDIMENT SAMPLE LOG SHEET**

Page \_\_\_ of \_\_\_

Project Site Name: _____	Sample ID No.: _____
Project No.: _____	Sample Location: _____
<input type="checkbox"/> Surface Soil	Sampled By: _____
<input type="checkbox"/> Subsurface Soil	C.O.C. No.: _____
<input type="checkbox"/> Sediment	Type of Sample:
<input type="checkbox"/> Other: _____	<input type="checkbox"/> Low Concentration
<input type="checkbox"/> QA Sample Type: _____	<input type="checkbox"/> High Concentration

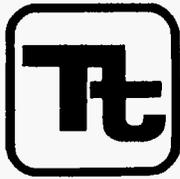
GRAB SAMPLE DATA:			
Date:	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time: _____			
Method: _____			
Monitor Reading (ppm): _____			

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

<b>OBSERVATIONS / NOTES:</b>	<b>MAP:</b>

<b>Circle if Applicable:</b>	<b>Signature(s):</b>
MS/MSD      Duplicate ID No.: _____	



TETRA TECH NUS, INC.

# STANDARD OPERATING PROCEDURES

Number SA-1.3	Page 1 of 20
Effective Date 09/03	Revision 7
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved D. Senovich <i>ds</i>	

Subject  
SOIL SAMPLING

## TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 PURPOSE.....	2
2.0 SCOPE.....	2
3.0 GLOSSARY .....	2
4.0 RESPONSIBILITIES.....	3
5.0 PROCEDURES.....	3
5.1 OVERVIEW.....	3
5.2 SOIL SAMPLE COLLECTION.....	4
5.2.1 Procedure for Collecting Soil Samples for Volatile Organic Compounds.....	4
5.2.2 Procedure for Collecting Non-Volatile Soil Samples .....	6
5.2.3 Procedure for Collecting Undisturbed Soil Samples (ASTM D1587-83).....	6
5.3 SURFACE SOIL SAMPLING .....	7
5.4 NEAR-SURFACE SOIL SAMPLING .....	7
5.5 SUBSURFACE SOIL SAMPLING WITH A HAND AUGER .....	8
5.6 SUBSURFACE SOIL SAMPLING WITH A SPLIT-BARREL SAMPLER (ASTM D1586-84) .....	9
5.7 SUBSURFACE SOL SAMPLING USING DIRECT PUSH TECHNOLOGY.....	10
5.8 EXCAVATION AND SAMPLING OF TEST PITS AND TRENCHES .....	10
5.8.1 Applicability.....	10
5.8.2 Test Pit and Trench Excavation .....	10
5.8.3 Sampling in Test Pits and Trenches .....	12
5.8.4 Backfilling of Trenches and Test Pits.....	15
5.9 RECORDS .....	15
6.0 REFERENCES.....	16
 <u>ATTACHMENTS</u>	
A SOIL & SEDIMENT SAMPLE LOG SHEET .....	17
B SPLIT-SPOON SAMPLER.....	18
C TEST PIT LOG .....	19
D REMOTE SAMPLE HOLDER FOR TEST PIT/TRENCH SAMPLING.....	20

Subject  SOIL SAMPLING	Number SA-1.3	Page 2 of 20
	Revision 7	Effective Date 09/03

## 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.

Subject  SOIL SAMPLING	Number SA-1.3	Page 3 of 20
	Revision 7	Effective Date 09/03

#### 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

Subject  SOIL SAMPLING	Number SA-1.3	Page 4 of 20
	Revision 7	Effective Date 09/03

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.

Subject  SOIL SAMPLING	Number SA-1.3	Page 5 of 20
	Revision 7	Effective Date 09/03

#### 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  $\pm$ 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.

Subject  SOIL SAMPLING	Number SA-1.3	Page 6 of 20
	Revision 7	Effective Date 09/03

### 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.

Subject  SOIL SAMPLING	Number SA-1.3	Page 7 of 20
	Revision 7	Effective Date 09/03

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.

Subject  SOIL SAMPLING	Number SA-1.3	Page 8 of 20
	Revision 7	Effective Date 09/03

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.

Subject  SOIL SAMPLING	Number SA-1.3	Page 9 of 20
	Revision 7	Effective Date 09/03

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 were encountered the sample material should still be collected from the core's

Subject  SOIL SAMPLING	Number SA-1.3	Page 10 of 20
	Revision 7	Effective Date 09/03

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).

Subject  SOIL SAMPLING	Number SA-1.3	Page 11 of 20
	Revision 7	Effective Date 09/03

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.

Subject  SOIL SAMPLING	Number SA-1.3	Page 12 of 20
	Revision 7	Effective Date 09/03

### 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.

Subject  SOIL SAMPLING	Number SA-1.3	Page 13 of 20
	Revision 7	Effective Date 09/03

- 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 In-Pit Sampling

Under rare conditions, personnel may be required to enter the test pit/trench. This is necessary only when soil conditions preclude obtaining suitable samples from the backhoe bucket (e.g., excessive mixing of 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:

Subject  SOIL SAMPLING	Number SA-1.3	Page 14 of 20
	Revision 7	Effective Date 09/03

- 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,

Subject  SOIL SAMPLING	Number SA-1.3	Page 15 of 20
	Revision 7	Effective Date 09/03

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.

Subject  SOIL SAMPLING	Number SA-1.3	Page 16 of 20
	Revision 7	Effective Date 09/03

- 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.

Subject  SOIL SAMPLING	Number SA-1.3	Page 17 of 20
	Revision 7	Effective Date 09/03

**ATTACHMENT A  
SOIL & SEDIMENT SAMPLE LOG SHEET**



Tetra Tech NUS, Inc.

**SOIL & SEDIMENT SAMPLE LOG SHEET**

Page \_\_\_ of \_\_\_

Project Site Name: _____		Sample ID No.: _____	
Project No.: _____		Sample Location: _____	
<input type="checkbox"/> Surface Soil	_____	Sampled By: _____	
<input type="checkbox"/> Subsurface Soil	_____	C.O.C. No.: _____	
<input type="checkbox"/> Sediment	_____	Type of Sample:	
<input type="checkbox"/> Other:	_____	<input type="checkbox"/> Low Concentration	
<input type="checkbox"/> QA Sample Type:	_____	<input type="checkbox"/> 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

<b>OBSERVATIONS/NOTES:</b>		<b>MAP:</b>
<b>Circle if Applicable:</b>		<b>Signature(s):</b>
<input type="checkbox"/> MS/MSD	<input type="checkbox"/> Duplicate ID No.: _____	

Subject

SOIL SAMPLING

Number

SA-1.3

Page

18 of 20

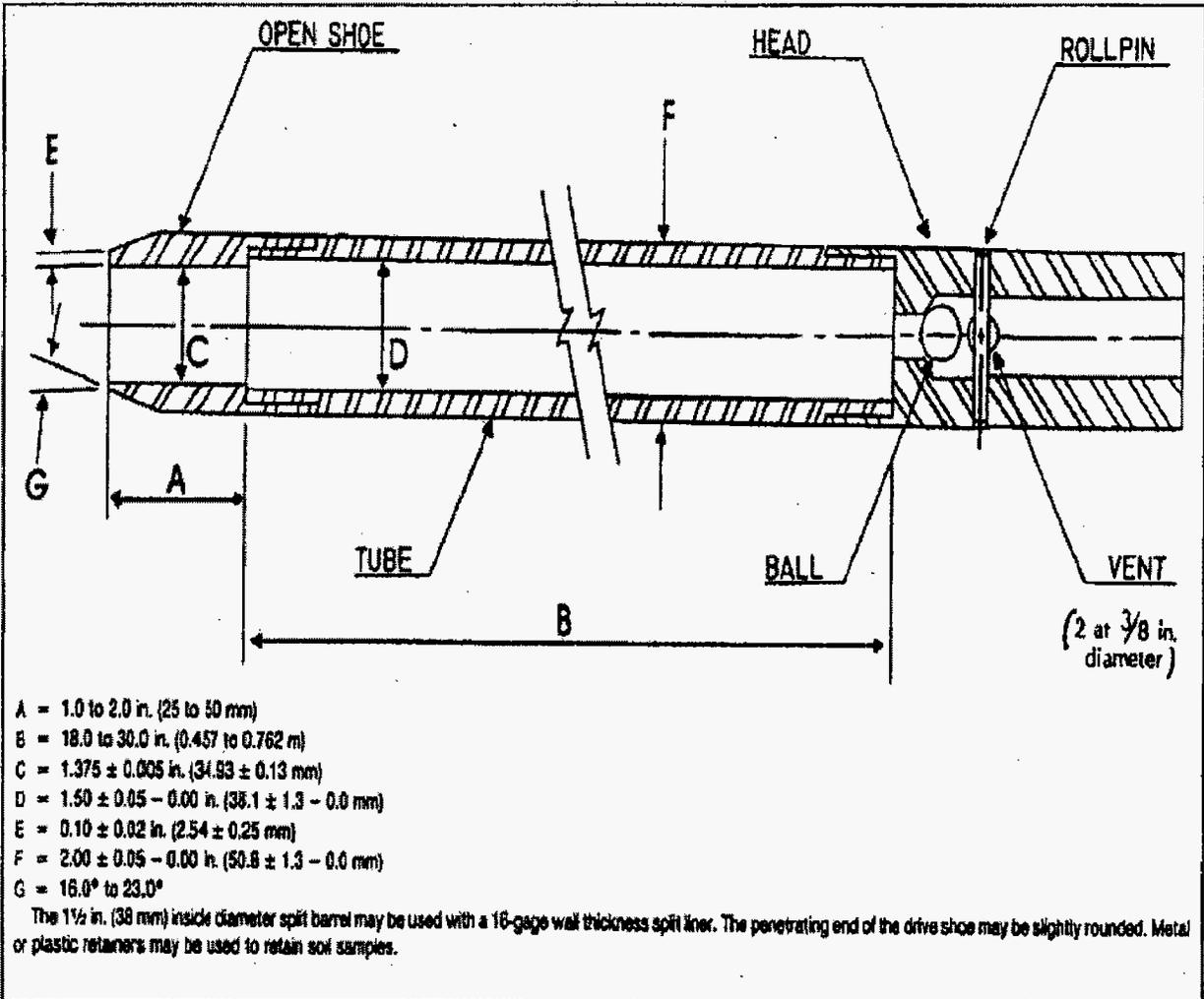
Revision

7

Effective Date

09/03

ATTACHMENT B  
SPLIT-SPOON SAMPLER





Subject

SOIL SAMPLING

Number

SA-1.3

Page

20 of 20

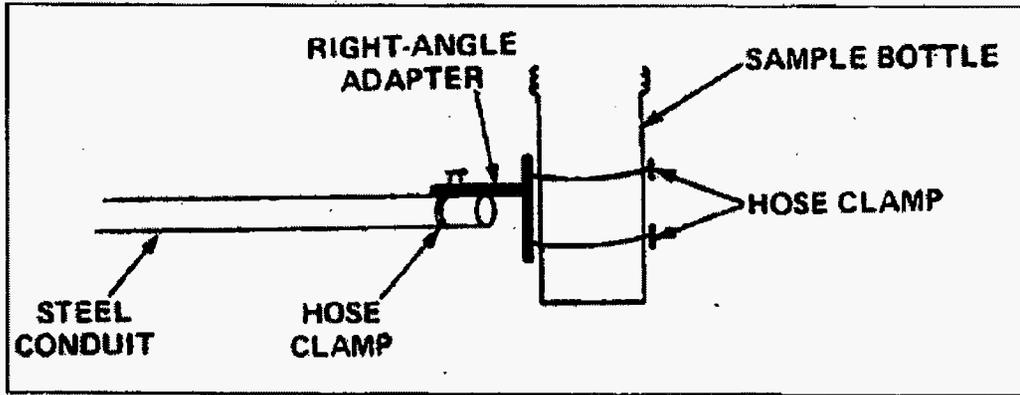
Revision

7

Effective Date

09/03

**ATTACHMENT D**  
**REMOTE SAMPLE HOLDER FOR TEST PIT/TRENCH SAMPLING**



**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

Prepared By: Mike Thomas Date: 7/96

Approved By: \_\_\_\_\_

Group Supervisor: Michael S. Thomas Date: 11/15/00

Operations Manager: J. Sinton Date: 10/23/00

QA Officer: Deborah J. Nadeau Date: 10-23-00

General Manager: Dennis F. Kufan Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10-23-00	10/23/00
02	removed references to medium level extraction. New logbook figures minor changes through out	LAD	020305 <del>DN</del> LAD 020305	020305
03	updated compound list changes in wording to clarify updated logbook	LAD	04/06	04/06

---

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

---

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

---

I acknowledge receipt of copy \_\_\_ of document **SOP CA-500-03**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_ of document **SOP CA-500-03**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

---

## **1.0 SCOPE AND APPLICATION**

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for pesticides/PCBs analysis.

### **1.1 Definitions**

### **1.2 Responsibilities**

This method is restricted to use by, or under the supervision of, analysts experienced in the extraction of sediment/soil samples for pesticides/PCBs analysis. Each analyst must demonstrate the ability to generate acceptable results with this method.

It is the responsibility of all Katahdin personnel involved in the preparation of solid samples for pesticides/PCBs analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for the data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials, and appropriate segregation of hazardous wastes. Everyone involved with the procedure must be familiar with the material safety data sheets for all the materials used in this procedure. Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures.

### **1.3 Safety**

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Management Plan and must follow

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

---

appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Management Plan and Safety Manual.

---

**2.0 SUMMARY OF METHOD**

Pesticides/PCBs are extracted from solid samples by sonication with a methylene chloride/acetone solution (1:1 by volume) following EPA Method 3550, current revision. The resulting extract is dried, concentrated, and solvent exchanged to hexane for analysis by GC.

This SOP applies to low level extraction of pesticide/PCB pollutants from solid sample matrices.

---

**3.0 INTERFERENCES**

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Whenever possible, plastic items in this lab, must be replaced with metal, teflon or other non-phthalate plastic substitute.

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

---

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be needed to minimize interferences.

---

#### **4.0 APPARATUS AND MATERIALS**

Prior to use, all glassware must be rinsed three times with methylene chloride.

- 4.1 Beakers - 400 mL
- 4.2 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL  
Evaporative flask - 500 mL  
Snyder column - 3-ball macro
- 4.3 Powder funnels, 100 mm diameter, 35 mm stem
- 4.4 Vacuum filtration flask - 500 mL Erlenmeyer
- 4.5 Buchner funnel, porcelain, Coors® with 85 mm plate diameter (or equivalent)
- 4.6 Sonic disruptor – Misonix XL2015 (or equivalent), equipped with dual titanium horn extenders for extracting two samples at a time.
- 4.7 Spatula - stainless steel
- 4.8 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)
- 4.9 Boiling chips - 12 mesh, silicon carbide (or equivalent)
- 4.10 Water bath - eight position concentric ring bath or equivalent, equipped with a calibrated thermometer
- 4.11 Filter paper - 7.0 cm, Whatman, #4, or equivalent
- 4.12 Syringe - gas tight, 1.0 mL, solvent rinsed between each use
- 4.13 Balance – top-loading, capable of weighing to 0.1 g

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

---

4.14 Nitrogen evaporation apparatus

---

## 5.0 REAGENTS

- 5.1 Sodium sulfate - (ACS reagent grade) powdered, anhydrous, certified by the manufacturer/vendor as purified by heating to 400 °C prior to receipt by the laboratory. Solvent rinse immediately before use by rinsing three times with pesticide grade methylene chloride.
- 5.2 Sodium sulfate - (ACS reagent grade) granular, anhydrous, purified as described in section 5.1.
- 5.3 Methylene chloride - (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300mLs to 1.0 mL followed by GC/MS analysis.
- 5.4 Acetone - pesticide grade or equivalent
- 5.5 Organic-free sand, purified by baking at 400 °C for four hours. Method blanks serve as checks on the baked sand.
- 5.6 Surrogate spiking solution - Prepare a solution of decachlorobiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL each in acetone. Store the solution at -10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.
- 5.7 Pesticide Matrix spike/Lab control sample spiking solution - Prepare a spiking solution in pesticide grade methanol that contains all target analytes listed below:

Analyte	ug/mL
4,4'-DDD	0.5
4,4'-DDE	0.5
4,4'-DDT	0.5
Aldrin	0.5
alpha-BHC	0.5
beta-BHC	0.5
delta-BHC	0.5
Dieldrin	0.5
Endosulfan I	0.5
Endosulfan II	0.5
Endosulfan Sulfate	0.5

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

---

Endrin	0.5
Endrin Aldehyde	0.5
Endrin Ketone	0.5
gamma-BHC (Lindane)	0.5
Heptachlor	0.5
Heptachlor Epoxide	0.5
Methoxychlor	0.5
alpha-Chlordane	0.5
gamma-Chlordane	0.5

Store the solution at  $-10$  to  $-20$  °C ( $\pm 2$  °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

- 5.8 PCB Matrix Spike/Lab Control Sample Spiking Solution – Prepare spiking solution in pesticide grade acetone that contains PCB's Arochlor 1016 and Arochlor 1260 (1660), both at 5.0 ug/mL.

Store the solution at  $-10$  to  $-20$  °C ( $\pm 2$  °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

---

## **6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING**

Soil samples are collected in glass soil jars and stored at 4°C ( $\pm 2$ °C) until time of extraction.

Holding time for extraction of sediment/soil samples for Method 3550 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C ( $\pm 2$ °C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

---

## **7.0 PROCEDURES**

LOW LEVEL EXTRACTION OF SOIL/SEDIMENT FOR PESTICIDES/PCBs

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

---

The low level extraction procedure is designed for the preparation of soil/sediment samples that may contain analytes at levels lower than 20,000 ug/kg. The procedure involves extraction of pesticides and PCBs from an initial sample weight of  $30.0 \pm 0.1$  g using an ultrasonic cell disruptor.

Many solid samples may need to be cleaned up to reduce matrix interferences. The cleanup procedure employed will be dependent upon the nature of the interferences and the target compounds to be analyzed, and options may include acid wash, sulfur cleanup, florisil cleanup, or gel permeation chromatography (GPC). The Department Manager should be consulted to determine if a particular sample should be subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs. All extracts or extract splits for subsequent 8082 PCB analysis will, at a minimum, undergo acid cleanup. (Refer to SOP CA-525, current revision)

- 7.1 Discard any excess water on the sediment sample. Mix with a stainless steel spatula to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, or rocks, and note actions taken in the appropriate extraction logbook.
- 7.2 Weigh out a  $30.0 \pm 0.1$  g portion of sample into a labeled 400 mL beaker. Record sample weight to nearest 0.1 g in appropriate extraction logbook. Add between 30 g and 60 g of powdered sodium sulfate, as required, to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.
- 7.3 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one  $30.0 \pm 0.1$  g portion of purified sand in a labeled 400 mL beaker. Add 60 g sodium sulfate and mix well. (Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)
- 7.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one  $30.0 \pm 0.1$  g portion of purified sand in a labeled 400 mL beaker. Add 30 g sodium sulfate and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB LCS's must be prepared (refer to sections 5.7 and 5.8). If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements.

---

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

---

This information will be disseminated from the project manager or Department Manager.

- 7.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out  $30.0 \pm 0.1$  g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.1 g in appropriate extraction logbook. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB MS/MSD pairs must be prepared (refer to sections 5.7 and 5.8).
- 7.6 To all samples, method blank, LCS, and MS/MSD add 1.0 mL pesticide/PCBs surrogate spiking solution using a 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to each use.
- 7.7 To LCS and MS/MSD pairs add 1.0 mL of pesticide or PCB matrix spike/LCS spiking solution using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to spiking a different solution and when spiking is completed.
- 7.8 To assure optimum operation and maximum energy output, the sonicators must be tuned daily prior to extracting samples. The following tuning procedure must be performed with the sonicator probes vibrating in air.
  - 7.8.1 Turn OUTPUT CONTROL knob counter-clockwise to zero, and turn Pulsar Duty Cycle to off (or continuous mode).
  - 7.8.2 Press POWER SWITCH to ON (up) position. Engage the Timer Switch (W-375)
  - 7.8.3 Press and hold down the TUNE switch.
  - 7.8.4 Turn the Output Control Knob towards setting 10. Note the position of the needle on the % output power meter. **Do NOT exceed 70%. If you reach 70% - STOP!!** Rotate the Tuning Knob clockwise or counter-clockwise until a minimum (not maximum) reading (usually less than 20%) is obtained.
  - 7.8.5 Turn the Output Control Knob towards setting 10. Again, note the position of the needle and do not exceed 70%. Rotate the Tuning Knob until you obtain a minimum reading of 20% or below.

---

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

---

- 7.8.6 Release the TUNE switch. **CAUTION: Do NOT touch probe. Probe is still active.**
- 7.8.7 Turn OUTPUT CONTROL KNOB counter-clockwise to zero (or disengage timer).
- 7.8.8 Confirm that the sonicators were tuned by recording the date and/or percent in the extractions logbook.
- Note:** If the unit will not be used immediately, please turn the power switch to off.
- 7.9 Prior to extracting any samples, ensure that the sonicator probes are decontaminated by rinsing with a methylene chloride wash bottle. Collect the waste in a waste beaker. It may sometimes be necessary to wipe the upper part of each probe with a methylene chloride dampened KimWipe. Repeat this decontamination step between each sample on each probe.
- 7.10 To the mixed and spiked blank and LCS, add 100 mL of the 1:1 methylene chloride/acetone solution and proceed with steps 7.11 through 7.14. After step 7.14, repeat these steps with two more mixed and spiked samples.
- 7.11 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula to loosen up the mixture prior to extracting. Position beaker in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4 inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.
- 7.12 Sonicate for 3 minutes with the output control knob set at 10, and mode switch on "pulsed" and % duty cycle knob set at 50%. While the mixture is sonicating, one should be able to see all, or most of the material, moving in the beaker under the influence of the energized probes. If not, stir the mixture again.
- 7.13 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter; prerinse flask, funnel and filter with methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask through Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered.
- 7.14 Repeat the extraction two additional times using 100 mL portions of 1:1 methylene chloride:acetone. Before each extraction, make certain that the sodium sulfate is free-flowing and not a consolidated mass. As required, break up large lumps with the clean spatula. Decant the extraction solvent into the Buchner funnel after each sonication. On the final sonication, pour the entire sample contents into the

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

---

Buchner funnel and rinse thoroughly with 1:1 methylene chloride:acetone to complete the quantitative transfer of the extract.

#### CONCENTRATION OF LOW LEVEL EXTRACTS

- 7.15 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels.
- 7.16 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow to drain.
- 7.17 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.
- 7.18 If samples are not to be GPC'd follow Steps 7.19 through 7.23 to concentrate extracts to final volume of 10.0 mLs. Otherwise proceed to GPC cleanup procedure as described in the current revision of Katahdin SOP CA-513.
- 7.19 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.
- 7.20 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 2 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

---

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

---

- 7.21 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with  $\approx$ 1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N<sub>2</sub> sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.
- 7.22 The extract is solvent exchanged into hexane using the N-evap apparatus and then concentrated to a final volume of 10 mL for analysis by GC/ECD. Add approximately 9 mL of hexane (10 mL total volume) to the concentrator tube. Concentrate the extract as described in Step 7.21, using hexane to rinse the internal wall of the concentrator tube and the N-evap sparging pipet. When the apparent volume reaches 1 -2 mL, remove the concentrator tube and allow it to cool..
- 7.23 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane extract to 10 mL in either a 12 or 16 mL vial using the appropriate "reference vial" for volume comparison.
- 7.24 Transfer the sample label from the K-D's to the vials. Store extract vials at a temperature of  $4 \pm 2$  °C until ready for analysis. Indicate in the extractions logbook the box number and "tray location" of the individual extract vials.
- 7.25 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. All sample extracts for 8081 pesticide analysis do not undergo further cleanup unless requested by the client. All sample extracts for combined 8081/8082 analyses must be split. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. Prior to splitting, contents of vial must be shaken well. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

---

## 8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

---

analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 A method blank must be extracted for each and every item listed below:

- Each day of extraction (24 hours midnight - midnight)
- Each extraction method
- Every 20 samples extracted in a 24-hour period

8.2 A laboratory control sample (LCS) is required for each and every item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticides and/or PCBs)

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

---

## **9.0 METHOD PERFORMANCE**

Refer to the applicable analytical SOP.

---

## **10.0 APPLICABLE DOCUMENTS/REFERENCES**

Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Method 3550B, USEPA SW-846, Third Edition, Final Update III, December 1996.

---

Table 1 Summary of Method Modifications

Figure 1 Example of Pest./PCB Soil Sample Prep Logbook Page

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-500-03	METHOD 3550, current revision
Apparatus/Materials	N-evap apparatus used for solvent exchange	Macro K-D used for solvent exchange
Reagents		
Sample preservation/handling		
Procedures	Solvent exchange to hexane is performed using N-evap apparatus with addition of approximately 5 - 8 mL hexane	Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane
QC - Spikes	Refer to analytical SOP	
QC - LCS		
QC Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT PESTICIDES/PCBs ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

P/P SON

KATAHDIN ANALYTICAL SERVICES, INC.  
 ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: SW846 3550 (sonication)  SW846 3540 (soxhlet)  SW846 3545 (ase)   
 Analytical Method: SW846 8081  SW846 8082  CLP OLM03.1  CLP OLM04.2   
 Date QC Started: 6-2-06 QC Expiration Date: 6-16-06 QC Batch ID \_\_\_\_\_  
 Surrogate ID: GCO333 Spike ID: A<sup>+</sup> GCO325 Spike ID: PCB GCO334  
 Solvent Lot # Meth: C03H16 Solvent Lot # Acet: B37E60 Sonicator Horns Tuned: <20%

Date Extracted	Est. Init.	Sample ID	Initial Vol.	Surr. Vol.	Spike Vol.	Fraction		Final Vol.	Date Conc.	Trey Location	Initials	Clean-Up			Comments
						Par	PCB					GPC	Fltr.	Add Wash	
6-2-06	KF	WS28719-1	30g	1.0mL	NR	✓	✓	10mL	6-4-06	PP676 E9	KF			✓	For 6-3008 For 6-3008L
		WS28720-1				✓				E9					
		WS28719-2			1.0mL	✓				E10					
		-3				✓				E11				✓	
		WS28720-2				✓				E12				✓	
		-3				✓									



P/P SON

Date Extracted	Est. Init.	Sample ID	Initial Vol.	Surr. Vol.	Spike Vol.	Fraction		Final Vol.	Date Conc.	Trey Location	Initials	Clean-Up			Comments
						Par	PCB					GPC	Fltr.	Add Wash	
6-2-06	KF	WW2454-1	30g	1.0mL	NR	✓	✓	10mL	6-4-06	PP676 E1	KF			✓	RE
		-2				✓				F2					
		-3				✓				F3					
		-4				✓				F4					
		-5				✓				F5					
		-6				✓				F6					
		-7				✓				F7					
		-8				✓				F8					
		-9				✓				F9					
		-10				✓				F10					
		-11				✓				F11					✓
		WW2638-1				✓	✓			F12					
		-2				✓	✓			PP677 A1					
		-3				✓	✓			A2					
		WW2645-26				✓				A3					
		-27				✓				A4					
		-28				✓				A5					
		-29				✓				A6					
		-30				✓				A7					

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

Prepared By: Mike Thomas Date: 7/98

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: J. Benton Date: 11/15/00

QA Officer: Deborah J. Nadeau Date: 11.15.00

General Manager: Dennis F. Kufan Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	11.15.00	11/15/00
02	Added definitions to section 1.1. Wording changed or added to clarify sections 5, 6, 8, + 9. New figure	MRC	11.08.04	11.08.04
03	Sect. 7.1.2 - adding the step to rinse forceps also. 7.10 adding condenser temperature and output voltage of variable transformer	LAD	04/06	04/06

---

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

---

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

---

I acknowledge receipt of copy \_\_\_ of document **SOP CA-524-03**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_ of document **SOP CA-524-03**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

---

## **1.0 SCOPE AND APPLICATION**

The purpose of this SOP is to describe the procedure for extracting pesticides/PCBs from solids such as soils, sludges, and wastes using Method 3540. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures including methods 8081 for pesticides and 8082 for PCB's.

### **1.1 Definitions**

**METHOD BLANK** (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

**LABORATORY CONTROL SAMPLE (LCS)**: A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

**MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)**: Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

**SURROGATES**: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

### **1.2 Responsibilities**

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for pesticide/PCB analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

---

method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training and Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for pesticide/PCB analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

### 1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

### 1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual.

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

---

**2.0 SUMMARY OF METHOD**

- 2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.
- 2.2 The extract is then dried, concentrated, and exchanged into hexane for GC analysis. Sulfuric acid cleanup is performed on extracts for 8082 PCB analysis.
- 

**3.0 INTERFERENCES**

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be needed to minimize interferences.

---

**4.0 APPARATUS AND MATERIALS**

- 4.1 a) Soxhlet extractor – 45/50 top joint and 24/40 lower joint.
- b) 500 mL flat-bottom boiling flask
- c) Allihn cooling water condenser

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

---

- 4.2 Powder Funnels – 100 mm top diameter, 35 mm stem
- 4.3 Kuderna-Danish (K-D) apparatus
  - 4.3.1 Concentrator tube - 10-mL
  - 4.3.2 Evaporation flask - 500-mL
  - 4.3.3 Snyder column - Three-ball macro
- 4.4 Nitrogen evaporation (N-EVAP) apparatus.
- 4.5 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride
- 4.6 Water bath - Heated, with concentric ring cover, capable of temperature control ( $\pm 5^{\circ}\text{C}$ ). The bath should be used in a hood.
- 4.7 Vials - Glass, 4, 12, or 16 mL with Teflon-lined screw caps
- 4.8 Glass wool (fiberglass) - baked at  $400^{\circ}\text{C}$  for a minimum of 4 hours or overnight.
- 4.9 Heating mantles - Rheostat controlled.
- 4.10 Disposable glass Pasteur pipets, 5  $\frac{3}{4}$ ", and bulbs.
- 4.11 Drying oven - capable of maintaining  $105^{\circ}\text{C}$  for glassware drying.
- 4.12 Muffle oven – capable of maintaining  $400^{\circ}\text{C}$  for baking glass wool and organic-free sand.
- 4.13 Beakers, 250 or 400 mL
- 4.14 Top-loading balance - capable of weighing to 0.1 g.
- 4.15 Spatulas, stainless-steel
- 4.16 Long forceps, stainless-steel
- 4.17 Metal clips – for securing Soxhlets to boiling flasks

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

---

**5.0 REAGENTS**

- 5.1 Sodium sulfate (granular, anhydrous and powdered, anhydrous) (ACS reagent grade), Na<sub>2</sub>SO<sub>4</sub>. Certified by the manufacturer/vendor as purified by heating at 400°C for 4 hours prior to receipt by the laboratory.
- 5.2 Methylene chloride - (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300 mL to 1 mL followed by GC/MS analysis.
- 5.3 Acetone – (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300 mL to 1 mL followed by GC/MS and GC analysis.
- 5.4 Organic-free sand, purified by baking at 400 °C at a minimum of 4 hours or overnight. Method blanks serve as checks on the baked sand.
- 5.5 Surrogate spiking solution - Prepare a solution of decachlorobiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL in acetone.
- 5.6 Matrix Spike/Lab Control Sample spiking solution
  - 5.6.1 Pesticide spike solution – prepare in pesticide grade methanol containing the analytes listed below at concentrations of 0.5 ug/mL.

4,4'-DDD
4,4'-DDE
4,4'-DDT
Aldrin
alpha-BHC
beta-BHC
delta-BHC
Dieldrin
Endosulfan I
Endosulfan II
Endosulfan Sulfate
Endrin
Endrin Aldehyde
Endrin Ketone
gamma-BHC (Lindane)
Heptachlor
Heptachlor Epoxide
Methoxychlor

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

---

- 5.6.2 PCB spike solution – prepare Aroclor 1660 (Aroclor 1016 and 1260) in pesticide grade acetone at a concentration of 5.0 ug/mL.
- 5.7 Store the solutions mentioned in sections 5.5 and 5.6 at 4°C ( $\pm 2^\circ\text{C}$ ) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use and must be replaced every 6 months or sooner if degradation is evident.
- 

**6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING**

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C ( $\pm 2^\circ\text{C}$ ).

Holding time for extraction of sediment/soil samples for Method 3540 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C ( $\pm 2^\circ\text{C}$ ) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

---

**7.0 PROCEDURES**

- 7.1 Preparing the Soxhlet Extraction Apparatus
- 7.1.1 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.
- 7.1.2 Add ~ 250 mLs of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Rinse the stainless steel forceps and pre-baked glass wool with Methylene chloride. Working in a hood, place a plug of the glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample.

---

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

---

7.2 Sample Handling

7.2.1 Sediment/soil samples - Decant and discard any water layer on a sediment sample. Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2 Gummy, fibrous, or oily materials not amenable to mixing should be cut, shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

7.3 Weigh out a  $30.0 \pm 0.1$  g portion of sample into a labeled 400 mL beaker. Record sample weight to nearest 0.1 g in appropriate extraction logbook. Add between 30 to 60 g of powdered sodium sulfate, as required, to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one  $30.0 \pm 0.1$  g portion of purified sand in a labeled 400 mL beaker. Add 60 g sodium sulfate and mix well. (Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one  $30.0 \pm 0.1$  g portion of purified sand in a labeled 400 mL beaker. Add 30 g sodium sulfate and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB LCS's must be prepared (refer to section 5.6). If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out  $30.0 \pm 0.1$  g portions of the sample

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

---

designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.1 g in appropriate extraction logbook. Add 30 g sodium sulfate to each to produce a free-flowing mixture, and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB MS/MSD pairs must be prepared (refer to section 5.6).

- 7.7 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be careful not to have any of the solid material fall into the extract flask through the large vapor tube.
- 7.8 To all samples, method blank, LCS, and MS/MSD add 1.0 mL of the pesticide/PCBs surrogate spiking solution using a 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in the extraction logbook. Thoroughly rinse syringe with solvent between each use.
- 7.9 To LCS and MS/MSD add 1.0 mL of either the pesticide or PCBs matrix spike/LCS spiking solutions using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification codes in the extraction logbook. Thoroughly rinse syringe with solvent between each use.
- 7.10 Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water condensers into the 45/50 joints of the extractors. After the joints have been rinsed with Methylene Chloride, condensers should be set to a temperature of 15°C. Save the pieces of aluminum foil for covering the Soxhlets when the extraction is complete. Switch on the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 55% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until the level reaches that of the small siphon tube and then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 18-24 hours. Be sure the chiller/recirculator temperature is set low enough to provide enough cooling capacity for the number of extractions in the batch.
- 7.11 When the extraction is complete, allow the extracts to cool before dismantling. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Remove the Allihn condenser and replace the aluminum foil on top of the extractor. Move the extractors to a hood and detach the extractor from the extract flask. Try to drain as much solvent as

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

---

possible from the extractor into the flask. Cover the flask with aluminum foil and store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

- 7.12 Immediately remove the extracted soil/sodium sulfate mixtures from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries.

#### CONCENTRATION OF THE EXTRACTS

- 7.13 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add a few boiling chips to the K-D. Insert fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels.
- 7.14 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow to drain.
- 7.15 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.
- 7.16 If samples are not to be GPC'd follow Steps 7.17 through 7.23 to concentrate extracts to final volume of 10.0 mLs. Otherwise proceed to GPC cleanup procedure as described in the current revision of Katahdin SOP CA-513.
- 7.17 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel, add one or two clean boiling stones to the K-D evaporative flask and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.
- 7.18 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

---

chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches  $\approx 2$  mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with  $\approx 1$  mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with  $\approx 1$  mL methylene chloride.

- 7.19 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with  $\approx 1$  mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N<sub>2</sub> sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.
- 7.20 The extract is solvent exchanged into hexane using the N-evap apparatus and then concentrated to a final volume of 10 mL for analysis by GC/ECD. Add approximately 5 - 8 mL of hexane to the concentrator tube and slightly raise the temperature of the water bath to  $\sim 45$  °C if necessary to facilitate concentration of the hexane. Concentrate the extract as described in Step 7.19, using hexane to rinse the internal wall of the concentrator tube and the N-evap sparging pipet. When the apparent volume reaches 1 -2 mL, remove the concentrator tube and allow it to cool. Be sure to lower the bath temperature to 39 °C after solvent exchange is completed.
- 7.21 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane extract to 10 mL in either a 12 or 16 mL vial using the appropriate "reference vial" for volume comparison.
- 7.22 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of  $4 \pm 2$  °C until ready for analysis. Indicate in the extractions logbook the box number and "tray location" of the individual extract vials.
- 7.23 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. All sample extracts for 8081 pesticide analysis do not undergo further cleanup unless requested by the client. All sample extracts for combined 8081/8082 analyses must be split. One portion must be acid cleaned for

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

---

8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

---

## **8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA**

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

Each extraction analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

---

Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

---

## **9.0 METHOD PERFORMANCE**

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

---

## **10.0 APPLICABLE DOCUMENTS/REFERENCES**

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3540C, SW-846, Third Edition, Updates I, II, IIA, IIB, and III Revised December 1996, US EPA.

---

### **LIST OF TABLES AND FIGURES**

Table 1	Summary of Method Modifications
Figure 1	Example of Logbook Page

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-524-03	METHOD 3540, current revision
Apparatus/Materials		
Reagents		
Sample preservation/handling		
Procedures	<ol style="list-style-type: none"> <li>1. Use 30 grams of sample and 30 grams of sodium sulfate.</li> <li>2. Use 250 mL of methylene chloride</li> <li>3. Hexane solvent exchange performed at N-EVAP stage.</li> </ol>	<ol style="list-style-type: none"> <li>1. Use 10 grams of sample and 10 grams of sodium sulfate.</li> <li>2. Use 300 mL of methylene chloride</li> <li>3. Hexane solvent exchange performed at Macro KD stage.</li> </ol>
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		



**TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD**

Prepared By: Greg Lull Date: 7/2002  
 Approved By: \_\_\_\_\_  
 Group Supervisor: Keith Tanguay Date: 09/11/02  
 Lab Operations Mgr: J. C. Benton Date: 9/11/02  
 QA Officer: Deborah J. Nadeau Date: 9.11.02

**Revision History:**

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Sect. 1-clarified CCB, Sect. 2-clarified phosphoric acid conc. Sect. 7- reworded software and instr. start up, added TS to calc.; removed steps not currently in practice. Sect. 8 -updated LCS and CV information. Updated Table 1	LAD	01/07	01/07

---

**TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD**

---

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

---

I acknowledge receipt of copy \_\_\_ of document SOP CA-741-01, titled **Determination of Total Organic Carbon in Solids Using the EPA Region II Lloyd Kahn Method.**

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_ of document SOP CA-741-01, titled **Determination of Total Organic Carbon in Solids Using the EPA Region II Lloyd Kahn Method.**

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

**TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA  
REGION II LLOYD KAHN METHOD**

---

## 1.0 SCOPE AND APPLICATION

This SOP describes the procedures used by Katahdin Analytical Services, Inc. technical personnel to determine Total Organic Carbon (TOC) in solids in accordance with EPA Region II Lloyd Kahn method.

This method is applicable to sediment, sludges, and soil samples. The detection limit for this method is 100 µg C and a method PQL of 400 µg/g.

### 1.1 Definitions/Acronyms

TC – Total carbon  
IC – Inorganic Carbon  
TOC – Total Organic Carbon

Method Blank - A deionized water sample that is carried through the entire analytical procedure in the same manner as a sample.

LCS/ICV - Laboratory Control Sample/ Initial Calibration Verification. One LCS/ICV per batch is prepared from a separate source from the CCV and calibration curve standards. LCS/ICV verifies the calibration curve.

CCV - Continuing Calibration Verification. The CCV is made from the same source as the calibration. One CCV is run every ten samples.

CCB - Continuing Calibration Blank. The CCB is an empty sample boat with no reagents added. One CCB is run every ten samples.

### 1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of TOC in solids by the Lloyd Kahn Method. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of TOC in solids by the Lloyd Kahn method to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

**TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA  
REGION II LLOYD KAHN METHOD**

---

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

### 1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method have not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their department manager, or designee, appropriate for the job functions they will perform.

### 1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in adherence with the Katahdin Hazardous Waste Management and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

---

## 2.0 SUMMARY OF METHOD

**Total Carbon (TC)** is measured utilizing a carbonaceous analyzer with a boat sampling module and 900°C furnace attached. The resulting combustion converts carbon to carbon dioxide (CO<sub>2</sub>) in the presence of oxygen. The amount of CO<sub>2</sub> derived from a sample is directly

**TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD**

---

proportional to the concentration of carbonaceous material in the sample and is then measured by a non-dispersive infrared detector (NDIR).

To determine **Total Organic Carbon** (TOC), however, carbonate and bicarbonate ions contributing to the TC result must be accounted for. This is achieved by adding 1:1 phosphoric acid to the sample and combusting it at 103° C for 10 minutes to remove any **Inorganic Carbon** (IC) before analyzing the sample. The **Total Carbon** result then equals the **Total Organic Carbon**.

---

**3.0 INTERFERENCES**

Volatile organics in the sediment may be lost in the decarbonation step resulting in a low bias.

---

**4.0 APPARATUS AND MATERIALS**

- 4.1 Shimadzu model TOC-Vcph with NDIR.
  - 4.2 SSM-5000A 970°C furnace with boat sampling module.
  - 4.3 Mettler AE 100 balance (accurate to 0.1 mg) or equivalent.
  - 4.4 Ceramic boats.
  - 4.5 Drying oven capable of maintaining 103-105°C
  - 4.6 Oxygen gas
- 

**5.0 REAGENTS**

- 5.1 Dextrose Solid (40% Carbon by weight = 400,000 ug/g C)

Calibration Standards:

- 0.0 mg - Calibration Blank
- 1.0 mg - 400ugC
- 5.0 mg - 2000ugC
- 10.0 mg - 4000ugC
- 20.0 mg - 8000ugC
- 60.0 mg - 24,000ugC

---

**TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD**

---

(Upper range limit for TC is 30,000ugC)

- 5.2 Dextrose Solid (40% Carbon by weight = 400,000 ug/g C) used as LCS run at 8000-12000ugC (20-30mg). Standard must be a source different from CCV source.
- 5.3 Sodium Carbonate, anhydrous (11.3% Carbon by weight = 113,000 ug/g C)

Calibration Standards:

0.0 mg	- Calibration Blank
3.5 mg	- 400ugC
17.7 mg	- 2000ugC
35.3 mg	- 4000ugC
70.7 mg	- 8000ugC
212.0 mg	-24,000ugC

(Upper range limit for IC is 25,000 ug C)

- 5.4 1:1 Phosphoric acid / DI water solution
- 5.5 Sodium Hydrogen Carbonate (14.28% Carbon by weight = 142,857ug/gC) used as LCS run at 4000ugC level (28 mg).

---

**6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING**

Collect sediments in a glass jar with Teflon or aluminum foil. Cool and maintain at 4° (±2) C. Analyze within 14 days.

---

**7.0 PROCEDURES**

**SET UP AND CALIBRATION**

- 7.1. Turn on TOC-Vcph analyzer, SSM-5000A furnace, and oxygen supply and let warm up for one hour or until the temperature readouts show 970° C for the TC and 200° C for the IC.
- 7.2. Start the TOC-V software program by clicking the TOC-V icon on the desktop and selecting sample table editor. Click the "new" icon followed by "sample run". Choose SSM-5000A from the pull down menu. This activates a sample field spreadsheet in which calibrations, controls, and samples can be inserted and run. Turn on the TOC aqueous and soil instruments and the oxygen. Click the "connect" icon and choose

---

**TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD**

---

“Settings on PC”. This will start the gas flows through both units and will activate the NDIR. Wait for the TC furnace to read 970°C before beginning analysis. Also place any sample boats that will be used in the furnace for several minutes to bake off any remaining residue.

- 7.3. Determine whether TC or IC will be analyzed for the run. A 6 point curve (for either TC or IC) must be run quarterly to verify the calibration. Using a calibrated analytical balance, the standards listed in section 5.1 for TC or in section 5.3 for IC should be used. The instrument must recover  $\pm 10\%$  of these true values. The calibration may also be updated as necessary as demonstrated by failure of the Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV). An ICV is run at the beginning of every sample sequence and a CCV is run every 10 samples. To verify baseline stability and demonstrate any cross contamination issues, a boat blank or Continuing Calibration Blank (CCB) is run at the beginning of a sample sequence and every ten samples as well. A laboratory control sample (LCS) and duplicate (LCSD) are run at the beginning of a sample sequence and every 20 samples as a secondary source calibration check.
- 7.4. The value of the calibration checks must fall within the control limits (80-120% recovery). If not, rerun the sample up to two more times. If the calibration check is still out of the acceptable recovery range, recalibrate the instrument and repeat the procedure. If the problem persists, remake the standard and repeat the procedure.

#### ANALYSIS

- 7.5. Using a calibrated analytical balance, weigh out 50-200mg of the sample in a tared sample boat. Record the weight in the TOC soil logbook (Figure 1).
- 7.6. If the sample is being analyzed for TC, the boat is inserted into the TC boat port. After placing the boat in the port and closing the cover, allow the instrument to reestablish the baseline (the atmospheric CO<sub>2</sub> that enters the closed system will create an initial small peak) by waiting 2 minutes or until the “baseline fluctuation” warning is back to “ready”.
- 7.7. Enter the sample information into the spreadsheet by clicking the insert sample icon. Select “tc method soils” as the method and click next. Type in the sample name, followed by next twice, then finish. Click the start icon and enter the file name for the sample run. The instrument will then prompt for the sample weight to be entered. The instrument will prompt to push boat into sampling position.
- 7.8. If the sample is being analyzed for IC, the boat is inserted into the IC boat port. After placing the boat in the port and closing the cover, allow the instrument to restabilize just as in the TC analysis. Once the “baseline fluctuation” warning is back to a ready

---

**TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD**

---

status, and before inserting the boat, apply between 0.2- 0.5 ml of the 1:2 phosphoric solution using the pump bottle attached to the IC port. Insert the boat immediately and allow the inorganic carbon to evolve and be evaluated.

- 7.9. To view the sample peak in real time, click view sample window in the menu bar at the top of the screen. This will show the NDIR activity as CO<sub>2</sub> evolves.
- 7.10. After integration, back the boat out of the furnace to the cooling position and then to the sample preparation position as prompted by the software. When sample run is complete, print sample table (Figure 2) and sample results (Figure 3).

#### CALCULATIONS AND REPORTING OF RESULTS

- 7.11. Calculate the TC or IC (whichever is being performed) concentration using the following equation:

$$\frac{\text{Abs C value (instrument reading) in ug}}{\text{Sample Weight (g)}} \times \frac{100}{\%TS} = \text{TC or IC result in ug/g C}$$

- 7.12. Workgroup samples and get run ID. Enter true values for the LCS and MS and save. Go back to the spreadsheet and enter "LLOYD" in the comments section for the samples you wish to report. Change QC to match workgroup. Data is then exported by selecting the ASCII export option from the file menu. Select "save as" and choose parsefiles on LVSlims. Select TOC and type file name. Click save and wait for data to export, then review data in wetrev.
- 7.13. A batch sheet is generated (Figure 4). Raw data, calibrations, and batch sheets are reviewed for completeness and accuracy by the Inorganic Department Manager or other qualified designee.
- 7.14. Printouts of instrument calibrations and sample data are filed in the lab for approximately 3 months for reference by analysts. Prior calibrations are archived and all are available for retrieval.

---

## 8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

See below and refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed below and in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The

---

**TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD**

---

corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

- 8.1 An ICB is analyzed at the beginning of the run and a CCB is analyzed every ten samples thereafter. The ICB and CCBs are boat blanks. The boat should be analyzed empty.
- 8.2 Analyze an LCS and LCSD (20-30 mg of Dextrose = 8000-12000ugC for TC) / (28 mg Sodium Hydrogen Carbonate = 4000 ug C for IC) with each batch of 20 samples. Acceptance criteria is 80-120% of expected value.
- 8.3 Run a CCV (20-30 mg Dextrose = 8000-12000 ug C for TC) / (35.3 mg Hydrogen Carbonate = 4000 ug C for IC) every 10 samples and at the end of each batch. Acceptance criteria is 80-120% of expected value. Run a CCB or boat blank every 10 samples. Results for this blank must not be greater than the reporting limit (PQL).
- 8.4 Run a duplicate every 20 samples. Run a matrix spike every 10 samples by weighing out the sample and adding 10 mg of dextrose to it for the TC spike or 35.3 mg of hydrogen carbonate for the IC spike. The recovery can be determined by calculating the theoretical yield from the sample result based on the weight as compared to the native result and adding 4000 ug C that was added from the spike component. The actual yield divided by the theoretical yield will give the recovery.

---

**9.0 METHOD PERFORMANCE**

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL's are determined annually per type of instrument filed with the Inorganic Department

**TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD**

---

Manager and with the QAO. The MDL standards are prepared following the procedures outlined in this SOP.

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL. Refer to the current revision of EPA 9060 and the Lloyd Kahn method for other method performance parameters and requirements.

---

**10.0 APPLICABLE DOCUMENTS/REFERENCES**

Determination of Total Organic Carbon in Sediment, Lloyd Kahn, USEPA Region II, 7/88.

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW846, third Edition, Final Update III, December 1996, Method 9060.

TOC-V series SSM-5000A user's manual.

Installation and Operation of Shimadzu's Solid Sample Module.

---

**LIST OF TABLES AND FIGURES**

Table 1	QC Requirements
Table 2	Summary of Method Modifications
Figure 1	Example of TOC Soil Logbook Page
Figure 2	Example of TOC Soil Instrument Spreadsheet Printout
Figure 3	Example of TOC Soil Instrument Results Printout
Figure 4	Example of TOC Soil Batch Sheet

**TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD**

TABLE 1

QC REQUIREMENTS – LLOYD KAHN

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS	One per prep batch	80-120% recovery	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
ICV/CCV	ICV at the beginning of the analysis and one after every 10 samples: same conc. as LCS/ICV	80-120% recovery	(1) Investigate source of problem. (2) If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reanalyze all samples back to last acceptable CCV recovery
Sample Quadruplicate	One every twenty samples.	≤ 30% RPD and < 3 times the standard deviation	(1) If lab QC in criteria and matrix interference suspected, flag data (2) Else, reanalyze
Matrix spike	One MS per ten samples	75-125% recovery	(1) If LCS in criteria and matrix interference suspected, flag data (2) Else, reanalyze
Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file
MDL study	Once per year	Ideally, PQL = 3 to 5 X the MDL.	Repeat MDL

**TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD**

TABLE 2  
 SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-741-01	METHOD LLOYD KAHN
Apparatus/Materials		
Reagents	Glucose solid for TC calibration (Dextrose for LCS) Sodium Carbonate for IC calibration (Sodium Hydrogen Carbonate for LCS)	Potassium Hydrogen Phthalate solution used for calibration.
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

**TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD**

FIGURE 1

EXAMPLE OF TOC SOLIDS LOGBOOK PAGE

Katahdin Analytical Services, Inc.  
Carbon Analysis of Solid Samples - Shimadzu TOC-V<sub>CPH</sub> / SSM-5000A

Analysis Type and Method (Check One)	
<input type="checkbox"/> Total Carbon (SW846 9060M)	<input type="checkbox"/> Total Inorganic Carbon (SW846 9060M)
<input type="checkbox"/> Total Organic Carbon (SW846 9060)	<input type="checkbox"/> Other (Specify):
<input checked="" type="checkbox"/> Total Organic Carbon (Lloyd Kahn)	

Spiking Information	Calibration Information
LCS Spike Source ID / Compound: <u>Acetone SW1628</u>	Calibration Date: <u>082206</u>
CCV Spike Source ID / Compound: <u>SW1449</u>	Calibration Analyst: <u>AMB</u>
MS Spike Source ID / Compound: <u>SW1628</u>	

KATAHDIN Sample Number	Sample Wt. (mg)	Sample Type * (Circle One)	Spike Added (mg)
Blank		Wet Dry	
1 LCS		Wet Dry	29.3 <sup>TV=1720</sup>
2 UWL4746-7	191.5	Wet Dry	
3 -7 Dup	190.7	Wet Dry	
-7 Dup	190.0	Wet Dry	
-7 Dup	190.6	Wet Dry	
-8	186.8	Wet Dry	
-8 MS	164.8	Wet Dry	9.9 <sup>TV=3263</sup>
-9	183.2	Wet Dry	
-10	190.8	Wet Dry	
UWL474-1	168.8	Wet Dry	
-2	176.1	Wet Dry	
CCV		Wet Dry	26.0 <sup>TV1040</sup>
CCB		Wet Dry	
		Wet Dry	

\* "Wet" = field-moist sample (as received). "Dry" = oven-dried sample.

Analyst: <u>AMB</u>	Analysis Date: <u>091606</u>
Reviewer:	Review Date:

**TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD**

FIGURE 2

EXAMPLE OF TOC SOLIDS INSTRUMENT PRINTOUT

	Analysis	Sample Name	Dilutio	Result	Comment	Status	Action	Date / Time
1	SSM-TC	Blank	1.000	SSM-TC:0.000 u		Completed		09/16/06 03:54:48
2	SSM-TC	LCS	1.000	SSM-TC:11408		Completed		09/16/06 04:05:21
3	SSM-TC	WW4746-7	1.000	SSM-TC:7.795 u		Completed		09/16/06 04:15:24
4	SSM-TC	WW4746-7D	1.000	SSM-TC:2.308 u		Completed		09/16/06 04:22:10
5	SSM-TC	WW4746-7D	1.000	SSM-TC:10.76 u		Completed		09/16/06 04:33:49
6	SSM-TC	WW4746-7D	1.000	SSM-TC:10.26 u		Completed		09/16/06 04:46:24
7	SSM-TC	WW4746-8	1.000	SSM-TC:0.8961		Completed		09/16/06 04:53:53
8	SSM-TC	WW4746-8M	1.000	SSM-TC:4050 u		Completed		09/16/06 05:06:32
9	SSM-TC	WW4746-9	1.000	SSM-TC:19.84 u		Completed		09/16/06 05:16:14
10	SSM-TC	WW4746-10	1.000	SSM-TC:71.33 u		Completed		09/16/06 05:27:15
11	SSM-TC	WW4774-1	1.000	SSM-TC:4.253 u		Completed		09/16/06 05:35:55
12	SSM-TC	WW4774-2	1.000	SSM-TC:21.86 u		Completed		09/16/06 05:43:31
13	SSM-TC	CCV	1.000	SSM-TC:9766 u		Completed		09/18/06 05:58:03
14	SSM-TC	CCB	1.000	SSM-TC:0.000 u		Completed		09/16/06 06:03:06
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								

**TITLE: DETERMINATION OF TOTAL ORGANIC CARBON IN SOLIDS USING THE EPA REGION II LLOYD KAHN METHOD**

FIGURE 3

EXAMPLE OF TOC SOIL INSTRUMENT RESULTS PRINTOUT

AKMB 01/24/07 01:53:34 PM 116y0091606132

Instr. Information

System SSM-5000A  
Detector Combustion  
Catalyst Regular Sensitivity  
Cell Length short

Sample

Sample Name: Blank  
Sample ID: < Untitled >  
Origin: tc method SOILS.met  
Chk. Result

Type	Anal.	Dil.	Density	Result
Unknown	SSM-TC	1.000	1.000mg/gL	SSM-TC:0.000 ug

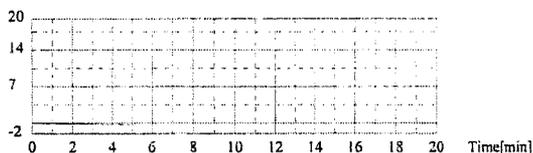
1. Det

Anal.: SSM-TC

No.	Area	CNV	Abs C	Conc.	Weight	Volume	Ex.	Cal. Curve	Date / Time
1	0.000	0.000	0.000ug	0.000ug	100.0mg	1.000L		tc:ca1082206a2006_08_22_15_38_36.cal	09/16/06 03:54:48 PM

Mean Area 0.000  
Mean CNV 0.000  
Mean Conc. 0.000ug

Signal[mV]



Sample

Sample Name: LCS  
Sample ID: < Untitled >  
Origin: tc method SOILS.met  
Chk. Result

Type	Anal.	Dil.	Density	Result
Unknown	SSM-TC	1.000	1.000mg/gL	SSM-TC:11408 ug

1. Det

Anal.: SSM-TC



TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

Prepared By: Mike Thomas Date: 09/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: JCBenton Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10.24.00

General Manager: Dennis P. Keegan Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10.24.00	10/24/00
02	Addition of compounds to Figure 2.	DN	3.28.02	3.28.02
03	Definitions added to section 1.1. Wording was added or changed to clarify sections 4, 5, 6, 7, 8 + 9. Minor changes throughout. New figures.	HRC	11.08.04	11.08.04
04	Updated Sect. 5.0 with current spike solutions prep. Removed section on medium level soil extraction. Replaced Figure 3 and 4 with current LCS/MS spike components. Minor corrections to sect. 1.3, 4.24, 6.0 and 7.12. Updated logbook	LAD	04/06	04/06

---

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

---

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

---

I acknowledge receipt of copy \_\_\_ of document **SOP CA-512-04**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_ of document **SOP CA-512-04**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

---

## **1.0 SCOPE AND APPLICATION**

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for analysis of extractable semivolatile organic compounds. This SOP is specifically applicable to EPA Method 3550.

This SOP describes the procedures for extraction of low concentrations of semivolatile organics and extraction of medium level concentrations of semivolatile organics from sediment/soil samples. The concentration ranges covered by these procedures may be considered to be approximately 330 ug/kg for semivolatile and 6.7 ug/kg for SIM-semivolatile for the low level analysis and >20,000 ug/kg for medium level analysis for semivolatile extractables. Samples are normally extracted following the low level extraction procedure. If GC/MS analysis or prior history indicates potential target compound concentration ranges encompassed by the medium level technique, then the medium level protocol is followed. Note that the terms "low level" and "medium level" are not used here as a judgement of degree of contamination but rather as a description of the concentration ranges that are encompassed by the "low" and "medium" level procedures.

### **1.1 Definitions**

**METHOD BLANK** (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

**LABORATORY CONTROL SAMPLE (LCS)**: A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

**MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)**: Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

**SURROGATES**: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks,

---

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

---

standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

### 1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

### 1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

---

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual.

---

**2.0 SUMMARY OF METHOD**

For the low level soil extraction, a 30 gram portion of sediment/soil is mixed with anhydrous powdered sodium sulfate and extracted with 1:1 methylene chloride/acetone (v/v) using an ultrasonic probe.

For the medium level soil extraction, a 1 gram portion of sediment/soil is transferred to a vial and extracted with methylene chloride using the Micro-tip probe.

---

**3.0 INTERFERENCES**

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory.

At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis. Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be needed to minimize interferences.

---

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

---

#### **4.0 APPARATUS AND MATERIALS**

Prior to use, all glassware must be rinsed three times with methylene chloride. Brand names and catalog numbers are included below for illustration purposes only.

- 4.1 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.
- 4.2 Sonicator ultrasonic processor XL – Misonix (or equivalent) equipped with dual titanium horn extenders for extracting two samples at a time.
- 4.3 Powder funnels, 100 mm diameter, 35 mm stem
- 4.4 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL  
Evaporative flask - 500 mL  
Snyder column - 3-ball macro
- 4.5 Powder funnels, 100 mm diameter, 35 mm stem
- 4.6 Vacuum filtration flask - 500 mL Erlenmeyer
- 4.7 Buchner funnel, porcelain, Coors® with 85 mm plate diameter (or equivalent)
- 4.8 Beakers - 400 mL
- 4.9 Boiling chips - approximately 12 mesh, silicon carbide (carborundum or equivalent). Soxhlet extract overnight in methylene chloride.
- 4.10 Water bath - eight position concentric ring bath, or equivalent, equipped with a calibrated thermometer. The bath should be used in a hood.
- 4.11 Balance - capable of accurately weighing  $\pm 0.1$  g.
- 4.12 Vials and caps – 1.8 mL with PTFE/silicone septa and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.
- 4.13 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)
- 4.14 Pasteur pipets - disposable, 5  $\frac{3}{4}$  “.
- 4.15 Nitrogen evaporation apparatus.

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

---

**5.0 REAGENTS**

- 5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, certified by the manufacturer/vendor as purified heating to 400°C prior to receipt by the laboratory. Solvent rinse immediately prior to use by rinsing three times with pesticide grade methylene chloride. (Jost Chemical anhydrous powder, catalog #2797 or equivalent, and Jost Chemical granular crystals, catalog #2796 or equivalent).
- 5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated by lot prior to use by concentration of approximately 400 mL to 1.0 mL followed by GC/MS analysis.
- 5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand.
- 5.4 Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations.

<b>Compound</b>	<b>Conc.</b>
phenol-d <sub>5</sub>	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-d <sub>5</sub>	50 ug/mL
terphenyl-d <sub>14</sub>	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method

- 5.5 Combined SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

<b>Compound</b>	<b>Conc. ug/mL</b>
Fluorene-d <sub>10</sub>	2.0 ug/mL
2-Methylnaphthalene-d <sub>10</sub>	2.0 ug/mL
Pyrene-d <sub>10</sub> .	2.0 ug/mL
Pentachlorophenol	2.0 ug/mL
Tribromophenol	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

---

solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

- 5.6 Base/Neutral and Acid (SVOA) Matrix Spike/Lab Control Sample Spiking Solution - Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 50 ug/mL for base/neutrals and 100 ug/mL for acids. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.
- 5.7 Base/Neutral and Acid (SVOA APPENDIX IX) Matrix Spike/Lab Control Sample Spiking Solution. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 100 µg/mL for each compound. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem
- 5.8 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2 ug/mL for base/neutral. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL of methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

---

**6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING**

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3540 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

---

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

---

## 7.0 PROCEDURES

### EXTRACTION OF LOW LEVEL SOILS

The low level extraction procedure is designed for the preparation of soil/sediment samples that may contain analytes at levels lower than 20,000 µg/kg for SVOA or SVOA APP IX or 6.7 µg/kg for SIM-SVOA. The procedure involves extraction of semivolatile organics from an initial sample weight of 30 g using an ultrasonic cell disruptor.

Some solid samples may need to be cleaned up to reduce matrix interferences. The cleanup procedure employed is gel permeation chromatography (GPC). The organic department manager should be consulted to determine if a particular sample should be subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs.

Sign chain-of-custody when removing and replacing samples in storage locations, and fill out the sample preparation/extraction log with the necessary information before starting the extraction. Prerinse all glassware three times with methylene chloride.

- 7.1 Decant and discard any water layer on a sediment sample. Mix with a stainless steel spatula to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, and rocks, and note actions taken in the appropriate extraction logbook.
- 7.2 The following steps should be performed rapidly to avoid loss of the more volatile extractable. Weigh out a  $30.0 \pm 0.1$  g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.1 g in appropriate extraction logbook. Add between 30 g and 60 g of anhydrous powdered sodium sulfate as required for producing a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.
- 7.3 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare a method blank, weigh out one  $30.0 \pm 0.1$  g portion of purified sand in a labeled 400 mL beaker. Add 60 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

---

- 7.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one  $30.0 \pm 0.1$  g portion of purified sand in a labeled 400 mL beaker. Add 30 g sodium sulfate and mix well. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.
- 7.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.1 g in appropriate extraction logbook. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well.
- 7.6 To all samples, method blank, LCS, and MS/MSD add 1.0 mL of the appropriate base/neutral and acid surrogate spiking solution listed below using the pre-rinsed 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution.
- 7.6.1 If the request is for SVOA, use the SVOA surrogate solution.
- 7.6.2 If the request is for SIM-SVOA, use the SIM-SVOA surrogate solution.
- 7.7 To the LCS and the MS/MSD add 1.0 mL of the appropriate base/neutral and acid (SVOA) matrix spike/LCS spiking solution listed below using a 1.0 mL gas tight syringe. Record the matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse the syringe with solvent when spiking is completed.
- 7.7.1 If the request is for SVOA, use the SVOA Spiking Solution.
- 7.7.2 If the request is for SIM-SVOA, use the SIM-SVOA Spiking solution.
- 7.7.3 If the request is for SVOA Appendix IX, use the SVOA Appendix IX Spiking solution.
- 7.8 To assure optimum operation and maximum energy output, the sonicators must be tuned daily prior to extracting samples. The following tuning procedure must be performed with the sonicator probes vibrating in air.
- 7.8.1 Turn OUTPUT CONTROL knob counter-clockwise to zero. This automatically switches the duty cycle to continuous mode.

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

---

- 7.8.2 Press and hold down the power switch to on.
- 7.8.3 Press and hold down the TUNE switch. Check if the counter is less or equal to 20%; otherwise, rotate the Tuning Knob (tuning button) clockwise until a reading of 20% or less is obtained.
- 7.8.4 Release the TUNE switch.
- 7.8.5 Turn OUTPUT CONTROL KNOB counter-clockwise to 50 and the power switch off.
- 7.8.6 Confirm that the sonicators were tuned by recording the date and/or percent in the extractions logbook.
- 7.9 Prior to extracting any samples, ensure that the sonicator probes are decontaminated by rinsing three times with a methylene chloride wash bottle. Collect the waste in a waste beaker. It may sometimes be necessary to wipe the upper part of each probe with a methylene chloride dampened KimWipe. Repeat this decontamination step between each sample on each probe.
- 7.10 To the mixed and spiked blank and LCS, add 100 mL of the 1:1 methylene chloride/acetone (V/V) solution and proceed with steps 7.11 through 7.14. After step 7.14, repeat these steps with two more mixed and spiked samples.
- 7.11 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula to loosen up the mixture prior to extracting. Rinse the spatula with methylene chloride and collect the rinsing into a correspondent beaker. Position the beaker in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4 inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.
- 7.12 Sonicate for 3 minutes with the output control knob set at 10, and mode switch on "pulsed" and % duty cycle knob set at 50%. While the mixture is sonicating, one should be able to see all, or most of the material, moving in the beaker under the influence of the energized probes. If not, stir the mixture again.
- 7.13 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter. Prerinse the flask, funnel and filter with methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask and Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered.

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

---

- 7.14 Repeat the extraction two more times (sec 7.11 – 7.14) using 100 mL portions of 1:1 methylene chloride: acetone. Before each extraction, make certain that the sodium sulfate is still free-flowing and not a consolidated mass. As required, break up large lumps with the spatula. Decant the extraction solvent into the Buchner funnel after each sonication. On the final sonication, pour the entire sample contents into the Buchner funnel and rinse thoroughly with methylene chloride to complete the quantitative transfer of the extract. Use the vacuum pump to pull all the extract into the flask

#### CONCENTRATION OF LOW LEVEL EXTRACTS

- 7.15 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels.
- 7.16 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mls of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mls of methylene chloride and allow to drain.
- 7.17 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.
- 7.18 If samples are not to be GPC'd, follow Steps 7.19 through 7.24 to concentrate extracts to final volume of 1 mL. Otherwise proceed to GPC cleanup procedure as described in the current revision of Katahdin SOP CA-513.
- 7.19 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel, add one or two clean boiling stones to the K-D evaporative flask and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.
- 7.20 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the

---

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

---

concentrator tube reaches  $\approx 6$  mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-extraction must occur immediately.** Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with  $\approx 1$  mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with  $\approx 1$  mL methylene chloride.

- 7.21 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with  $\approx 1$  mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N<sub>2</sub> sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.
- 7.22 When the apparent volume reaches  $\approx 0.5$  mL, remove the concentrator tube and allow it to cool.
- 7.23 Complete the quantitative transfer of the extract to a 1.8 mL vial by using methylene chloride. Adjust the volume of the methylene chloride extract to 1.0 mL using the 1.8 mL reference vial for volume comparison.
- 7.24 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of  $4 \pm 2$  °C until ready for analysis. Indicate in the extraction logbook the box number and "tray location" of the individual extract vials.

---

## 8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

---

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of Semivolatile Organics for quality control acceptance criteria.

Each extraction analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

---

## **9.0 METHOD PERFORMANCE**

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

---

Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

---

**10.0 APPLICABLE DOCUMENTS/REFERENCES**

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3550B, USEPA SW-846, Third Edition, Update III, December 1996

---

**LIST OF TABLES AND FIGURES**

Table 1	Summary of Method Modifications
Figure 1	Example of Logbook Page
Figure 2	LCS/Matrix Spike Component List
Figure 3	Appendix IX LCS/Matrix Spike Component List

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

TABLE 1  
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-512-04	METHOD 3550, current revision
Apparatus/Materials		
Reagents		
Sample preservation/handling		
Procedures		
QC - Spikes	Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC - MDL		

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

SV 500

Protocol: SW846 Analytical Method: SW846/8270  
 Analysis: Semivolatile Organics Extraction Technique: SONIC 3550  
 Date QC Started: 2-9-06 SOX 3540  
 QC Expiration Date: 2-23-06 ASE 3545  
 QC Batch ID: \_\_\_\_\_ Matrix: SOLID  
 SURROGATE ID: SV2093 SPMCK ID: SV2089

CLEAN-UP: GC screen, GPC, Florisil, Acid Wash, Other: Sonicator Horns Tuned: yes  
 Solvent Lot # Metric: B43E90  
 Solvent Lot # Acetone: B32K60

Sample ID	Container	Volume	Matrix	Date	Method	Notes
2906 TR	W05378-1	309	1ML NR	2-9-06	SW15 E8	very yellow, possible extraction
	-2		1ML		E9	
	-3				E10	
2906 TR	BLANK	309	1ML NR	2-10-06	SW16 B1	

Sample ID	Container	Volume	Matrix	Date	Method	Notes
2906 TR	W05461-1	309	1ML NR	2-9-06	SW108 D12	very dark - could not go below 2-23-06
	W05461-1			2-10-06	SW116 B2	
	W0533-4				B3	
	W05461-1				B4	
	-2				B5	
	-3				B6	
	-4				B7	
	-5				B8	
	-6				B9	
	-7				B10	
	-8				C1	
	-9				C2	
	-10				C3	
	-11				C4	
	W05461-1			2-10-06	SW16 B5	
	-2				C6	
	-3				C7	
	-4				C8	
	-5				C9	
	-6				C10	

QAEX117      000005      QAEX117      0000054

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

<b>BASE/NEUTRALS</b>	
1,1-Biphenyl	Bis (2-Chloroisopropyl) ether)
1,2,4-Trichlorobenzene	Bis (2-ethylhexyl) phthalate
1,2-Dichlorobenzene	Butylbenzyl phthalate
1,3-Dichlorobenzene	Caprolactam
1,4-Dichlorobenzene	Carbazole
2,4-Dinitrotoluene	Chrysene
2,6-Dinitrotoluene	Dibenz (a, h) anthracene
2-Chloronaphthalene	Dibenzofuran
2-Methylnaphthalene	Diethyl phthalate
2-Nitroaniline	Diethyladipate
3,3'-Dichlorobenzidine	Dimethyl phthalate
3-Nitroaniline	Di-n-butylphthalate
4-Bromophenylphenyl ether	Di-n-octyl phthalate
4-Chloroaniline	Fluoranthene
4-Chlorophenylphenyl ether	Fluorene
4-Nitroaniline	Hexachlorobenzene
Acenaphthene	Hexachlorobutadiene
Acenaphthylene	Hexachlorocyclopentadiene
Acetophenone	Hexachloroethane
Aniline	Indeno (1,2,3-cd) pyrene
Anthracene	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	o-toluidine
Benzo (ghi) perylene	Phenanthrene
Benzo (k) fluoranthene	p-toluidine
Benzyl alcohol	Pyrene
Bis (2-chloroethoxy) methane	Pyridine
Bis (2-chloroethyl) ether	

<b>ACIDS</b>		
2, 3, 4, 6-Tetrachlorophenol	2,4-Dinitrophenol	4-Methylphenol
2,3,4,6-Tetrachlorophenol	2,6-Dichlorophenol	4-Nitrophenol
2,4,5-Trichlorophenol	2-Chlorophenol	Benzoic acid
2,4,6-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4-Dichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dimethylphenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
	4-Chloro-3-methylphenol	Phenol

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachlorpropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthaquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylbenz(a)anthracene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitrobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Proamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

TITLE: **SAMPLE RECEIPT AND INTERNAL CONTROL**

Prepared By: Andrea Colby Date: 6/2002  
 Approved By: \_\_\_\_\_  
 Group Supervisor: Andrea Colby Date: 6/6/02  
 Lab Operations Mgr: J. C. Burton Date: 6/5/02  
 QA Officer: Deborah J. Nadeau Date: 6/6/02

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
04	Changed cover sheet, minor changes to sections 7.1, 7.6, 7.7.4, 7.10 + 7.20. Complete rewrite of sections 7.11 + 7.12 to comply with new KIMS	DN	6/6/02	6/6/02
05	Added verbal date entry to KIMS. Added reference to immediate internal COC book. Added Department Manager reference. Added section 7.7.3. updated new incoming	DN	05/04	05/04
06	Added procedure + logbook page for checking turbidity of drinking water samples. Changed wet chem shorts board to a book (included example page). Added custodial procedures for food/micro. Added VOA soil freezer storage.	DN	01-26-04	01-26-04
07	Added instructions to create lettered labels. Changed sample locations to reflect new building. Removed Figures 8 and 10. Updated Table and Figures w/ current ones. Added wording to Sect. 7.7.5 to clarify how pH measurements are taken.	LAD	02/07	02/07

---

TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

---

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

---

I acknowledge receipt of copy \_\_\_ of document **SD-902-07**, titled **Sample Receipt and Internal Control**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_ of document **SD-902-07**, titled **Sample Receipt and Internal Control**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

---

## 1.0 SCOPE AND APPLICATION

Katahdin Analytical Services, Inc. requires the use of specific receiving, acceptance, identification, storage, and distribution procedures for samples it accepts for analyses. These procedures assure that:

- samples are uniquely identified,
- samples are protected from loss or damage,
- essential sample characteristics are preserved,
- any alteration of samples (e.g., filtration, preservation) is documented,
- the correct samples are analyzed, and
- a record of continuous sample custody and utilization is established.

The purpose of this SOP is to describe the procedures used for the receipt and tracking of samples received by Katahdin Analytical Services, Inc. (Katahdin).

### 1.1 Definitions

SDG: Sample Delivery Group – A group of samples to be reported as one data package.

### 1.2 Responsibilities

It is the responsibility of all Katahdin staff who receive samples or handle samples in the course of analysis to follow the procedures set forth in this SOP, to document their understanding of the procedures in their training files (refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability") and to suggest changes and revisions when appropriate. All technical staff are responsible for monitoring their immediate areas, stopping an activity when a problem is detected or suspected, initiating corrective action when needed, documenting any actions taken, and notifying the appropriate individual (e.g., Department Manager, Operations Manager, QAO). The primary responsibility for implementing real-time corrective actions and maintaining an effective QA self-inspection system resides with Katahdin staff. When problems are identified, Katahdin personnel are expected to attempt to resolve situations within the scope of their technical knowledge, and to seek assistance from peers and the Department Manager as necessary.

It is the responsibility of Department Managers to oversee the adherence to Katahdin QC practices and internal documentation of laboratory activities within their area, to take corrective actions where needed and communicate problems to the Operations Manager, QAO or Vice President/President when warranted.

---

TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

---

It is the responsibility of the Operations Manager to oversee adherence to Katahdin QA/QC practices by all laboratory groups under his/her authority, to help identify problems and assure resolution, facilitate corrective action where needed, and to communicate problems and concerns to the QAO and Vice President/President.

It is the responsibility of the Quality Assurance Officer (QAO) to oversee adherence to this SOP, to conduct periodic audits of each laboratory, to track corrective action reports, resolution, and documentation, and to communicate concerns and report findings to the Vice President/President. The QA Officer shall function independently from laboratory operations and be able to evaluate data objectively and perform assessments without outside influence. The QA Officer has the authority to independently halt production operations (including data reporting) if warranted by quality problems.

### 1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Chemical Hygiene Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

### 1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the receipt of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed,

---

TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

---

placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

---

## **2.0    SUMMARY OF METHOD**

Not applicable.

---

## **3.0    INTERFERENCES**

Not applicable.

---

## **4.0    APPARATUS AND MATERIALS**

- 4.1    Radiation monitor (when required)
  - 4.2    Thermometer – Digital probe style capable of reading 0.1°C.
  - 4.3    Capillary tubes – 75 mm Hematocrit Tubes, disposable
  - 4.4    Wide range pH test strips, pH 0 to 14pH, EMD ColorpHast or equivalent.
  - 4.5    Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.
  - 4.6    Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.
- 

## **5.0    REAGENTS**

Preservatives - refer to Table 1, Sampling and Preservation Requirements, for specifics.

---

## **6.0    SAMPLE COLLECTION, PRESERVATION AND HANDLING**

Refer to Table 1, Sampling and Preservation Requirements, for specifics.

---

## **7.0    PROCEDURES**

REQUIREMENTS FOR USE OF RADIATION MONITOR

---

TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

---

- 7.1     Samples originating from USACOE sites and previously determined radioactive sites need to be screened for radioactivity upon receipt at the lab as a requirement of the program governing the site.
- 7.2     One radiation scanning device is available for screening samples for radiation. The meter is calibrated with a standard of known radioactivity on an annual frequency. A back-up meter is stored by the QAO.
- 7.3     A "hot" sample is defined as any sample that causes the radiation scanning device to read a level above the control limit for background radiation. The control limit for background radiation in the sample receiving area should be statistically determined annually by taking a series of measurements with both meters. Because some subjectivity is involved in reading the meters, at least two individuals should participate in the study.
  - 7.3.1    Measure and record a minimum of seven background readings.
  - 7.3.2    Calculate the mean and standard deviation of the background readings.
  - 7.3.3    Calculate the control limit for background radiation as  $X + 3s$ , where  $X$  is the mean and  $s$  is the standard deviation of the background readings.
  - 7.3.4    Maintain documentation that includes the calculated background control limit and the date the background radiation study was performed.

#### PROCEDURES FOR SAMPLE CUSTODIAN

- 7.4     When samples (except for non-environmental food samples) are dropped off, by either a delivery service (i.e. FEDEX or UPS) or by the client, the Chain-of-Custody (COC) should be signed immediately. The client or delivery service shall sign that they have relinquished custody to the laboratory. The laboratory shall sign and record the date and time that custody is accepted. (Refer to Figure 1 for Katahdin COC)
- 7.5     Enter the date and time of sample receipt and the client name into the next available work order/login number in the sample receipt logbook (Figure 2). Initial each entry (line) to maintain a record of the individual who assigned each group of samples a discreet lab work order/login number.
- 7.6     Inspect the condition of custody seals, cooler, ice condition and samples received. Note any non-intact conditions on the Sample Receipt Condition Report (SRCR - Figure 3). The project manager is notified of any discrepancies or problems with sample receipt. The PM contacts the client as necessary. If breakage of a potentially hazardous sample is discovered, the packing container with all the samples inside is

---

TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

---

closed, sealed, and moved to a hood in the organic extractions area or to the smaller hood in the login area if space permits. One of the three Katahdin Emergency Response Coordinators is notified. Disposition of the broken and other possibly contaminated samples will be determined on a case-by-case basis in accordance with the laboratory's handling procedures for hazardous waste as outlined in the Katahdin Chemical Hygiene Plan and Safety Manual and the Katahdin Hazardous Waste Management Program. Generally, when a sample has broken and has mixed with any ice in the cooler, that liquid will be poured off into 2 liter plastic containers and labeled as "do not use". These containers will be disposed of as soon as the disposition of the appropriate samples has been determined through analysis.

7.7    If there is no breakage of a potentially hazardous sample:

7.7.1   If applicable, check samples for radiation emission by passing a calibrated radiation monitor over the samples; the meter should be moved slowly and deliberately across the sample containers, maintaining a distance of about 3-5 inches from the sample containers. Contact the safety officer or the Operations Manager immediately for further instructions if the meter displays a reading above background level as defined in Procedure Section 7.3 above. Otherwise, proceed with Section 7.7.2.

7.7.2   Check cooler temperatures using the thermometer assigned to the sample receipt area. If a cooler temperature blank is present, insert the thermometer in the temperature blank; otherwise place the thermometer in the cooler as close to a sample as possible. Once the thermometer reading has stabilized, record the temperature on the Sample Receipt Condition Report. Cooler temperatures should be  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ . Any temperature falling outside of this range must be noted on the SRCR and reported to the appropriate Katahdin project manager.

7.7.3   Note the condition of the ice or ice packs. If the ice has melted and the temperature is out of acceptance criteria, note this on the SRCR. For samples that are hand delivered to the laboratory immediately after collection, the temperature blank and/or cooler temperature will most likely not meet the acceptance criteria. The samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice. Note this on the SRCR. If samples (that were just collected) have not arrived on ice, note this on the SRCR, and start the cooling process as soon as possible after arrival at the laboratory.

7.7.4   Inventory the samples against the chain of custody (COC). If the COC is incomplete, the sample custodian must inform the appropriate Katahdin project manager (PM). The PM may make changes to correct or complete

---

TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

---

the COC, but all changes must be initialed and dated. Changes must be noted on the SRCR. Any discrepancies between the samples and the COC must also be noted on the SRCR.

- 7.7.5 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, check if samples are in proper containers and received correct pretreatment (e.g., filtration, preservation) for the analyses requested. For aqueous parameters requiring preservation, check pH by inserting a clean capillary tube into the sample and dabbing the tube on wide range pH paper. If the pH is not clearly either less than 2 or greater than 12, the appropriate narrow range pH paper must be used. NOTE: The pH of volatile organic (VOA) samples is checked and recorded by the analyst after completion of analysis and not by sample receipt personnel. The used capillary tube is discarded and a new capillary tube is used for each sample. For metals and wet chemistry parameters, additional preservative is added if the pH is not in the range specified in the Sampling and Preservation Requirements Table. No more than 10% of the original sample volume should be added as preservative. If the client has noted that the sample reacts violently (i.e., foams and bubbles) upon preservation, add no more preservative to the sample. Some clients may wish to be contacted if their samples are found to be improperly preserved. Record all preservation discrepancies on the Sample Receipt Condition Report including the lot number of the preservative added. If additional preservative is added, a sticker with the type of preservative, date and time added, final pH and custodian's initials must be placed on the sample container.

Note: Preservatives are obtained from the larger containers in the bottle preparation room. Fill smaller 100 mL or 250 mL containers and record the lot number on the bottle. These containers should be kept in the hood in the sample receipt area.

- 7.7.6 For samples requiring filtration as pretreatment (i.e. for dissolved metals), the work order/login numbers are recorded in the filtration logbook (see Figure 4). The samples are filtered by the Metals Group.

7.7.6.1 A 500 mL filter flask and filter funnel are acid rinsed three times in a 10% nitric acid bath, then three times with Laboratory Reagent Grade Water.

7.7.6.2 A vacuum pump is attached.

7.7.6.3 A 0.45 micron filter is rinsed three times with 5% nitric acid and three times with Laboratory Reagent Grade Water. The rinsate is discarded.

---

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

---

- 7.7.6.4 A sufficient sample aliquot is filtered and preserved with concentrated nitric acid.
- 7.7.6.5 The bottles are labeled with the work order/login number and other sample information and stored at 4° C until the time of digestion.
- 7.7.7 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, determine if sufficient volume of sample is present for analysis. Note discrepancies on the SRCR.
- 7.7.8 For drinking water samples, enter the appropriate information (work order, date, etc.) into the Measured Turbidity and Preservation of Incoming Samples Logbook. Inform the appropriate analyst of the sample. The turbidity must be measured prior to sample preparation. If the turbidity is <1 NTU, the sample does not have to be digested prior to metals analysis. If the turbidity is >1 NTU, the sample must be digested prior to metals analysis. The sample must be preserved after the turbidity measurement is taken. Record the appropriate information in the logbook (Figure 5).
- 7.7.9 Notify the PM immediately if there are any discrepancies or problems with sample receipt. The PM will contact the client for information and resolution as necessary.
- 7.8 Review any paperwork that accompanies the sample(s) submitted for analysis along with laboratory-generated information. This includes shipping forms, letters, chain-of-custody forms, sample labels, Incoming Sample Information Sheets (ISIS), quotes, memos, etc. (examples included as Figures 6 - 8).
- 7.9 Resolve any questions or concerns raised by steps 7.1-7.8 by consulting the correspondence files or client services personnel or communicating directly with the client. Note in the notes section of the SRCR any deviations from normal sample handling or analytical procedures (e.g., client requests analysis although hold-time expired).
- 7.10 Notify appropriate section managers of any "RUSH" analyses or quick hold times. List any samples for analyses that have short hold times in the "Wet Chemistry Shorts and Rushes Logbook" (Figure 9) in the wet chemistry laboratory. Be sure to list the client, number of samples and date and time of the earliest sample. GC or GC/MS personnel must be informed when ENCORES are received so that they may be scheduled for extrusion. Microbiology personnel should also be informed of any microbiology samples that arrive. Parameters that routinely require short analytical hold times are:

---

TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

---

Coliforms	Color	pH
Nitrate/Nitrite	Dissolved Oxygen	Turbidity
Ferrous iron	Orthophosphate	Hex. Chromium
MBAS	BOD	Free CO <sub>2</sub>
Sulfite	ENCORE soil samples	

7.11 When non-environmental food samples are delivered to the laboratory, they are taken immediately to the food/microbiology laboratory and stored in the refrigerators there. A copy of the Chain-of-Custody is left with the analysts. The original paperwork is forwarded to sample log in where the job is logged into the KIMS system.

7.12 The following information is documented via the Katahdin Information Management System (KIMS) and a work order/login COC report (Figure 10) is generated for the samples received:

7.12.1 Log onto KIMS by entering employee ID under "Username", employee specific password under "Password" and KIMS under "Database".

7.12.2 Once logged onto KIMS select "Sample Management" and then "Login".

7.12.3 Select "New" and the next available Login ID number will automatically be entered. Select "OK" and the Sample Definition screen will open.

Note: If a Work Order number has already been opened, select "change" and type in the appropriate number to access the information.

7.12.4 In the Sample Definition Screen, enter the following information.

Client ID -           Enter client sample description.

ReceiveDate -       Enter in date samples were received in the lab in the format YY-Month-DD.

CollectDate -       Enter in date samples were collected in the format YY-Month-DDTIME.

TAT -                Enter TAT for hardcopy report.

DueDate -           Due date will automatically be calculated based on calendar days.

VerbalDate -        Manually type in verbal due date.

---

TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

---

- QuoteRef -           Enter quote number if applicable.
- Project -            Enter project number if applicable.
- Account -            Enter client specific account number.
- Account Name -      Account name will automatically be entered.
- Collected By -     Enter name/initials of sampler listed on COC. If unknown, enter "Client".
- Locator -            May be used for client ID information when requested by the project manager.
- Site -                Enter project site name.
- Description -        May be used for long client Ids when requested by the project manager.
- Discount -           No entry-not currently used.
- Priority -            No entry-not currently used.
- Fact. -               No entry-not currently used.
- Expected -           No entry-not currently used.
- Comments -          Enter MS/MSD, verbal due date and any sample irregularities if applicable.
- OrderDate -         Current date is automatically entered.
- Matrix -             Enter sample matrix code where
- |                          |                  |
|--------------------------|------------------|
| AQ = Aqueous             | SLD = Food Solid |
| SL = Solid, Soil, Sludge | AR = Air         |
| FP = Free Product        | SWAB = Swab      |
| WP = Wipe                | SAL = Saline     |
| NOAQ = NonAqueous        | TIS = Tissue     |
| DW = Drinking Water      |                  |
- Product Code -      Enter analysis code per test requested on COC.

---

TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

---

Type -	Product code type will automatically be entered where S = Stand alone P = Parent C = Children
Fact. -	No entry-default is 1.
Price -	This is left as is by sample log-in. During project management review of the work order, the prices are entered based on quotes or standard prices.
Cost -	No entry needed.
Lev -	No entry needed.
Type -	Container type will automatically be entered.
Bot -	Enter number of containers per test for printing of labels.
Login Info -	Parameter Data Screen will open. Enter following information
	KAS Proj. Manager- Initials of Katahdin person overseeing the project.
	Client PO#- Client purchase order.
	Project- Project name.
	Cooler Temperature- Temperature blanks or cooler temps.
	Delivery Services- Method of delivery to the lab.
	QC Level- QC Level of report and regulatory agency (ie., IV NFESC).
	SDG ID- Sample Delivery Group ID if applicable.
	SDG Status- Begin, Continue or End.
	Analysis Instructions- PM will enter special instructions regarding project.
	Report Instructions- PM will enter special instructions regarding project.
	Regulatory List- Used for federal programs.
	EDD Format- Specific KAS EDD format.

---

TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

---

Select "SAVE" and then "CANCEL".

Addresses -           Select "Addresses" and the Address Links screen will open. The billing address is the default address of the account. Enter the client account code under "Project/Account" and select the report to contact under "Address Type". Select the appropriate boxes for report, report CC and invoice CC. Select "SAVE" and then "CLOSE".

Create Containers -   Select "Create Containers". Letters will be assigned to each sample number. Select "OK" until letters have been assigned to each sample number. To manually assign letters, Select "Enter Container IDs" and "OK". Enter sample numbers including letters and select "OK", "Close", "Yes" to save changes, "Cancel" and "Cancel".

- 7.12.5 To print the login report, select "Reports", "Login" and "Login COC". Enter login number under "Login Number". Select "OK", "Run Report" and then "Print".
- 7.13 To print labels unique to each bottle, select "Reports", "Login" and "Labels". Enter login number under "Login/Prelogin", select "Background (IDX)" and select F9 on keyboard under "Select Sample Label". Select "OK" and then "Print". After labels print out select "Cancel".
- 7.14 Affix permanent sample number labels to sample containers, assuring that sample IDs on labels correspond to sample bottle IDs. Mark any known hazardous samples with appropriate warning labels. Do not obscure client ID on the bottles.
- 7.15 Place samples in their designated storage locations and log them in, noting initials, date and time, work order/login and sample numbers, and storage location on the internal laboratory chain of custody form (Figure 11). Place form in the appropriate binder in the log in area. Non-environmental food samples do not get an internal COC and are taken immediately to the food/microbiology lab for storage.

Storage location of the samples is determined by type of sample and/or type of analysis, as outlined below. Most samples are stored in the walkin cooler, which is organized by test type and work order/login number.

---

TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

---

Specific storage locations are described below. NOTE: Samples originating in the State of North Carolina must be stored in locations maintaining a storage temperature of 1 - 4.4 °C.

1. Aqueous samples for wet chemistry (except hardness, see Step 4 below) - left aisle as you enter walk-in cooler. TOC vials are to be stored in the trays designated for TOC samples (orange row).
2. Aqueous samples for organic extractions – right aisle as you enter walk-in cooler.
3. Non-aqueous samples (all analyses except volatile organics) - to the right and towards the back as you enter walk-in cooler. Non-aqueous samples for volatile organics are stored in the “white” refrigerator located Volatiles Laboratory.

Aqueous samples for metals and/or hardness analyses – right and left aisles towards the front as you enter walk-in cooler.

4. Samples (aqueous and solid) for volatile organics analyses (VOA)– All aqueous samples and soil samples in VOA vials (preserved with methanol or sodium bisulfate) are stored in the “blue” refrigerator in the Volatiles Laboratory. VOA soils in jars or ENCORE samplers are stored in the “white” refrigerator in the Volatiles Laboratory. VOA samples known or suspected to be hazardous (such that cross-contamination of other samples might occur) are placed in a “paint can” and stored in the walk-in.
5. Soil samples for volatile organics analyses (VOA) that are unpreserved or preserved with Laboratory Reagent Grade Water are stored in the freezer across from the wet chemistry laboratory.

7.16 Sample Receipt gives the Work order/login COC report and confirmation of the job, as logged-in, to the appropriate Katahdin project manager. All chain-of-custody and other receipt documentation must accompany the job. The project manager reviews the job for accuracy and completeness. Any unresolved issues should be resolved at this time. The project manager then dispatches the work order/login to the individual department worklists. The dispatched work order/login package is then filed in Data Management where the complete package will eventually be compiled.

7.17 Project or Program-specific log-in forms are completed as needed and submitted to the Project Manager with the original Work order/login package. These forms may include any or all of the following (Figures 3 and 8).

---

TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

---

Sample Receipt Condition Report  
non-EPA CLP DC-1 Form

- 7.18 Complete the sample log-in notebook for "Time Logged In".
- 7.19 The temperature of all sample storage refrigerators and freezers is recorded daily by assigned individuals. Notebooks containing a record of each refrigerator and freezer temperature history are used for this purpose and are maintained by the assigned individuals. Temperatures above or below the acceptance range are to be brought to the attention of a Department Manager, Operations Manager, or Quality Assurance Officer. Such an occurrence and the actions taken to correct it must be noted in the comments column of the temperature recording notebook next to the temperature measurement. (See Figure 12).

PROCEDURES FOR CHEMISTS

- 7.20 When removing a sample from its storage location, record on the laboratory internal chain-of-custody (from the appropriate department) the sample number, date and time it was removed, chemist who removed it, and the analysis to be conducted or reason for removal.
- 7.21 If the samples have not been logged in yet and they need to be pulled in order to analyze short holding time parameters, the analyst taking the sample must use the designated logbook (Immediate Internal COC – Figure 13) to sign the samples out. Many circumstances lead to analysts having to pull samples before they are logged into the KIMS system. It is everyone's responsibility to ensure that all samples can be accounted for at all times. Failure to do so can create confusion and bottle necks for others trying to access the samples.
- 7.22 If sample is not consumed by an analysis, return the remaining sample to its assigned storage location and enter the date and time returned on the laboratory internal chain-of-custody record.
- 7.23 If analysis consumes the entire sample, indicate this on the laboratory internal chain-of-custody record.

---

**8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA**

Each thermometer used to monitor sample storage or cooler temperatures must be calibrated annually against a NIST traceable thermometer. The QAO is responsible for ensuring that the thermometer(s) are scheduled for yearly calibration and for maintaining the calibration

---

TITLE:           SAMPLE RECEIPT AND INTERNAL CONTROL

---

records. All other procedures and documentation listed in this SOP must be followed at all times.

---

## 9.0    METHOD PERFORMANCE

Not applicable.

---

## 10.0   APPLICABLE DOCUMENTS/REFERENCES

"Handbook for Analytical Quality Control in Water and Wastewater Laboratories," U.S. EPA EMSL Office of Research and Development, March 1979.

Code of Federal Regulations 40, Parts 136 and 141.

"Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," SW-846 Chapters 1 & 2, USEPA, Third Edition, including Updates I, II, IIA, and IIB, III June, 1997.

Katahdin Analytical Services, Inc., Chemical Hygiene Plan and Safety Manual, current revision.

---

## LIST OF TABLES & FIGURES

Table 1	Sampling and Preservation Requirements
Figure 1	Example of Katahdin Chain-of-Custody Form
Figure 2	Example of Sample Receiving Logbook
Figure 3	Example of Katahdin Sample Receipt Condition Report
Figure 4	Example of Sample Filtration Logbook
Figure 5	Measured Turbidity and Preservation of Incoming Samples Logbook
Figure 6	Example Shipping Form - Federal Express Airbill
Figure 7	Example of Laboratory Incoming Sample Information Sheet (ISIS)
Figure 8	Example of Non-EPA CLP DC-1 Form
Figure 9	Example of Wet Chemistry Shorts and Rushes Logbook
Figure 10	Example Katahdin Work order/login COC Report
Figure 11	Example of Katahdin Internal Chain-of-Custody Form
Figure 12	Example of Refrigerator Temperature Logbook
Figure 13	Example of Immediate Internal COC Logbook

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1  
SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
<b>GENERAL CHEMICAL ANALYSES</b>					
Acidity	305.1	100 mL	P,G	1,2	14 days
Alkalinity-Manual Titrimetric	310.1	100 mL	P,G	1,2	14 days
Ammonia-Nitrogen with distill-Auto. Phenate	350.1	1 L	P,G	1,3	28 days
Ammonia-Nitrogen-Automated Phenate	350.1, 350.2	250 mL	P,G	1,3	28 days
Anions (Cl, Br, SO4, NO2, NO3)	300.0	250 mL	P, G	1	48hr/28days
Bicarbonate, Carbonate (see pH & alkalinity)	calc.				
Biochemical Oxygen Demand-Carbonaceous	405.1	1 L	P,G	1	48 hours
Biochemical Oxygen Demand-Total	405.1	1 L	P,G	1	48 hours
Bromide	320.1	500 mL	P,G	1	28 days
Chemical Oxygen Demand-Manual Colorimetric	410.4	100 mL	P,G	1,3	28 days
Chloride-Automated Ferricyanide	325.2	100 mL	P,G	1	28 days
Chlorine, Residual	SM4500-Cl G	100 mL	P,G	1,9	ASAP
Chromium, Hexavalent	SM3500Cr D / SW7196	200 mL	P,G	1,9	24 hours
Color, Apparent	110.2	100 mL	P,G	1,2	48 hours
Cyanide, Amenable-Spectrophotometric	335.1	250 mL	P,G	1,5	14 days
Cyanide, Total-Spectrophotometric	SM4500CN C 335.3, 335.4	250 mL	P,G	1,5	14 days
Dissolved Oxygen(Lab)-Membrane Electrode	360.1	500 mL	G	1	ASAP
Ferrous Iron - Colorimetric	SM3500-Fe D	250mL	P	1	24 hrs
Fluoride with distillation, Potentiometric ISE	SM4500F C/340.2	500 mL	P only	1	28 days
Fluoride, Potentiometric ISE	340.2	200 mL	P only	1	28 days
Free CO <sub>2</sub>	SM4500-CO <sub>2</sub> C	250mL	P	1	24 hrs.
Hardness, Total-Manual Titrimetric	130.2,SM2340C	250 mL	P,G	4	6 months
MBAS, Extraction-Colorimetric	SM5540C	1 L	P,G	1	48 hours
Nitrate+Nitrite-Automated Cadmium Reduction	353.2	100 mL	P,G	1,3	28 days
Nitrate-Automated Cadmium Red./Diazotization	353.2	100 mL	P,G	1	48 hours
Nitrite-Automated Diazotization	353.2	100 mL	P,G	1	48 hours
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	1664	(2) 1 L	glass only	1,11	28 days
pH (Laboratory)	150.1	100 mL	P,G	1,2	24 hours
Phenolics, Total Recoverable-Manual 4AAP	420.1	1000 mL	glass only	1,3	28 days
Phosphate, Ortho- Ascorbic Acid	365.2	100 mL	P,G	1	48 hours
Phosphate,Total	365.4	100 mL	P,G	1,3	28 days
Solids-Filterable Residue (TDS),Gravimetric180	160.1	250 mL	P,G	1	7 days
Solids-Nonfilterable Residue (TSS)	160.2	500 mL	P,G	1	7 days
Solids-Settleable Solids (SS)	160.5	1 L	P,G	1	48 hours

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
<b>GENERAL CHEMICAL ANALYSES</b>					
Solids-Total Solids	160.3	250 mL	P,G	1	7 days
Solids-Total Volatile (TVS)	160.4	250mL	P,G	1	7 days
Solids-Volatile Filterable Residue (VDS)	160.1/160.4	250 mL	P,G	1	7 days
Solids-Volatile Nonfilterable Residue (VSS)	SM 2540 F	500 mL	P,G	1	7 days
Specific Conductance-Wheatstone Bridge	120.1	100 mL	P,G	1,2	28 days
Sulfate-Turbidimetric	375.4	100 mL	P,G	1	28 days
Sulfide-Iodometric	376.1	500 mL	P,G	1,7	7 days
Sulfite-Titrimetric	377.1	500 mL	P,G	1,9	ASAP
Tannin/Lignin-Colorimetric	SM 5550 B	100 mL	P,G	1	7 days
TKN-Auto Block Digest, Spect.	351.2	100 mL	P,G	1,3	28 days
Total Inorganic Carbon	415.1	(2) 40 mL	VOA vial	1	28 days
Total Inorganic Carbon if with TOC	415.1	(2) 40 mL	VOA vial	1	28 days
Total Organic Carbon-Oxidation	415.1	(2) 40 mL	VOA vial	1,3	28 days
Total Organic Halogen	9020	500 mL	Amber Glass	1,3	28 days
Turbidity	180.1	100 mL	P,G	1	48 hours
<b>ELEMENTAL ANALYSES</b>					
Chromium, Hexavalent	7196/6010	500 mL	P,G	1,9	24 hrs
GFAA(Furnace) Elements	SM 3113/ 200 series	500 mL	P,G	4	6 months
ICP Elements	200.7/6010	500 mL	P,G	4	6 months
ICP MS Elements	200.8/6020	500 mL	P,G	4	6 months
Low Level Mercury	1631	500 mL	G	NA	90 days
Mercury	245.1/7470	500 mL	P,G	4	28 days
<b>GC ORGANIC ANALYSES</b>					
BTEX & MTBE	602 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(-)
EDB, DBCP & 1,2,3-TCP	504.1	(2) 40 mL	VOA vial	1,8,9	14 days(-)
Extractable Petroleum Hydrocarbons	MADEP/EPH	(2) 1000 mL	Amber Glass	12	14days/40days
Formaldehyde	556	(2) 40 mL	VOA vial	1,8,9	14 days(-)
Fuel Oil in Water	8015Modified	(2) 1000 mL	Amber Glass	1,8	7days/40days
Fuel Oil in Water	ME HETL 4.1.25	(2) 1000 mL	Amber Glass	1,8	7days/40days
Gasoline in Water	8015Modified	(2) 40 mL	VOA vial	1,8	14 days
Gasoline in Water	ME HETL 4.2.17	(2) 40 mL	VOA vial	1,8	14 days
Glycols	8015Modified	(2) 40 mL	VOA vial	1,8,9	14 days(-)
Herbicides	8151	(2) 1000 mL	Amber Glass	1	7days/ 40days
Methane, Ethane & ethene	RSK 175	(2) 40 mL	VOA vial	1,8,9	14 days(-)
PCB's	608 & 8082	(2) 1000 mL	Amber Glass	1	7days/40days

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
<b>GC ORGANIC ANALYSES</b>					
Pesticides	608 & 8081	(2) 1000 mL	Amber Glass	1	7days/40days
Pesticides and PCB's	608 & 8081/8082	(2) 1000 mL	Amber Glass	1	7days/40days
Purgeable Aromatics	602 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(-)
Purgeable Halocarbons	601 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(-)
Purgeables, Total	601 & 602	(2) 40 mL	VOA vial	1,8,9	14 days(-)
Purgeables, Total	8021	(2) 40 mL	VOA vial	1,8,9	14 days(-)
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP/VPH	(2) 40 mL	VOA vial	11	14days
<b>GC/MS ORGANIC ANALYSES</b>					
Acid Extractables-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Acid Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extract.-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Drinking Water Volatiles - Low Level	524.2	(3) 40 mL	VOA vial	1,8,9,10	14 days(-)
PCB Homologues	680	(2) 1000 mL	Amber Glass	1	7days/40days
Polyaromatic Hydrocarbons	8270/8270 SIM	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Volatile Organics	8260	(2) 40 mL	VOA vial	1,8,9	14 days(-)
Volatile Organics-Priority Pollutants	624	(2) 40 mL	VOA vial	1,8,9	14 days(-)
<b>HPLC ANALYSES</b>					
HPLC-Explosives	8330, 8332	(2) 1000 mL	Amber Glass	1	7days/40days
<b>MICROBIOLOGICAL ANALYSES</b>					
Coliform, Fecal	SM 9222D	100 mL	P,G	1,6	6 hours
Coliform, Total	SM 9222B	100 mL	P,G	1,6	30 hours
Coliform and E-coli, Total	SM9223B	100 mL	P,G	1,6	30 hours
E-coli	SM9213D	100 mL	P,G	1,6	6 hours
Heterotrophic Plate Count	SM9215B	100 mL	P,G	1,6	30 hours

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1 (cont.)  
SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
<b>GENERAL CHEMICAL ANALYSES</b>		<b>4 oz=100 g</b>			
% Carbon	9060 mod.	4 oz	Soil Jar	1	28 days
Ammonia-Nitrogen-Automated Phenate	350.1 mod.	4 oz	Soil Jar	1	28 days (^)
Anions	9056	4 oz	Soil Jar	1	48hrs to 28 days from slurry (^)
Cation Exchange Capacity	9081	4 oz	Soil Jar	1	14days/7days (^)
Chloride-Automated Ferricyanide	9251/300.0	4 oz	Soil Jar	1	28days from slurry (^)
Cyanide, Amenable-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Cyanide, Total-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Fluoride, Potentiometric ISE	300.0 mod./340.2	4 oz	Soil Jar	1	28 days (^)
Lime Equivalency	310.1 mod.	4 oz	Soil Jar	1	28 days (^)
Nitrate+Nitrite-Automated Cadmium Reduction	300.0 mod./353.2	4 oz	Soil Jar	1	28 days (^)
Nitrate-Automated Cadmium Red./Diazotization	300.0 mod./353.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Nitrite-Automated Diazotization	300.0 mod./353.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	9071	4 oz	Soil Jar	1	28 days (^)
Organic Nitrogen-Auto. Block Digest.,Spectro.	350.1/351.2 mod.	4 oz	Soil Jar	1	28 days (^)
pH (Laboratory)	9045	4 oz	Soil Jar	1	24 hours (^)
Phenolics, Total Recoverable-Manual 4AAP	Mod. 9065	4 oz	Soil Jar	1	28 days (^)
Phosphate, Ortho- Ascorbic Acid	300.0 mod./365.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Phosphate,Tot.-Auto Ascorbic Acid/Block Dig.	Mod. 365.4	4 oz	Soil Jar	1	28 days (^)
Solids-Ash	SM 2540 F	4 oz	Soil Jar	1	28 days (^)
Solids-Total Solids	CLP-CIP	4 oz	Soil Jar	1	28 days (^)
Solids-Volatile Solids	SM 2540 F	4 oz	Soil Jar	1	28 days (^)
Specific Conductance-Wheatstone Bridge	Mod. 9050	4 oz	Soil Jar	1	28 days (^)
Sulfate-Turbidimetric	9036/9038	4 oz	Soil Jar	1	28 days from slurry (^)
Sulfide-Iodometric	9030	4 oz	Soil Jar	1	7days from slurry (^)
Sulfide-Monier-Williams	40CFR-425	4 oz	Soil Jar	1	28 days (^)
Sulfite-Titrimetric	ASTM D3987/377.1 mod.	4 oz	Soil Jar	1	24 hrs from slurry (^)
TKN-Auto Block Digest,Spectro.	351.2 mod.	4 oz	Soil Jar	1	28 days (^)
Total Organic Halogen	9020/9021	4 oz	Soil Jar	1	28 days (^)
Total Petroleum Hydrocarbons-Extraction, IR	9071	4 oz	Soil Jar	1	28 days (^)
<b>ELEMENTAL ANALYSES</b>					
ICP Elements	6010	4 oz	Soil Jar	1	6 months
ICP MS ELelements	6020	4 oz	Soil Jar	1	6 months
GFAA(Furnace) Elements	7000series	4 oz	Soil Jar	1	6 months
Mercury	7471	4 oz	Soil Jar	1	28 days

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1 (cont.)  
SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
<b>ELEMENTAL ANALYSES (cont.)</b>		<b>4 oz=100 g</b>			
Chromium, Hexavalent	3060/7196	4 oz	Soil Jar	1	30dys/24hrs
<b>GC ORGANIC ANALYSES</b>					
BTEX & MTBE	8021	(2) 40 mL	VOA Vial	1	14 days
Explosives - HPLC	8330, 8332	4 oz	Soil Jar	1	14days/40days
Extractable Petroleum Hydrocarbons	MADEP/EPH	4 oz	Soil Jar	1	7days/40days
Fuel Oil	ME HETL 4.1.25	4 oz	Soil Jar	1	14days/40days
Fule Oil	8015 mod.	4 oz	Soil Jar	1	14days/40days
Gasoline	ME HETL 4.2.17	(2) 40 mL	VOA Vial	1	14 days
Gasoline	8015 mod.	(2) 40 mL	VOA Vial	1	14 days
Herbicides	8151	4 oz	Soil Jar	1	14days/40days
PCB's	8082	4 oz	Soil Jar	1	14days/40days
PCB's in Oil	8082	4 oz	VOA Vial	1	40 days
Pesticides	8081	4 oz	Soil Jar	1	14days/40days
Pesticides and PCB's	8081/8082	4 oz	Soil Jar	1	14days/40days
Purgeable Aromatics	8021	(2) 40 mL	VOA Vial	1	14 days
Purgeable Halocarbons	8021	(2) 40 mL	VOA Vial	1	14 days
Purgeables, Total	8021	(2) 40 mL	VOA Vial	1	14 days
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA Vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP/VPH	(2)40 mL	VOA vial	13	28days
<b>HPLC ANALYSES</b>					
HPLC-Explosives	8330, 8332	4 oz	Soil Jar	1	7days/40days
<b>GC/MS ANALYSES</b>					
Acid Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Acid Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Polyaromatic Hydrocarbons	8270/8270SIM	4 oz	Soil Jar	1	14 days/40 days
Semivolatile Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Semivolatile Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Volatile Organics – High Soil (>200 ug/kg)	5035/8260	Please refer to Table 6-2	Encore or similar sampler or VOA Vial or soil jar	14	Extruded w/in 48 hrs. Analyzed w/in 14 days
Volatile Organics – Low Soil (<200 ug/kg)	5035/8260	Please refer to Table 6-2	Encore or similar sampler or VOA Vial	14 or 15	Extruded w/in 48 hrs. Analyzed w/in 14 days
Volatile Organics-Priority Pollutants	8260	(2) 40 mL	VOA Vial	1	14 days
Volatile Organics-TCL	8260	(2) 40 mL	VOA Vial	1	14 days

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
<b>RCRA - HAZARDOUS WASTE CHARAC.</b>					
Corrosivity-pH	9045	4 oz	Soil Jar	1	24 hours (^)
Ignitability-Flash Point (closed cup)	1010	4 oz	Soil Jar	1	14 days (^)
Reactivity-Reactive Cyanide	7.3.3.2	4 oz	Soil Jar	1	14 days
Reactivity-Reactive Sulfide	7.3.4.1	4 oz	Soil Jar	1	7 days
<b>TCLP</b>					
TCLP Extraction-Volatile Organics	1311	100 g	Soil Jar	1	14 days
TCLP Extraction-Semivolatiles	1311	200 g	Soil Jar	1	14 days
TCLP Extraction-Pesticides & Herbicides	1311	400 g	Soil Jar	1	14 days
TCLP Extraction-Metals	1311	200 g	Soil Jar	1	28 days
TCLP Analysis-Volatile Organics	8260	see above	Soil Jar	1	14 days
TCLP Analysis-Metals	6010/6020	see above	Soil Jar	1	180 days
TCLP Analysis-Mercury	7470	see above	Soil Jar	1	28 days
TCLP Analysis-Semivolatiles	8270	see above	Soil Jar	1	7 days/40 days
TCLP Analysis-Pesticides	8081	see above	Soil Jar	1	7 days/40 days
TCLP Analysis-Herbicides	8151	see above	Soil Jar	1	7 days/40 days

METHODS OF PRESERVATION
1 = Cool at 4 Degrees Celsius
2 = Settled
3 = H2SO4 to pH<2
4 = HNO3 to pH<2
5 = NaOH to pH>12
6 = 1 mL 0.1M Na2S2O3 or 1 10 mg pellet
7 = 1 mL 2NZnAc/L & NaOH
8 = 2 drops 1:1 HCl
9 = No headspace
10 = Na2S2O3, if chlorinated
11 = HCl to pH < 2
12 = 5 mL of HCL
13 = 15 mL of methanol
14 = methanol
15 = sodium bisulfate



TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 2

EXAMPLE OF KATAHDIN SAMPLE RECEIPT LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

SAMPLE LOG IN

Date Received	Time Received	Date Logged In	Time Logged In	Work Order	Client	Initials
				SA 0094		
				SA 0095		
				SA 0096		
				SA 0097		
				SA 0098		
				SA 0099		
				SA 0100		
				SA 0101		
				SA 0102		
				SA 0103		
				SA 0104		
				SA 0105		
				SA 0106		
				SA 0107		
				SA 0108		
				SA 0109		
				SA 0110		
				SA 0111		
				SA 0112		
				SA 0113		
				SA 0114		
				SA 0115		
				SA 0116		
				SA 0117		
				SA 0118		
				SA 0119		
				SA 0120		
				SA 0121		
				SA 0122		
				SA 0123		
				SA 0124		

Signed By: \_\_\_\_\_

Date: \_\_\_\_\_

Reviewed By: \_\_\_\_\_

Date: \_\_\_\_\_







TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 6

EXAMPLE SHIPPING FORM – FEDERAL EXPRESS AIRBILL

996  
1000 **FedEx USA Airbill** Express Tracking Number **8427 1827 3180**

**1 From** This portion can be removed for Recipient's records.  
Date 2/12/04 FedEx Tracking Number 842718273180

Sender's Name DISTRIBUTION Phone [REDACTED]

Company [REDACTED]

Address [REDACTED]

City JACKSONVILLE State FL ZIP 32256-1208

**2 Your Internal Billing Reference** 112600436

**3 To** Recipient's Name ANDREA COLBY Phone 287 874-7400

Company KATAHDIN

Address 600 TECHNOLOGY WAY

City WARREN State ME ZIP 04074

**8427 1827 3180**



**Recipient's Copy**

**4a Express Package Service** Packages up to 150 lbs. Delivery commitment may be made in some areas.

FedEx Priority Overnight Next business morning.  FedEx Standard Overnight Next business day.  FedEx First Overnight Next business morning. Delivery to select locations.

FedEx 2Day Next business day.  FedEx Express Saver Next business day.  FedEx 2Day Freight Next business day.  FedEx 3Day Freight Third business day.

**4b Express Freight Service** Packages over 150 lbs. Delivery commitment may be made in some areas.

FedEx 1Day Freight\* Next business day.  FedEx 2Day Freight Next business day.  FedEx 3Day Freight Third business day.

**5 Packaging** \*Features subject to BSM

FedEx Envelope\*  FedEx Pak\*  Other

**6 Special Handling** Includes FedEx address on package.

SATURDAY Delivery Available with FedEx Priority Overnight, FedEx 2Day, FedEx Priority Overnight and FedEx Standard Overnight ZIP codes.  HOLD Weekday at FedEx Location Not available for FedEx Priority Overnight and FedEx Standard Overnight.  HOLD Saturday at FedEx Location Available with FedEx Priority Overnight and FedEx Standard Overnight.

Does this shipment contain dangerous goods?  No.  Yes.  Yes.  Yes.  Dry Ice.  Cargo Aircraft Only.

**7 Payment** Bill to:  Shipper  Recipient  Third Party  Credit Card  Cash/Check

Total Packages 1 Total Weight 51.00 lbs Total Charges [REDACTED]

**8 Release Signature** Sign to authorize delivery without company signature.

**447**

**NO POUCH NEEDED.**  
See back for peel and stick application instructions.

RECIPIENT: PEEL HERE



TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 8  
 EXAMPLE OF NON-EPA CLP DC-1 FORM

SAMPLE LOG-IN SHEET

Lab Name: <u>Katahdin Analytical Services, Inc.</u>		Page _____ of _____		
Received By (Print Name): _____		Log-in Date: _____		
Received By (Signature): _____				
Case Number: _____		<b>CORRESPONDING</b>		
Sample Delivery				
Group No.: _____		<b>CLIENT SAMPLE #</b>	<b>SAMPLE TAG #</b>	<b>ASSIGNED LAB #</b>
SAS Number: _____				<b>REMARKS: CONDITION OF SAMPLE SHIPMENT, ETC.</b>
<b>REMARKS:</b>				
1. Custody Seal(s)	Present/Absent* Intact/Broken			
2. Custody Seal Nos.: _____				
3. Chain-of-Custody Records	Present/Absent*			
4. Traffic Reports or Packing List	Present/Absent*			
5. Airbill	Airbill/Sticker Present/Absent*			
6. Airbill No.: _____				
7. Sample Tags	Present/Absent*			
Sample Tag Numbers	Listed/Not Listed on Chain-of-Custody			
8. Sample Condition:	Intact/Broken*/ Leaking			
9. Does information on custody records, traffic reports, and sample tags agree?	Yes/No*			
10. Date Received at Lab: _____				
11. Time Received: _____				
<b>Sample Transfer</b>				
Fraction: _____				
Area #: _____				
By: _____				
On: _____				

\* Contact Client and attach record of resolution

Reviewed By: \_\_\_\_\_  
 Date: \_\_\_\_\_

Logbook No.: \_\_\_\_\_  
 Logbook Page No.: \_\_\_\_\_



TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 10

EXAMPLE OF KATAHDIN WORK ORDER/LOGIN COC REPORT



**Account:** KATAHD001  
Katahdin Analytical Services

**Project:**

**Primary Report Address:**  
Leslie Dimond  
Katahdin Analytical Services  
600 Technology Way  
P.O. Box 540  
Scarborough, ME 04070

**Primary Invoice Address:**  
Accounts Payable  
Katahdin Analytical Services  
600 Technology Way  
P.O. Box 540  
Scarborough, ME 04070

**Report CC Addresses:**  
**Invoice CC Addresses:**

**Katahdin Analytical Services**  
**Login Chain of Custody Report (Ino1)**  
Jan. 26, 2007  
03:51 PM

**Login Information**

ANALYSIS INSTRUCTIONS :  
CHECK NO. :  
CLIENT PO# :  
COOLER TEMPERATURE : n/a  
DELIVERY SERVICES : In House  
EDD FORMAT :  
MAIL DATE :  
PM : LAD  
PROJECT NAME : QC Holding Blanks  
QC LEVEL : I  
REGULATORY LIST :  
REPORT INSTRUCTIONS :  
SDG ID :  
SDG STATUS :

Page: 1 of 1

Laboratory Sample ID	Client Sample Number	Collect Date/Time	Receive Date	Verbal PR	Due Date	Comments
SA0395-1	WHITE FRIDGE	26-JAN-07 15:50	26-JAN-07		08-FEB-07	
<i>Metric</i>	<i>Product</i>	<i>Hold Date (shortest)</i>	<i>Bottle Type</i>	<i>Bottle Count</i>		
Aqueous	S SW8260FULL5ML	09-FEB-07		2		
SA0395-2	BLUE FRIDGE	26-JAN-07 15:50	26-JAN-07		08-FEB-07	
<i>Metric</i>	<i>Product</i>	<i>Hold Date (shortest)</i>	<i>Bottle Type</i>	<i>Bottle Count</i>		
Aqueous	S SW8260FULL5ML	09-FEB-07		2		

**Total Samples: 2                      Total Analyses: 2**



TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 12

EXAMPLE OF REFRIGERATOR TEMPERATURE LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

SAMPLE RECEIPT WALK-IN TEMPERATURE LOG

Corrective Action: Note in the "comments" column and notify the QAO or supervisor; document corrective actions taken and return to control.

Thermometer Location		Sample Receipt Walk-in 1	Comments
Acceptance Criteria		2 to 6 °C	
Date	Initials	Temp (°C)	
02/2/07	NDA	3.7	
02/05/07	DWM	5.8	
02/06/07	DWM	5.8	
02/07/07	DWM	5.8	
02/08/07	DWM	3.8	
02/09/07	DWM	3.5	
02/12/07	DWM	3.5	
02/13/07	DWM	3.5	

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 13

EXAMPLE OF IMMEDIATE INTERNAL COC LOGBOOK

**KATAHDIN ANALYTICAL SERVICES, INC.**  
**INTERNAL CUSTODY RECORD FOR IMMEDIATES**

QAQC143

CLIENT	PROJECT	CLIENT ID &/or WORK ORDER #	ANALYSIS	OUT date/time	IN date/time	INIT	Consumed?
Jacobs		WW4813-1A, -2A	ICP	9/13/06 0930	→ 0935	DJJ	yes <input checked="" type="checkbox"/> no
Jacobs		WW4883-1A	ICP	9/14/06 0100	→ 1000	DJJ	yes <input checked="" type="checkbox"/> no
CES		WW4965	BOD	9/20/06 0700	9/20/06 0700	CP	yes no
CCAB		WW4969	BOD	9/20/06 1000	↓	CP	yes no
GEMF		WW4970	BOD	↓	↓	CP	yes no
Jacobs		WW4962-1A, -2A	ICP	9/20/06 0900	→ 1000	DJJ	yes <input checked="" type="checkbox"/> no
Irving		WW4994	BOD	9/21/06 1000	9/21/06 1005	CP	yes <input checked="" type="checkbox"/> no
Hghlmer		WW4992 <sup>Ⓢ</sup>	BOD	9/21/06 1015		CP	yes no
NATIONAL		WW5000	TS, PEROXIDE PH, ST. BATH	9/21/06 1100	9/21/06 1217	↓	yes <input checked="" type="checkbox"/> no
WTC		WW5001	BOD	9/21/06 1300		CP	yes no
Ariens		WW5016	NO <sub>3</sub>	9/22/06 1100	9/22/06 1100	CP	yes <input checked="" type="checkbox"/> no
RANSOM		WW5010	↓	↓	↓	↓	yes <input checked="" type="checkbox"/> no
EconHaine		WW5029	BOD	9/22/06 1100		CP	yes no

0000095

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
ANALYSIS

Prepared By: Mike Thomas Date: 7/98

Approved By:

Group Supervisor: Michael Thomas Date: 11/15/00

Operations Manager: J. Bentler Date: 11/15/00

QA Officer: Deborah J. Nadeau Date: 11.16.00

General Manager: Deborah F. Neff Date: 11/20/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	11.16.00	11/16/00
02	Definitions added to section 1.1. Wording was added or changed to clarify sections 4, 5, 6, 7, 8+9. Minor changes throughout. New figures.	MRC	11.09.04	11.09.04
03 LND 6-26-06	Updated Sect. 7.0 to include SIM. Updated figures 2 and 3 to include current SVOA <sup>compounds</sup> mixers used. updated Sect. 5.0 to include all compounds analyzed for. updated logbook page. minor edits throughout.	LAD	04/06	04/06

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
ANALYSIS**

---

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

---

I acknowledge receipt of copy \_\_\_ of document **SOP CA-526-03**, titled **PREPARATION OF  
SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR  
SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS** .

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_ of document **SOP CA-526-03**, titled **PREPARATION OF  
SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR  
SUBSEQUENT EXTRACTABLE SEMIVOLATILE ANALYSIS** .

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
ANALYSIS**

---

## **1.0 SCOPE AND APPLICATION**

Method 3540 is a procedure for extracting semivolatile organic compounds from solids such as soils, sludges, and wastes using Method 3540. The Soxhlet extraction process ensures intimate contact the sample matrix with the extraction solvent.

This method is applicable to the isolation and concentration of water-insoluble and slightly water soluble organics in preparation for a variety of chromatographic procedures.

### **1.1 Definitions**

**METHOD BLANK** (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

**LABORATORY CONTROL SAMPLE (LCS)**: A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

**MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)**: Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

**SURROGATES**: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

### **1.2 Responsibilities**

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
ANALYSIS**

---

Katahdin SOP QA-805, current revision, "Personnel Training and Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that the members of his/her group follow this SOP, to assure that their work is properly documented, and to indicate periodic review of the pertinent logbooks.

### 1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

### 1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual.

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
ANALYSIS**

---

**2.0 SUMMARY OF METHOD**

- 2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.
  - 2.2 The extract is then dried and concentrated for subsequent 8270 Semivolatile Organics analysis.
- 

**3.0 INTERFERENCES**

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be needed to minimize interferences.

---

**4.0 APPARATUS AND MATERIALS**

- 4.1 a) Soxhlet extractor – 45/50 top joint and 24/40 lower joint.
  - b) 500 mL flat-bottom boiling flask

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
ANALYSIS**

---

- c) Allihn cooling water condenser
- 4.2 Powder Funnels – 100 mm top diameter, 35 mm stem
- 4.3 Kuderna-Danish (K-D) apparatus
  - 4.3.1 Concentrator tube - 10-mL
  - 4.3.2 Evaporation flask - 500-mL
  - 4.3.3 Snyder column - Three-ball macro
- 4.4 Nitrogen evaporation (N-EVAP) apparatus.
- 4.5 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride
- 4.6 Water bath - Heated, with concentric ring cover, capable of temperature control ( $\pm 5^{\circ}\text{C}$ ). The bath should be used in a hood.
- 4.7 Vials - Glass, 1.8-mL capacity, with polytetrafluoroethylene (PTFE)-lined septum vials.
- 4.8 Glass wool (fiberglass) - baked at  $400^{\circ}\text{C}$  for a minimum of 4 hours or overnight.
- 4.9 Heating mantles - Rheostat controlled.
- 4.10 Disposable glass pasteur pipets, 5  $\frac{3}{4}$ " and bulbs.
- 4.11 Drying oven - capable of maintaining  $105^{\circ}\text{C}$  for glassware drying.
- 4.12 Muffle oven – capable of maintaining  $400^{\circ}\text{C}$  for baking glass wool and organic-free sand.
- 4.13 Beakers, 250 or 400 mL
- 4.14 Top-loading balance - capable of weighing to 0.1 g.
- 4.15 Spatulas, stainless-steel
- 4.16 Long forceps, stainless-steel

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
ANALYSIS**

---

4.17 Metal clips – for securing Soxhlets to boiling flasks

---

**5.0 REAGENTS**

- 5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, certified by the manufacturer/vendor as purified heating to 400°C prior to receipt by the laboratory.
- 5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated, by lot, prior to use, by concentration of approximately 400 mL to 1.0 mL followed by GC/MS analysis.
- 5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand.
- 5.4 Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations.

Compound	Conc.
phenol- <sub>d5</sub>	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene- <sub>d5</sub>	50 ug/mL
terphenyl- <sub>d14</sub>	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

- 5.5 Combined SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
ANALYSIS**

---

<b>Compound</b>	<b>Conc. ug/mL</b>
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
Pentachlorophenol	2.0 ug/mL
Tribromophenol	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

- 5.6 Base/Neutral and Acid (SVOA) Lab Control Sample / Matrix Spike Spiking Solution - Prepare a spiking solution in methanol that contains the following mixes listed in Figure 2 at a concentration of 50 ug/ml for the base/neutral compounds and 100 ug/ml for the acid compounds. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.
- 5.7 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2 ug/mL for base/neutral. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL of methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.
- 5.8 Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution – Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at concentrations of 100 ug/ml. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

---

**6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING**

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
ANALYSIS**

---

Holding time for extraction of sediment/soil samples for Method 3540 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C ( $\pm 2^\circ\text{C}$ ) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

---

## **7.0 PROCEDURES**

### **7.1 Preparing the Soxhlet Extraction Apparatus**

7.1.1 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.

7.1.2 Add ~ 250 mLs of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Using stainless steel forceps and working in a hood, place a plug of the pre-baked glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample.

### **7.2 Sample Handling**

7.2.1 Sediment/soil samples - Decant and discard any water layer on a sediment sample. Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2 Gummy, fibrous, or oily materials not amenable to mixing should be cut, shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate to

---

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
ANALYSIS**

---

disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

- 7.3 The following steps should be performed rapidly to avoid loss of the more volatile extractables. Weigh out a  $30.0 \pm 0.1$  g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.1 g in appropriate extraction logbook. Add between 30 g and 60 g of anhydrous powdered sodium sulfate as required to produce a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.
- 7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one  $30.0 \pm 0.1$  g portion of purified sand in a labeled 400 mL beaker. Add 60 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)
- 7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one  $30.0 \pm 0.1$  g portion of purified sand in a labeled 400 mL beaker. Add 30 g sodium sulfate and mix well. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.
- 7.6 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out 30 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.1 g in appropriate extraction logbook. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well.
- 7.7 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be careful not to have any of the solid material fall into the extract flask through the large vapor tube.
- 7.8 To all samples, method blank, LCS, and MS/MSD add 1.0 mL of the appropriate base/neutral and acid surrogate spiking solution listed below using the pre-rinsed 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
ANALYSIS**

---

extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution.

- 7.8.1 If the request is for SVOA, use the SVOA Surrogate Solution.
- 7.8.2 If the request is for SIM-SVOA, use the SIM-SVOA surrogate solution as well as SVOA surrogate solution.
- 7.9 To the LCS and the MS/MSD add 1.0 mL of the appropriate base/neutral and acid (SVOA) matrix spike/LCS spiking solution listed below using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent when spiking is completed.
  - 7.9.1 If the request is for SVOA, use the SVOA Spiking Solution.
  - 7.9.2 If the request is for SIM-SVOA, use the SIM-SVOA Spiking solution as well as the SVOA spiking solution.
  - 7.9.3 If the request is for SVOA Appendix IX, use the SVOA Appendix IX Spiking solution as well as the SVOA spiking solution
- 7.10 Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water condensers into the 45/50 joints of the extractors. Save the pieces of aluminum foil for covering the Soxhlets when the extraction is complete. Switch on the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 55-60% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until the level reaches that of the small siphon tube and then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 18-24 hours. Be sure the chiller/recirculator temperature is set low enough to provide enough cooling capacity for the number of extractions in the batch.
- 7.11 When the extraction is complete, allow the extracts to cool before dismantling. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Remove the Allihn condenser and replace the aluminum foil on top of the extractor. Move the extractors to a hood and detach the extractor from the extract flask. Try to drain as much solvent as possible from the extractor into the flask. Cover the flask with aluminum foil and

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
ANALYSIS**

---

store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

- 7.12 Immediately remove the extracted soil/sodium sulfate mixtures from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries.

#### CONCENTRATION OF EXTRACTS

- 7.13 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride. Place the assembled K-D's under the funnels.
- 7.14 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow to drain.
- 7.15 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.
- 7.16 If samples are not to be GPC'd follow Steps 7.17 through 7.22 to concentrate extracts to final volume of 1 mL. Otherwise proceed to GPC cleanup procedure as described in the current revision of Katahdin SOP CA-513.
- 7.17 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel, add one or two clean boiling stones to the K-D evaporative flask and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.
- 7.18 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the

---

TITLE:     **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
              USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
              ANALYSIS**

---

chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches  $\approx$  4-6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-extraction must occur immediately.** Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with  $\approx$  1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with  $\approx$  1 mL methylene chloride.

- 7.19 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with  $\approx$ 1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N<sub>2</sub> sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.
- 7.20 When the apparent volume reaches  $\approx$ 0.5 mL, remove the concentrator tube and allow it to cool.
- 7.21 Complete the quantitative transfer of the extract to a 1.8 mL vial by using methylene chloride. Adjust the volume of the methylene chloride extract to 1.0 mL using the 1.8 mL reference vial for volume comparison.
- 7.22 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of  $4 \pm 2$  °C until ready for analysis. Indicate in the extractions logbook the box number and "tray location" of the individual extract vials.

---

**8.0     QUALITY CONTROL AND ACCEPTANCE CRITERIA**

A method blank must be extracted for each and every item listed below:

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
ANALYSIS**

---

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of extractable semivolatile organics for quality control acceptance criteria.

Each extraction analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
ANALYSIS**

---

the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

---

### **9.0 METHOD PERFORMANCE**

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

---

### **10.0 APPLICABLE DOCUMENTS/REFERENCES**

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3540C, SW-846, Third Edition, Updates I, II, IIA, IIB, and III Revised December 1996, US EPA.

---

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
ANALYSIS**

---

LIST OF TABLES AND FIGURES

Table 1	Summary of Method Modifications
Figure 1	Example of Logbook Page
Figure 2	LCS/Matrix Spike Component List
Figure 3	Appendix IX LCS/Matrix Spike Component List

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
 USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
 ANALYSIS**

TABLE 1  
 SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-526-03	METHOD 3540, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures	1. Use 30 grams of sample and 30 grams of sodium sulfate. 2. Use 250 mL of methylene chloride	1. Use 10 grams of sample and 10 grams of sodium sulfate. 2. Use 300 mL of methylene chloride
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		



**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1,1-Biphenyl	Bis (2-Chloroisopropyl) ether)
1,2,4-Trichlorobenzene	Bis (2-ethylhexyl) phthalate
1,2-Dichlorobenzene	Butylbenzyl phthalate
1,3-Dichlorobenzene	Caprolactam
1,4-Dichlorobenzene	Carbazole
2,4-Dinitrotoluene	Chrysene
2,6-Dinitrotoluene	Dibenz (a, h) anthracene
2-Chloronaphthalene	Dibenzofuran
2-Methylnaphthalene	Diethyl phthalate
2-Nitroaniline	Diethyladipate
3,3'-Dichlorobenzidine	Dimethyl phthalate
3-Nitroaniline	Di-n-butylphthalate
4-Bromophenylphenyl ether	Di-n-octyl phthalate
4-Chloroaniline	Fluoranthene
4-Chlorophenylphenyl ether	Fluorene
4-Nitroaniline	Hexachlorobenzene
Acenaphthene	Hexachlorobutadiene
Acenaphthylene	Hexachlorocyclopentadiene
Acetophenone	Hexachloroethane
Aniline	Indeno (1,2,3-cd) pyrene
Anthracene	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	o-toluidine
Benzo (ghi) perylene	Phenanthrene
Benzo (k) fluoranthene	p-toluidine
Benzyl alcohol	Pyrene
Bis (2-chloroethoxy) methane	Pyridine
Bis (2-chloroethyl) ether	

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2,4-Dinitrophenol	4-Methylphenol
2,3,4,6-Tetrachlorophenol	2,6-Dichlorophenol	4-Nitrophenol
2,4,5-Trichlorophenol	2-Chlorophenol	Benzoic acid
2,4,6-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4-Dichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dimethylphenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
	4-Chloro-3-methylphenol	Phenol

**TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION  
USING METHOD 3540 FOR SUBSEQUENT EXTRACTABLE SEMIVOLATILE  
ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachlorpropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthaquinone	Isosafrole
1-Chloronaphthalene	Kepona
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotropiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylbenz(a)anthracene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitrobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Proamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270  
– Modified for Selected Ion Monitoring (SIM)**

Prepared By: GC/MS Department Date: 6/98

Approved By:

Group Supervisor: J. Galay Date: 020101

Operations Manager: John C. Benton Date: 1/31/01

QA Officer: Deborah J. Nadeau Date: 1.31.01

General Manager: Dennis F. Keegan Date: 2/01/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8270C Mod.	Format changes added pollution prevention, added instrument and other calibration options. Other minor changes to sections 7, 8 & QA Table.	DN	1/31/01	1/31/01
02 8270C	Many changes in formatting. Some additions to sections 8 & TABLE 1 to comply with NAVY.	DN	09/30/04	09/30/04
03 8270C	Sect. 7.2: Removed "K" Instrument & added "R" instrument. Added Pentafluorophenol surr. to Tables 3, 5 and Sect. 8.2. Removed all references to TIC <sup>5</sup> .	LAD	04/06	04/06

---

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

---

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

---

I acknowledge receipt of copy \_\_\_ of document **SOP CA-213-03**, titled **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270– Modified for Selected Ion Monitoring (SIM)**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_ of document **SOP CA-213-03**, titled **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270– Modified for Selected Ion Monitoring (SIM)**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD  
8270 – Modified for Selected Ion Monitoring (SIM)**

---

## **1.0 SCOPE AND APPLICATION**

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel to prepare and analyze water and soil sample extracts for semivolatile organics by EPA SW-846 Method 8270, current revision, modified for selected ion monitoring.

In order to maintain consistency in data quality, this SOP consolidates all aspects of the analyses in one working document, to be revised as necessary.

### **1.1 Definitions**

**ANALYTICAL BATCH:** 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

**METHOD BLANK (laboratory reagent blank):** An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

**CALIBRATION CHECK:** Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

**CALIBRATION STANDARD (WORKING STANDARD):** A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

**LABORATORY CONTROL SAMPLE (LCS):** A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

**MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD):** Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative

---

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

---

percent difference between the samples is calculated and used to assess analytical precision.

**STANDARD CURVE (CALIBRATION CURVE):** A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

**STOCK STANDARD SOLUTION:** A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

**SURROGATES:** Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

**TARGET:** A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

**TARGET DB:** An oracle database used to store and organize all Target data files.

**QUICKFORMS:** A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

## 1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of semivolatile organic compounds by EPA Method 8270. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of semivolatiles by Method 8270 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

---

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD  
8270 – Modified for Selected Ion Monitoring (SIM)**

---

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Management Plan and follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves, and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, autosampler vials containing sample extracts in methylene chloride are returned to the SVOA hood, and the contents transferred to a labeled waste container. The contents of this container are disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

---

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

---

## **2.0 SUMMARY OF METHOD**

The process involves the extraction of semivolatiles from a sample using an appropriate solvent followed by clean up steps (where applicable) and concentration of the extract (refer to Katahdin SOP CA-502, "Preparation of Aqueous Samples for Extractable Semivolatile Analyses", SOP CA-512, "Preparation of Sediment/Soil Samples by Sonication Using Method 3550 for Subsequent Extractable Semi-Volatiles Analysis" and SOP CA-526, "Preparation of Sediment/Soil Samples by Soxhlet Extraction Using Method 3540 for Subsequent Extractable Semivolatile Analysis"). An aliquot of the final extract is injected into the gas chromatograph for compound separation by capillary column, followed by the electron impact mass spectrometer for identification and quantitation.

---

## **3.0 INTERFERENCES**

Interfering contamination may occur when a sample containing low concentrations of SVOCs is analyzed immediately after a sample containing high concentrations of SVOCs. Any samples that have suspected carryover must be reanalyzed.

---

## **4.0 APPARATUS AND MATERIALS**

- 4.1 GC: Hewlett Packard 5890 and/or 6890
- 4.2 Mass Spectrometers (MS): HP5973, HP5972 and/or HP5970
- 4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.
- 4.4 Autosamplers: HP 7673As
- 4.5 Hamilton syringes: 2.00 uL to 10 mL
- 4.6 Volumetric glassware: Grade A or equivalent
- 4.7 Columns: DB-5MS 30m, 0.25mm I.D., 25um film thickness, columns (J&W Scientific) or equivalent.

---

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

---

- 4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.
- 4.9 Data System: The Target software is used for processing data and generating forms.

---

**5.0 REAGENTS**

- 5.1 J.T. Baker Ultra Resi-Analyzed methylene chloride (or equivalent)
- 5.2 Purge and trap grade methanol
- 5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 "Standard Preparation and Documentation".

5.3.1 The expiration date for all standards is one year from date of opening the ampule. If the manufacturer's expiration date is before this one year date, the manufacturer's expiration must be followed. New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

The standards are prepared every 6 months and stored in screw-cap amber bottles with Teflon liners in the BNA standards freezer between uses. Standards prepared from various stock solutions must always use the first expiration date of any of the solutions used for preparation.

5.3.2.1 Calibration Mix – Prepare a standard in methylene chloride containing the compounds listed in Table 3. The final concentration of each compound is 10 ug/mL.

5.3.2.2 Internal Standard Solution – Prepare standard in methylene chloride containing 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12 at a final concentration of 80 ug/mL.

5.3.2.3 DFTPP Solution – Prepare standard in methylene chloride containing DFTPP at a final concentration of 25 ug/mL.

---

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

---

## 6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All semivolatile sample extracts must be analyzed within forty days following the date of extraction.

---

## 7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition and storage: C:\HPCHEM\1\DATA

Tune file: DFTPP.U

Method files: LSIMXX.M (all samples and standards)

Where:

XXX = the calibration number in chronological order

L = instrument ID (R, U, or X)

DFTPP390.M (DFTPP tuning acquisition)

NOTE: All acquisition parameters must be identical for LSIMXXX.M and DFTPP2. M.

Data Files: L\_ \_ \_ \_ .D, where \_ \_ \_ \_ is a number in chronological order from 0001 to 9999 and L is the instrument ID (R, U, or X). This file also contains the Quantitation output file.

Data Files for DFTPP: LD\_ \_ \_ .D, where \_ \_ \_ is a number in chronological order from 001 to 999 and L is the instrument ID (R, U, or X).

7.3 INSTRUMENT SPECIFIC PROCEDURES

It is the policy of the GC/MS group that all data be acquired in the batch mode. The following items must be checked prior to data acquisition in the batch mode:

- Ensure that the proper sequence and tune files are being used.

---

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD  
8270 – Modified for Selected Ion Monitoring (SIM)**

---

- Check the autosampler syringe (Is it clean? Does the plunger move freely? etc.), its alignment and make sure the solvent rinse vial is full. Ensure that the knurled nut holding the top of the syringe plunger is tight.
- Look at the batch to be analyzed and check the following:
  - Make sure that the data files are in numerical order with no duplication and that the method file is the same as that used for ICAL or Continuing Calibration analysis.
  - Bottle numbers match with the numbers on the autosampler tray.

After the batch has been deemed free of errors, start the batch by using the "Position and run" command under the SEQUENCE menu in MSTop.

7.4 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks or samples, the GC/MS system must be shown to meet the mass spectral key ion and ion abundance criteria for decafluorotriphenylphosphine (DFTPP) tabulated below. Pentachlorophenol, benzidine and DDT are also present in this standard.

DFTPP Key Ions and Ion Abundance Criteria	
Mass	Criteria
51	30.0-80.0 percent of mass 198
68	less than 2.0 percent of mass 69
69	present
70	less than 2.0 percent of mass 69
127	40.0 – 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent of mass 198
199	5.0-9.0 percent of mass 198
275	10.0-30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present, but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0-23.0 percent of mass 442

All ion abundances must be normalized to m/z 198, the nominal base peak.

The following are the GC/MS operating conditions for injection of DFTPP.

---

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

---

Initial column temperature hold	140°C for 3 minutes
Column temperature program	140-260°C at 10 degrees/minute
Final column temperature hold	275°C
Injection port temperature	250°C
Transfer line/source temperature	285°C
Injector - splitless, valve time	0.18 minutes
EPC	inlet B
Constant flow	ON
Constant flow pressure	10psi
Constant flow temperature	30°C
Vacuum comp.	ON
Run time	10-12 minutes
Scan start time	5.0 minutes
Sample volume	2.0 uL of 25 ng/uL DFTPP solution
Carrier gas	helium at @ 1.0 mL/minute
Mass range	35 to 500 amu
Number of A/D samples	4
GC Peak threshold	500 counts
Threshold	10 counts

Set up the run on the Enviroquant system using "Edit Sample Log Table". For a more detailed explanation of the Enviroquant software, consult the appropriate manual, Organic Department Manager, or senior chemist within the GC/MS group.

The DFTPP solution must be analyzed once at the beginning of each twelve hour period during which standards and/or samples are analyzed. The 12 hour time period for GC/MS system begins at the moment of injection of the DFTPP analysis. The time period ends after twelve hours has elapsed according to the system clock. The last injection must be accomplished prior to the expiration of 12 hours; conceivably, the run-time of an injection could end after the twelve hours.

When the DFTPP has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The DFTPP run is processed using the current algorithms in the Target software.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, DFTPP must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument DFTPP is not in criteria.

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

**7.5 INSTRUMENT CALIBRATION**

**7.5.1 Initial Calibration for Method 8270-SIM**

Prior to the analysis of samples and required method blanks, and after the instrument DFTPP tuning criteria have been met, the GC/MS system must be calibrated at six different concentrations: 0.20, 0.50, 1.0, 2.0, 3.0 and 5.0 ng/uL. This is done to determine instrument sensitivity and the linearity of GC/MS response for the semivolatile target and surrogate compounds.

Final conc. (ng/uL)	10 ng/uL SIM PAH Stock Added (uL)	10 ng/uL HCB/BEHP/PCP Stock Added (uL)	100 ug/uL 2,4-DBP Stock Added (uL)	MeCl <sub>2</sub> Added (uL)	Final Volume (uL)
0.20	20	100	1.0	879	1000
0.50	50	150	2.0	798	1000
1.00	100	200	4.0	696	1000
2.00	200	250	6.0	544	1000
3.00	300	300	8.0	392	1000
5.00	500	400	10.0	90	1000

If additional compound mixtures are added, the volume of MeCl<sub>2</sub> is adjusted to maintain a final volume of 1000 uL. A 100 uL aliquot of each of the standards above is spiked with 1 uL of internal standards and analyzed.

The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP with the following exceptions:

Column Temperature Program    40°C for 3 minutes to 320°C at 10°/minute

Final Column Temperature hold    320°C

Run Time    35 minutes (time may vary dependent upon column length)

Scan Start Time    6.0 minutes (time may vary dependent upon column length)

Injection volume    1 uL

---

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

---

The conditions are set up in the method file LSIMXXX.M

After analysis of the five calibration points, they must be quantitated and evaluated for adherence to QC criteria. Minimum requirements of ID files are the use of specific quantitation ions and quantitating a specific set of targets and surrogates with a set internal standard. Of particular importance when performing SIM analysis are the ion ratios. These requirements are found in Tables 3 and 5.

### 7.5.2 Initial Calibration Criteria

Relative response factors (RRFs) must be calculated and evaluated for each target compound and surrogate. The RRF is defined as follows:

$$\text{RRF} = \frac{A_x}{A_{IS}} \times \frac{C_{IS}}{C_x}$$

where:  $A_x$  = area of the primary ion for the target compound  
 $A_{IS}$  = area of the primary ion for the corresponding istd  
 $C_{IS}$  = concentration of the istd (ng/uL)  
 $C_x$  = concentration of the target compound

After the calibration points have been quantitated, update the calibration curve points using the Target data processing software to generate the RRF's and %RSD's for all analytes. If information is needed concerning the use of these programs, consult the Organic Department Manager or a senior chemist within the group.

Response factor criteria have been established for the calibration of the semivolatile target and surrogate compounds. These criteria must be met in order for the calibration curve to be considered valid. The percent RSD for each calibration check compound (CCC) must be less than or equal to 30 percent. There are three CCC's: Acenaphthene, Fluoranthene, and Benzo(a)pyrene. There are no criteria for the SPCC compounds.

#### 7.5.2.1 Linearity of Target Analytes

If the RSD of any target analyte is 15% or less, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

---

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

---

If the RSD of any target analyte exceeds 15%, then a calibration option outlined in section 7.0 of method 8000 will need to be employed.

Option 1 is a linear calibration using the average response factor. As per section 7.5.1.2 of method 8000 (Rev. 2, 12/96), "The mean of the RSD values for all analytes in the calibration is less than or equal to 15%. The mean RSD is calculated by summing the RSD values for each analyte and dividing by the total number of analytes."

Option 2 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration. The correlation coefficient for each target analyte and surrogate must be greater than or equal to 0.99.

Option 3 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.99.

If time remains in the clock after meeting the initial calibration acceptance criteria, samples may be analyzed. The calibration must be verified each twelve hour time period (time period starts from the moment of the DFTPP injection) for Method 8270-SIM. The SSTD1.0 in the curve may be used as the continuing calibration standard as long as it meets the continuing calibration acceptance criteria. All sample results must be quantitated using the initial calibration response factors.

### 7.5.3 Continuing Calibration

A check of the calibration curve must be performed once every twelve hours immediately following analysis of the tuning compound DFTPP. This check contains all target compounds and surrogates at a concentration of 1.0 ng/uL.

After quantitation of the 1.0 ng/uL continuing calibration check, response factors must be calculated and compared to the average response factors in the initial calibration. The Target program calculates the calibration check response factors and compares them to the average RFs in the calibration curve by calculating percent differences. The method 8270 CCC's must have

---

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD  
8270 – Modified for Selected Ion Monitoring (SIM)**

---

a % difference of +/- 20%D in order to be considered in criteria. These conditions must be met before method blank and/or sample analysis can begin.

If the continuing calibration check does not meet criteria, corrective action must be taken. Depending on the situation, corrective action may be as follows:

- Re-analyze the 1.0 ng/uL continuing calibration check.
- Change the septum; clean the injection port; install a clean, silanized quartz liner; cut off a small portion (1" to 3") of the front end of the capillary column. This is usually performed when chromatography is poor.
- Analyze a new initial calibration curve.

The last option, the generation of a new initial calibration curve, is usually chosen when percent difference are >30%. In these instances, there is little or no chance of a continuing calibration reanalysis meeting criteria. If there is any doubt concerning which corrective action to undertake, consult the Organic Department Manager or a senior chemist within the group.

If the continuing calibration does meet the criteria specified above then analysis may proceed using initial calibration response factors.

## 7.6 SAMPLE ANALYSIS

Sample extracts may be analyzed only after the GC/MS system has met tuning criteria, initial calibration and continuing calibration requirements. Ensure that the same instrument conditions are being used for tuning, calibration and sample analysis by reviewing the GC parameters using the "Edit entire method" option under the Method menu in MSTOP. Note that you can not edit a method if the instrument is running.

Extracts are stored in the refrigerator in the organics extraction laboratory at 4°C ±2°C. Remove them from the refrigerator and place them in the GC/MS laboratory semivolatile hood when ready for analysis.

Prepare a 1.8 mL clear glass vial (crimp top) with a disposable insert (350 uL). Add 100 uL of sample extract and 1.0 uL of the 80 ng/uL IS stock to the vial and then cap.

---

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD  
8270 – Modified for Selected Ion Monitoring (SIM)**

---

This gives a 0.8 ng/uL final concentration for the internal standard compounds. The samples are topped with Teflon lined crimp top caps.

## 7.7 FINAL DATA PACKAGE

### 7.7.1 Initial Data Review (IDR)

The initial data review is accomplished by the analyst who ran the samples and is a review of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed:

- Surrogate Recoveries
- Internal Standard Area Stability
- Method Blank Acceptance
- Chromatography
- Target Compound Detection/Quantitation/Review for false positives
- Laboratory Control Sample Recoveries
- Matrix Spike/Matrix Spike Duplicate Recoveries

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed. During this review, the analyst checks the integration of each individual peak. The hardcopy has false positives crossed out so they can be reviewed for appropriateness by the Organic Department Manager.

### 7.7.2 Chromatography

The chromatography should be examined for the presence or absence of any ghost peaks and can also be used as an indication of whether or not matrix interferences might be affecting surrogate recoveries and/or istd area recoveries. Whether or not the chromatography is acceptable is a judgment

---

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD  
8270 – Modified for Selected Ion Monitoring (SIM)**

---

call on the part of the analyst and should be used in conjunction with other monitored QC (e.g. surrogate recoveries) to determine the necessity of reanalyzing.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an "m" qualifier will automatically be printed on the quantitation report summary.

This manual integration package must then be submitted to the Department Manager or his/her designee, who will review each manual integration.

#### 7.7.3 Target Compound Detection/Quantitation

The semivolatile ID files have been set up to err on the side of false positives; that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits. It is the responsibility of the GC/MS analyst to use his/her technical judgment to determine if the identification of a target compound is correct or not.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the concentration of the largest target compound hit in the upper half of the initial calibration range. LCS and MS/MSD samples need not be diluted to get spiked analytes within the calibration range.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity > 10% must be present in the sample spectrum.
- The relative intensities of primary and secondary ions must agree within  $\pm 20\%$  between the standard and sample spectra.

---

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

---

- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgement of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial review should be accomplished at the beginning of a work shift for the previous set of analyses.

#### 7.7.4 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC label requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A complete review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

#### 7.8 INJECTION PORT LINER CLEANING AND SILANIZING PROCEDURE

- Remove the rubber o-ring from the liner and place the liner in a large Erlenmeyer flask.
- In the hood, pour nitric acid into the flask until the liner is covered. Place the flask on a hotplate and boil for 2-3 hours.
- Let cool; drain nitric acid and thoroughly flush the liner with water.
- Bake briefly in the muffle oven until liner is dry and cool to room temperature.
- Place the liner in a beaker, fill with Sylon and let it soak for at least two hours.

---

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

---

- Take out the liner and rinse it thoroughly with toluene.
  - Rinse the liner thoroughly with purge and trap grade methanol.
  - Bake the liner in the muffle oven for a minimum of three hours.
- 

## **8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA**

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

### **8.1 Method Blank Criteria**

A method blank is defined as a volume of a clean reference material (deionized distilled water for water samples, baked organic-free sand for soil/sediment matrices) that is carried through the entire analytical procedure. One method blank must be extracted with each group of samples of a similar matrix and must be analyzed on the GC/MS system that was used to analyze the samples.

---

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

---

An acceptable method blank must contain less than or equal to the PQL of any target compound.

If the method blank exceeds these contamination levels, the analytical system is considered out of control and corrective action must be taken before sample analysis.

Reanalysis of the blank is the first step of the corrective action; if that does not solve the problem, a Katahdin Corrective Action Report (CAR) will be initiated. Corrective action will be specified after consultation including the Department Manager, Operations Manager, and QA Officer.

## 8.2 Surrogate Recoveries

The five surrogates (Pentafluorophenol, 2-Methylnaphthalene-d10, 2,4-Dibromophenol, Fluorene-d10 and Pyrene-d10) must meet the current statistically derived acceptance limits. If statistical limits have not been established then the surrogate recovery must meet the nominal limits of 30-150%.

If specifications are not met, the sample (or blank) should be reanalyzed. If specifications are met in the reanalysis, this reanalysis should only be submitted. If surrogate specifications are not met in the sample or method blank reanalysis, a Corrective Action Report (CAR) should be initiated. Corrective action will be specified after consultation including the Department Manager and Operations Manager.

For further information regarding the acceptance of surrogate recoveries, consult the Organic Department Manager.

## 8.3 Internal Standard Responses

Internal standard responses and retention times (RT) in all samples and blanks must be evaluated as part of the technical data review. The method files have been set up to only detect compounds that fall within a set RT window. For Method 8270-SIM analysis, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the daily continuing calibration standard, reanalysis must occur. If the reanalysis meets criteria, only the in-criteria run should be reported. If the reanalysis is still out of criteria, both analyses should be included in the sample package set.

MS/MSD samples that do not meet the EICP area criteria above do not have to be reanalyzed.

---

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD  
8270 – Modified for Selected Ion Monitoring (SIM)**

---

8.4 Laboratory Control Sample (LCS)

An LCS must be performed for each group of samples of a similar matrix, for the following, whichever is more frequent:

- Every 20 samples of a similar matrix or similar concentration, or
- Every batch of samples extracted.

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the section supervisor, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts", current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 30-150% may be used for some projects or states (i.e. South Carolina).

The LCS recoveries for all analytes are evaluated. As a general rule, all, but ten percent of the compounds of interest, must fall within the established statistical limits. The common laboratory contaminants, phthalate esters, are not included in the ten percent. If less than ten percent of the recoveries fail the statistical limits, no corrective action is needed. If greater than ten percent of the recoveries fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgements while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

Please note that for compounds with only nominal limits (i.e. insufficient data points were available to generate statistical limits), no corrective action is required for out-of-criteria recoveries until enough data points are established to generate statistical limits.

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

---

**8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria**

Matrix Spike and Matrix Spike Duplicates must be extracted and analyzed for each group of up to 20 samples of a similar matrix or similar concentration. In the event insufficient sample volume is available an LCS/LCS Duplicate is extracted and analyzed in place of the MS/MSD.

Statistical limits are compiled annually for MS/MSD recoveries for a short list of the spiked compounds (Acenaphthene, Pentachlorophenol and Pyrene). Nominal limits of 30-130% are used for all other compounds. Generally, corrective action is only taken for the short list of the spiked compounds. The specific corrective actions will rely on analyst experience to make sound scientific judgements while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

A Corrective Action Report (CAR) must be filled out and filed if any criteria for percent recovery or relative percent difference are not met to document any decisions with reporting data.

---

**9.0 METHOD PERFORMANCE**

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the current revision of Method 8270 for other method performance parameters and requirements.

---

**10.0 APPLICABLE DOCUMENTS/REFERENCES**

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update III, December 1996, Method 8270C.

"USEPA Contract Laboratory Program Statement of Work for Organics Analysis," Rev. 02/88.

Code of Federal Regulations (40 CFR), Part 136, Appendix A, Rev. June, 1998.

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD  
8270 – Modified for Selected Ion Monitoring (SIM)**

---

LIST OF TABLES AND FIGURES

Table 1	QC Requirements
Table 2	Summary of Method Modifications
Table 3	Analyte Quantitation and Internal Standards
Table 4	Procedure Condensation
Table 5	SVOA Compounds and Characteristic Ions
Figure 1	Example of Runlog Logbook Page
Figure 2	Example of GC/MS Standards Receipt Logbook Entry
Figure 3	Example of SVOA Standards Preparation Logbook Entry

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

TABLE 1  
QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.4	Retune instrument, and verify
Six-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD $\leq$ 30 for RFs of the CCCs; Average %RSD < 15% for all compounds.	Repeat calibration if criterion is not met
Continuing calibration verification	Once per each 12 hours, prior to sample analysis	CCCs $\leq$ 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
ISs	Immediately after or during data acquisition of calibration check standard	Retention time $\pm$ 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Demonstration of ability to generate acceptable accuracy and precision	Once per analyst initially and annually thereafter	All recoveries within method QC acceptance limits.	Recalculate results; locate and fix problem; reextract/reanalyze P&A study for those analytes that did not meet criteria
Method blank	One per prep batch	No analytes detected > PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report samples that are <PQL or > 10X the blank result. Reprep a blank and the remaining samples.

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

TABLE 1 (cont'd)

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
LCS for all analytes	One LCS per prep batch	Statistically derived from lab data or nominal limits depending on the project. See also section 8.4 of this SOP.	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS rec. is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Surrogate spike	Every sample, control, standard, and method blank	Statistically derived limits.	(1) Check chromatogram for interference; if found, flag data (2) If not found, check instrument performance; if problem is found, correct and reanalyze (3) If still out reextract and analyze sample (4) If reanalysis is out, flag data
MS/MSD	One MS/MSD per every 20 samples	Statistically derived from lab data or nominal limits depending on the project. Nominal limits are used as default limits.	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL study	Once per year	Ideally, PQL = at least 3xMDL	Repeat MDL study

---

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

---

TABLE 2  
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-213-03	METHOD 8270, current revision
Apparatus/Materials	none	
Reagents	none	
Sample preservation/ handling	none	
Procedures	none	
QC - Spikes	none	
QC - LCS	none	
QC - Accuracy/Precision	none	
QC - MDL	none	

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

TABLE 3

ANALYTE QUANTITATION AND INTERNAL STANDARDS

<b>Internal Standard: 1,4-dichlorobenzene-d4</b>	Phenanthrene
Target and Surrogates:	Hexachlorobenzene (special)
Pentafluorophenol (surrogate)	Anthracene
Hexachloroethane (special)	Fluoranthene
<b>Internal Standard: Naphthalene-d8</b>	Carbazole (special)
Target and Surrogates:	Di-n-butylphthalate (special)
Naphthalene	<b>Internal Standard: Chrysene-d12</b>
1-Methylnaphthalene (dredge)	Target and Surrogates:
2-Methylnaphthalene	Pyrene
2-Methylnaphthalene-D10 (surrogate)	Benzo(a)Anthracene
<b>Internal Standard: Acenaphthene-d10</b>	Chrysene
Target and Surrogates:	Bis-(2-ethylhexyl)phthalate (special)
Biphenyl (dredge)	Pyrene-d10 (surrogate)
2,6 Dimethylnaphthalene (dredge)	<b>Internal Standard: Perylene-d12</b>
Acenaphthylene	Target and Surrogates:
Acenaphthene	Perylene (dredge)
Fluorene	Benzo(b)fluoranthene
2-Fluorene-d10 (surrogate)	Benzo(k)fluoranthene
2,4-Dibromophenol (surrogate)	Benzo(e)pyrene (dredge)
2-Chloronaphthalene (special)	Benzo(a)pyrene
<b>Internal Standard: Phenanthrene-d10</b>	Indeno(1,2,3-cd)pyrene
Target and Surrogates:	Dibenz(a,h)anthracene
Pentachlorophenol (special)	Benzo(ghi)perylene
1-Methylphenanthrene (dredge)	

---

TITLE: **ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD  
8270 – Modified for Selected Ion Monitoring (SIM)**

---

TABLE 4

PROCEDURE CONDENSATION

Clock

12 hours from injection of 50ng DFTPP.

Calibration Curve Criteria

<30% RSD for CCCS

<15% RSD average for all analytes in calibration standard

Continuing Calibration Check Criteria

<20% D for CCC compounds

Additional QC

LCS every extraction batch

MS/MSD every 20 samples

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**

TABLE 5

SVOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	PRIMARY ION	SECONDARY IONS
Naphthalene	128	129,127
2-Methylnaphthalene	142	115
Acenaphthylene	152	151,153
Acenaphthene	153	152,154
Fluorene	166	165,167
Phenanthrene	178	179,176
Anthracene	178	179,176
Fluoranthene	202	200,203
Pyrene	202	200,203
Benzo(a)anthracene	228	226
Chrysene	228	226
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,250
Indeno(1,2,3-cd)pyrene	276	277
Dibenz(ah)anthracene	278	279
Benzo(ghi)perylene	276	277
1-Methyl naphthalene (dredge)	142	115
Biphenyl (dredge)	154	76
2,6-Dimethyl Naphthalene (dredge)	156	141
1-Methyl phenanthrene (dredge)	192	191,193
Benzo (e) pyrene (dredge)	252	125
Perylene (dredge)	252	125
Carbazole	167	166,139
Pentachlorophenol	266	264,268
Hexachlorobenzene	284	282, 286
Bis(2-ethylhexyl)phthalate	149	167
2-Chloronaphthalene	162	127, 164
Di-n-butylphthalate	149	104 150
Hexachloroethane	117	201, 199
Pentafluorophenol (surrogate)	184	136
2-methylnaphthalene-d10 (surrogate)	152	125
Fluorene-d10 (surrogate)	176	175, 177
Pyrene-d10 (surrogate)	212	210, 213
2,4-Dibromophenol (surrogate)	252	63, 143
1,4-Dichlorobenzene-d4 (istd.)	152	115,150
Naphthalene-d8 (istd.)	136	134,137
Acenaphthene-d10 (istd.)	164	162,160
Phenanthrene-d10 (istd.)	188	189
Chrysene-d12 (istd.)	240	241,236
Perylene-d12 (istd.)	264	260

**TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD  
8270 – Modified for Selected Ion Monitoring (SIM)**

---

Primary ions must not be changed except in unusual instances where interference occurs with a co-eluting non-target analyte. In this case, a secondary ion may be used for quantitation with the following rules:

- (1) The corresponding standard(s) (initial calibration curve and continuing calibration standard) must be re-quantitated with the secondary ion.
- (2) Approval must be obtained from the Organic Department Manager or the laboratory Operations Manager.

The quantitation ion must then be changed back to the one specified in the table above after quantitation of the samples(s).

Secondary ions are recommended only and may be changed depending upon instrument conditions (sensitivity, etc.). However, it is Katahdin policy that a minimum of 2 ions (primary and one secondary) be used for all GC/MS analyses.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD  
8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 1

EXAMPLE OF RUNLOG LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES  
GC/MS SVOA INJ LOG INSTRUMENT: 5970-X

DATE OF DFTPP INJECTION: 03/009

JOB	SAMPLE	DATE	DF	ALS#	METHOD	GC/MS	CHEMIST	COMMENTS
	SD wa DFTPP	X0600	1	1	DFTPP90	20	JK	OK
	SSTV060X030	X9437		2	X625A022	10		✓
	150	36		3				✓
	100	37		4				✓
	30	40		5				✓
	OK	41		6				✓
	3510 MIX	42		7				OK
		43		8				
		44		9				
		45		10				
		46		11				
		47		12				
		48		13				
		49		14				
		50		15				
	PLANE	51		16				
		52		17				

QAMSS02

0000034

STANDARD	CODE
DFTPP	30856
CAL. STD.	S089-30 S0841-49
IS MIX	A600934

REVIEWED AND APPROVED BY: \_\_\_\_\_  
DATE: \_\_\_\_\_

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD  
8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK ENTRY

KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GCMS LABORATORY  
REVIEWED BY/DATE:

<i>Am 0946</i>	 125 Alford St. • New Haven, CT 06513 • USA Tel. 203-788-5200 • www.accustandard.com	FOR LABORATORY USE ONLY WARNING: This product contains a chemical known to the State of California to cause cancer.
APP-9-176-D-20X Pentachlorophenol 2.0 mg/mL in CH <sub>2</sub> Cl <sub>2</sub> Lot: B3D10100 Exp. Jan 10, 2013	1 mL	POISON
		STORAGE: Ambient <i>Real 3/16/06 JK</i>
<i>Am 0947</i>	 125 Alford St. • New Haven, CT 06513 • USA Tel. 203-788-5200 • www.accustandard.com	FOR LABORATORY USE ONLY
APP-9-090-50X 4,6-Dinitro-o-cresol 5.0 mg/mL in MeOH Lot: B1100296 Exp. Aug 18, 2012	1 mL	FLAMMABLE
		STORAGE: Ambient
<i>Am 0948</i>	 125 Alford St. • New Haven, CT 06513 • USA Tel. 203-788-5200 • www.accustandard.com	FOR LABORATORY USE ONLY
APP-9-145-50X p-Nitrophenol 5.0 mg/mL in MeOH Lot: B5050205 Exp. May 18, 2015	1 mL	POISON
		STORAGE: Ambient

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD  
8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 3

EXAMPLE OF SVOA STANDARDS PREPARATION LOGBOOK ENTRY

GC/MS SVOA STANDARD PREP LOGBOOK

ID	Description	Date	Time	Analyst	Compound	Volume	Concentration	Volume	Concentration	
50863	8270 Stock (w/o MeOH)	3-15-06	7-7-06	JK	AMP884	8270 Methyl	300	2-22-07	4.2ml	150 ug/ml
					AMP887	+	350	3-15-07		
					AMP891	APP 1X #2	600	3-2-07		
					AMP890	+	100	3-9-07		
					AMP870	+	200	7-7-06		
					AMP899	Organophos post	300	8-14-06		
					AMP873	Benzoic acid		3-9-07		
					AMP897	Hexachlorophene		2-22-07		
					AMP896	Benzoilone		3-9-07		
					AMP836	3,3'-Dichlorobenzene		3-14-07		
50861	DEA		3-13-07							
50860	MeCl <sub>2</sub>		-							
50864	8270 Level 1	3-15-06	7-7-06	JK	50863	8270 Stock	70	7-7-06	1.05ml	10 ug/ml
					50860	MeCl <sub>2</sub>	980			
50865	8270 Level 2	3-15-06	7-7-06	JK	50863	8270 Stock	150	7-7-06	0.90ml	25 ug/ml
					50860	MeCl <sub>2</sub>	750			
50866	8270 Level 3	3-15-06	7-7-06	JK	50863	8270 Stock	600	7-7-06	1.8ml	50 ug/ml
					50860	MeCl <sub>2</sub>	1200			
50867	8270 Level 4	3-15-06	7-7-06	JK	50864	8270 Stock	700	7-7-06	1.05ml	100 ug/ml
					50860	MeCl <sub>2</sub>	350			

Reviewed by/Date:

0000057

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

Prepared By: Peter Lemay Date: 7/96

Approved By:

Group Supervisor: Peter Lemay Date: 1/15/01

Operations Manager: John C. Benton Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1.22.01

General Manager: Dennis F. Kufan Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
02 8081A	Format changes, added pollution prevention, minor changes to sections 7, 8 and Table 1.	DN	1.22.01	1/22/01
03 8081A	Changes to comply with South Carolina requirements - added linear calibration option, retention time window criteria & other minor changes to surrogate criteria.	DN	5.21.01	5.21.01
04 8081A	Changed to practice of reporting higher value. Other minor changes to Table 1 + 2, section 7.5.3 and section 7.43.	DN	5.21.02	5.21.02
05 8081A	Added definitions and information for the new data processing system. Replaced several figures with updated ones.	MRC	05.04.04	05.04.04
06 8081A	added alternative CV Conc. Changed data checklist minor changes throughout added wording to section 8	LAD	3 03/05	3 03/05



---

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

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

---

I acknowledge receipt of copy \_\_\_ of document **SOP CA-302-07**, titled **ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_ of document **SOP CA-302-07**, titled **ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

## 1.0 SCOPE AND APPLICATION

This SOP describes all aspects of the analysis of extracts of solid and aqueous samples for Pesticides by EPA Method 8081, current revision, as performed by Katahdin Analytical Services, Inc. including sample analysis, data review, standard preparation and instrument calibration.

It is applicable to the following compounds: aldrin, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, chlordane, 4,4'-DDT, 4,4'-DDE, 4,4'-DDD, dieldrin, endosulfan I, endosulfan II, endosulfan sulfate, endrin, endrin aldehyde, heptachlor, heptachlor epoxide, toxaphene, endrin ketone, and methoxychlor. Extracts are analyzed by Gas Chromatography-Electron Capture Detector (GC-ECD).

### 1.1 Definitions

**ANALYTICAL BATCH:** 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

**METHOD BLANK (laboratory reagent blank):** An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; however a universal blank matrix does not exist for solid samples, and therefore, no matrix is used. The blank is taken through the appropriate steps of the process.

**CALIBRATION CHECK:** Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution, which is different from the stock used to prepare standards.

**CALIBRATION STANDARD (WORKING STANDARD):** A solution prepared from the stock standard solution, which is used to calibrate the instrument response with respect to analyte concentration.

**LABORATORY CONTROL SAMPLE (LCS):** A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

---

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

**MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD):** Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

**STANDARD CURVE (CALIBRATION CURVE):** A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

**STOCK STANDARD SOLUTION:** A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

**SURROGATES:** Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

**KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS) :** A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

**PE NELSON TURBOCHROM:** A data acquisition system that is used to collect chromatographic data. The system can also be used to archive raw data files.

**TARGET:** A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

**TARGET DB:** An oracle database used to store and organize all Target data files.

**QUICKFORMS:** A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

---

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

## 1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of pesticides by method 8081, current revision. Each analyst must demonstrate the ability to generate acceptable results with this method.

It is the responsibility of all Katahdin technical personnel involved in analysis by method 8081, current revision, to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

## 1.3 Health and Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

## 1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

---

## 2.0 SUMMARY OF METHOD

- 2.1 Method 8081 provides gas chromatographic conditions for the detection of ppb concentrations of certain organochlorine pesticides. Prior to the use of this method, appropriate sample extraction techniques must be used. Both neat and diluted organic liquids (Method 3580, waste dilution) may be analyzed by direct injection. A 2-5 ul aliquot of sample is injected into a gas chromatograph (GC) using the direct injection technique, and compounds in the GC effluent are detected by an electron capture detector (ECD).
  - 2.2 The sensitivity of Method 8081 usually depends on the concentration of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, Method 8081 may also be performed on samples that have undergone cleanup. Method 3660, Sulfur Cleanup, by itself or in conjunction with Method 3620, Florisil Column Cleanup, may also be used to eliminate interferences in the analysis.
- 

## 3.0 INTERFERENCES

- 3.1 Interferences by phthalate esters can pose a problem in pesticide determinations when using the electron capture detector. Common flexible plastics contain various amounts of phthalates. Care has to be taken to avoid using any plastic materials during the extraction process. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.
- 

## 4.0 APPARATUS AND MATERIALS

- 4.1 Gas chromatograph

---

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

- 4.1.1 GC Hewlett Packard 6890 or 5890 series I or II connected to the Turbochrom or Enviroquant data system, or equivalent.
- 4.1.2 Columns: Instruments are configured with a pre-column originating from the injection port which is connected to deactivated glass Y splitter that connects two different columns to two detectors. The most commonly used columns are: RTX-35 30M x 0.53 mm ID, RTX-5 30M x 0.53 MM ID, or RTX-1701 30M x 0.53 mm ID. Equivalent columns can be used.
- 4.1.3 Detectors: Electron capture detectors (ECD).
- 4.2 Volumetric flasks, class A: sizes as appropriate with the ground-glass stoppers.
- 4.3 Syringes: various sizes for preparing standards and injecting samples on the instrument.
- 4.4 Vials: various sizes and types including crimp tops.
- 4.5 Balances: Analytical, 0.0001 g
- 4.6 Refrigerator for storage of extracts and standards.

---

## 5.0 REAGENTS

- 5.1 Solvents
  - 5.1.1 Hexane: pesticide quality or equivalent for diluting samples and standards.
- 5.2 Standards
  - 5.2.1 Stock standard solutions: Solutions purchased from suppliers like Restek or other acceptable retailers. Expiration dates are one year from date of opening vial or sooner if manufacturers date is less. Upon receipt, all standards are logged into the appropriate logbook with the date of receipt, expiration date, source, lot number, solvent and concentration of compounds.
  - 5.2.2 Calibration standards: Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in a separate logbook.

---

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

Pesticide Working standards: Prepared by diluting the stock mix of 2000 ug/ml that contains all single component pesticides into hexane to give final concentrations of: 0.005, 0.01, 0.025, 0.05, 0.10, and 0.25 ug/ml. The mix, referred to as INDAB, also contains two surrogates: Tetrachloro-m-xylene and Decachlorobiphenyl, which are at the same concentrations as the pesticides.

Multicomponent Pesticide Working standards: Toxaphene is prepared by diluting the Toxaphene stock solution to a concentration of 1.0 ug/ml. Technical chlordane is prepared similarly except to a concentration of 0.50 ug/ml.

Evaluation Mix: Prepared by diluting the stock solution to a concentration of 0.20 ug/mL.

---

## 6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Extracts must be stored under refrigeration and analyzed within 40 days of extraction.

---

## 7.0 PROCEDURES

EXTRACTION - Refer to the appropriate SOP for the correct extraction procedure. In general, water samples are extracted using methods 3510 or 3520 while solid samples use methods 3540, 3545 or 3550.

### 7.1 INSTRUMENT CONDITIONS

Refer to the instrument logbook for the current column and conditions.

Typical conditions are: Makeup flow: 60 ml/min Ar/Methane  
Column flow: 3.75 ml/min  
Injector Temp: 200  
Detector Temp: 300  
Oven Ramp: 160(0) - 5/min - 260(10)  
Run time: 24 min  
Injection size: 2 uL

---

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

## 7.2 CALIBRATION

7.2.1 The GC system is calibrated using the external standard calibration procedure. A six-point calibration standard mix of the INDAB mix listed in Reagents Section 5.2.2 is prepared along with a single point standard of Toxaphene and Technical Chlordane.

If the sample contains Toxaphene, a six-point calibration curve is analyzed. If the sample contains Chlordane and the analysis request is for Technical Chlordane, a six-point calibration curve is analyzed. If the analytical request is for the two components alpha-Chlordane and gamma-Chlordane, these two compounds are quantitated from the INDAB mix.

Toxaphene is calibrated using the 5 to 10 major peaks of the standard. The heights of the 5 to 10 peaks are averaged. A calibration curve is prepared in Target using the average of the peak heights of the 5 to 10 peaks against the concentration of the standard.

Technical Chlordane is calibrated using the 3 to 5 major peaks of the standard. The heights of the 3 to 5 peaks are averaged. A calibration curve is prepared in Target using the average of the peak heights of the 3 to 5 peaks against the concentration of the standard.

Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. The Target system will calculate a peak height for each compound. A calibration curve can be prepared in Target using the peak height against the concentration of the standard. A non-linear calibration applying a second order polynomial (quadratic fit) equation is used to prepare the curve. In order to be used for quantitative purposes, the correlation coefficient must be greater than or equal to 0.990. The quadratic equation is:

$$y = ax^2 + bx + c$$

where: y = Instrument response  
b = Slope of the line  
x = Concentration of the calibration standard  
c = The intercept

Please note that a non-linear calibration model may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear

---

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

calibration for compliance work originating in their state. In these cases, a linear calibration model must be used. The linear equation is

$$y = bx + c$$

where: y = Instrument response  
b = Slope of the line  
x = Concentration of the calibration standard  
c = The intercept

The calibration curve is calibrated the same way as the second order polynomial equation except that a five-point calibration standard mix is used.

7.2.2 The working calibration curve must be verified on each 12-hour shift that samples are to be analyzed by injecting the mid-point calibration standard.

### 7.3 RETENTION TIME WINDOWS

7.3.1 Three injections of all single component standard mixtures and multiresponsive products throughout the course of a 72-hour period.

7.3.2 The standard deviation of the three retention times is calculated for each single component standard. For multiresponsive products, a major peak from the envelope is chosen and a standard deviation is calculated using the three retention times for that peak.

7.3.3 Plus or minus three times the standard deviation of the retention times for each standard is used to define the retention time window; however, the experience of the analyst should weight heavily in the interpretation of chromatograms. For multiresponsive analytes, the analyst should use the retention time window, but should primarily rely on pattern recognition.

7.3.4 Retention time windows are calculated for each standard on each GC column and whenever a new GC column is installed. The data is kept on file in the laboratory.

7.3.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being too narrow. The windows are:  $\pm 0.05$  for Heptachlor, Aldrin and all BHC compounds,  $\pm 0.07$  for all other target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive by carefully evaluating the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal

---

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of  $\pm$  0.03 minutes must be used if the established retention time window is less than 0.03 minutes.

#### 7.4 GAS CHROMATOGRAPHIC ANALYSIS

- 7.4.1 Before calibration is performed, and at the beginning of each 12 hour shift, the system is evaluated for analyte degradation by the analysis of a standard mix containing only endrin and 4,4'-DDT, often called an evaluation mix (EVAL):

<u>COMPOUND</u>	<u>CONCENTRATION</u>
Endrin	0.20 ng/uL
DDT	0.20 ng/uL

The % breakdown of DDT and the % breakdown of Endrin is calculated using the following formulas (PH = Peak Height):

$$\% \text{ Breakdown DDT} = \frac{(\text{PH [DDD]} + \text{PH [DDE]})}{(\text{PH [DDD]} + \text{PH [DDE]} + \text{PH [DDT]})} * 100$$

$$\% \text{ Breakdown Endrin} = \frac{(\text{PH [Endrin Aldehyde]} + \text{PH [Endrin Ketone]})}{(\text{PH of [Endrin Aldehyde]} + \text{PH of [Endrin Ketone]} + \text{PH of [Endrin]})} * 100$$

The breakdown of either DDT or Endrin in the evaluation mix cannot exceed 15%. If there is breakdown of either compound exceeding 15% before starting a calibration, instrument maintenance must be performed. A calibration can not be run until the evaluation mix meets the acceptance criteria. If the exceeding breakdown occurs during the analysis sequence, then any samples analyzed after a failing evaluation mix must be reanalyzed. Reanalysis can not resume until after an acceptable evaluation mix.

- 7.4.2 Gently shake sample extracts before vialing for analysis.
- 7.4.3 All instrument injections are performed using the direct injection technique with an autosampler set for 2-5 uL injection volumes.
- 7.4.4 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration as listed in section 7.2 followed by sample extracts interspersed with mid-concentration calibration standards. Before any samples are analyzed the instrument must be calibrated by

---

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

analyzing a six-point calibration or a 0.05ppm concentration standard (calibration verification standard). If a CV is run, the calculated concentration must not exceed a difference of  $\pm 15\%$ . Each sample analysis must be bracketed with an acceptable initial calibration and closing CV or an opening CV and a closing CV for each 12-hour shift. The closing CV standard is at 0.25ppm. The calibration standard must also be injected at intervals of not less than once every twenty samples (after every 10 samples is recommended) and at the end of the analysis sequence. If the CV fails, the instrument is checked for any obvious problems and maintenance is performed if deemed necessary. All samples that were injected after the last standard that last met the QC criteria must be evaluated to prevent mis-quantitations and possible false negative results, and re-injection of the sample extracts may be required. However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e.  $>15\%$ , and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed, as the CV standard has demonstrated that the analyte would have been detected were it present. In contrast, if an analyte above the QC limits was detected in a sample extract, then re-injection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 15% below the initial calibration response, then re-injection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present.

- 7.4.5 The center of the retention time window for each analyte and surrogate is established by using the absolute retention time for each analyte and surrogate from the initial calibration.
- 7.4.6 The identification of Pesticides is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes. An analyte is tentatively identified when a peak from a sample falls within the absolute retention time window. Each tentative identification must be confirmed using a second GC column of dissimilar stationary phase or using another technique such as GC/MS. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.
- 7.4.7 If the response for an analyte exceeds the calibration range of the system, the sample must be diluted and reanalyzed.

---

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

- 7.4.8 If peak detection and identification are prevented due to interferences, the hexane extract may need to undergo a cleanup. The extract may be subjected to a florisil cleanup (method 3620) and/or a sulfur cleanup (method 3660). Whenever a sample receives a cleanup, the associated QC must also be subjected to the same cleanup(s) and reanalyzed.
- 7.4.9 When a GC system is determined to be out of control because either a CV can not pass or a six point calibration does not meet the coefficient of determination criteria, instrument maintenance is likely necessary. Routine instrument maintenance may involve changing the septum, replacing the liner, clipping the pre-column, replacing the Y connector, or replacing the column. This information is recorded in the instrument run log (Figure 1). When an instrument requires more severe maintenance like replacing the ECD or an electronic board, this information is written in the instrument maintenance logbook.
- 7.4.10 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration after the file is processed through the appropriate calibrated method.
- 7.4.11 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

7.4.11.1 Water: Concentration (ug/L) =  $(C) (Vt)/(Vs)$

7.4.11.2 Soil/Sediment: Concentration (mg/kg) =  $(C) (Vt)/(Ws) (D)$

where, C = concentration calculated by Turbochrom in ug/ml  
Vt = Volume of total extract including any instrument dilutions  
Vs = Volume of sample extracted  
Ws = Weight of sample extracted  
D = Decimal total solids

## 7.5 Data Review

### 7.5.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed samples. This

---

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- ◆ QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.
- ◆ Surrogate recovery
- ◆ Chromatography: cleanups, manual integration.
- ◆ Target compound detection: quantitation, confirmation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next workday. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.7.

#### 7.5.2 Surrogate recovery

All recoveries must meet the most recently laboratory established acceptance limits, which are listed on the Laboratory Surrogate Acceptance Limit sheet.

The sample is evaluated for recoveries of the two surrogates. The recoveries of both surrogates are evaluated on both the primary and secondary column. The higher recovery from both columns is reported on the analytical report for both surrogates. The sample chromatogram is reviewed for any interferences before determining whether to accept a sample based on the surrogate recoveries. If the surrogate recovery is affected by matrix interference, the sample result may be accepted with narration. If the recovery of one surrogate is outside of the laboratory established acceptance limit on one or both columns, and the second is acceptable, the data is narrated. If the recoveries for both surrogates are not acceptable because the recoveries are high and the sample does not contain any analytes above the PQL, the data is narrated. If the recoveries for both surrogates are low and there is no apparent matrix effect, the sample is reextracted.

For method blanks, if the recoveries of both surrogates are low or high, and the blank does not contain any target analytes above the PQL, and the recoveries of both surrogates in the sample(s) are acceptable, the data is narrated. If the recoveries in the blank are low and it does not contain any target analytes above the PQL, and the recoveries in the samples are acceptable but the sample contains one or more target analytes above the PQL, the sample may be reextracted.

---

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

For laboratory control samples (LCS), if the only discrepancy in the extraction batch is with the LCS, and the analyte spike recoveries are acceptable, the data is narrated. If the recoveries of both the surrogates and the analyte spikes are low, the samples may need to be reextracted. For specific procedures on how to manually integrate, refer to Katahdin SOP QA-811, Manual Integration, current revision.

#### 7.5.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries. If the chromatogram indicates interferences, then a cleanup may be needed. See section 7.4.7.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an "m" qualifier will automatically be printed on the quantitation report summary.

#### 7.5.4 Target Compound Detection

The chromatogram from channel A is evaluated with that from channel B. If a target analyte is present on both channels and the concentration is within the calibration range, and the quantitation from both chromatograms agrees within  $\pm 50\%$ , the analyte is considered to be present in the sample. The higher of the two concentrations is reported.

In order to avoid reporting false positives, identified peaks on a chromatogram may need to be undetected electronically in Target. The possible scenarios are: If an analyte is present on one column but its concentration is below the PQL, if an analyte is present on one column but does not confirm on the other channel, if an analyte is present on both columns but the concentrations differ by more than 50%, or if an analyte is present but its retention time is  $\pm 0.04$  minutes or more than the retention time of the analyte in the preceding CV.

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

## 7.6 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A completed review checklist (Figure 2) is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

---

## 8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

---

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

- 8.1 For each analytical batch (up to 20 samples), a method blank, laboratory control sample (LCS), matrix spike and matrix spike duplicate are analyzed. They are carried through all stages of the sample preparation and analysis steps.
- 8.2 Spike concentrations: The LCS and the MS/MSD are spiked with the twenty single component pesticides at the same concentration. The spike concentrations are:

	WATER ug/L	SOILS ug/Kg
Pesticides	0.50	16.7

The surrogate spike concentrations in the final extract are:

	WATER ug/ml	SOILS ug/ml
Tetrachloro-m-xylene(TCX)	0.10	0.10
DCB	0.10	0.10

- 8.3 LCS and MS/MSD acceptance criteria and Corrective Action: All QC samples are calculated for percent recovery of the spiked analyte(s). The recoveries are compared to laboratory established acceptance limits. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision.

Please note that established acceptance limits that are wider than 70-130% may not be allowable for certain states, federal programs, or clients. For South Carolina, the acceptance limits for the spiked analytes will be 70-130% or narrower.

If any spike compound in the laboratory control sample falls outside of the established recovery acceptance limit window, the QC sample is considered to be out of control and any sample that is associated should be reextracted. However, if the recovery is high and the associated samples do not contain the specific compound(s), the data can possibly be accepted with narration.

If a spike compound is outside of the acceptance limits in the matrix spike sample but is acceptable in the LCS, the data is considered acceptable. The cause of the failure is possibly attributable to matrix interference. However, if the compound fails in both the LCS and the MS/MSD, the result for that analyte is suspect and may not be reported for regulatory compliance purposes.

- 8.4 Surrogate acceptance criteria and Corrective Action: Surrogate recoveries are calculated on all samples, blanks and spikes. The recoveries are compared to laboratory established acceptance limits.

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

When a sample has a surrogate that falls outside of the laboratory established acceptance limit window, the problem should be investigated. If the recovery looks like it is affected by the sample matrix, the sample may be reinjected to confirm matrix interference. When a sample has no detectable surrogate recovery, the sample should be reextracted.

- 8.4 CAR: Whenever data is not acceptable because of a failing LCS or surrogate recovery, a corrective action report (CAR) must be initiated as soon as possible.
- 

## 9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the current revision of Method 8081 for other method performance parameters and requirements.

---

## 10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition. Promulgated Update III dated December, 1996, Method 8081A.

Katahdin Analytical Services, Inc., SOP CA-106, Standard Preparation, Documentation and Traceability.

Katahdin Analytical Services, Inc., SOP CA-515, Preparation of Aqueous Samples for Pesticides/PCBs Analysis-Methods 3510 and 3520.

Katahdin Analytical Services, Inc., SOP CA-500, Preparation of Soil/Sediment Samples by Sonication Using Method 3550 for Subsequent Pesticides/PCBs Analysis.

Katahdin Analytical Services, Inc., SOP CA-524, Preparation of Soil/Sediment Samples by Soxhlet Extraction Using Method 3540 for Subsequent Pesticides/PCBs Analysis.

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

## LIST OF FIGURES

Table 1 QC Requirements  
Table 2 Summary Of Method Modifications  
Figure 1 Instrument Run Log  
Figure 2 Review Checklist  
Figure 3 PQLs

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

TABLE 1  
QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch of twenty or fewer samples	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are < PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS	One per prep batch of twenty or fewer samples	Statistically derived limits. Note that limits wider than 70-130% are not allowable for some states, programs or clients, i.e. South Carolina	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are < PQL, narrate. Otherwise, reprep a blank and the remaining samples.
CCV	If calibration curve previously analyzed, analyze daily before samples and after every 20 samples.	$\pm 15\% D$	(1) Evaluate the samples: If the $\%D > +15\%$ and sample results are <PQL, narrate. If $\%D > \pm 15\%$ only on one channel, narrate. If $\%D > \pm 15\%$ for the closing CV, and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples back to last acceptable CV.
Matrix Spike\ Matrix Spike Duplicate	One for every set of 20 samples	Same as for LCS	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

TABLE 1, cont'd

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
6 pt of INDAB mix with mid-pt cal of Toxaphene and Chlordane	Initial cal prior to sample analysis	6pt calibration coefficient of determination $\geq 0.990$	(1) Repeat Initial calibration (2) If single pt cal Toxaphene, or Chlordane is identified in analysis of sample, 6 pt calibration run of identified compound with reanalysis of sample.
Demonstrate ability to generate acceptable P & A using 4 replicate analyses of a QC check standard	One time per analyst initially and annually thereafter.	All recoveries within method QC acceptance limits	Recalculate results; locate and fix problem; rerun P & A study for those analytes that did not meet criteria prior to sample analysis
MDL study	Once per year	Ideally, PQL = at least 3 * MDL..	Repeat MDL study

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
 DETECTOR (GC/ECD): SW-846 METHOD 8081

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-302-07	METHOD 8081, current revision
Apparatus/ Materials	None	
Reagents	None	
Sample preservation/ handling	None	
Procedures	<p>7.3.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being too narrow. The windows are: <math>\pm 0.05</math> for Heptachlor, Aldrin and all BHC compounds, <math>\pm 0.07</math> for all other target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of <math>\pm 0.03</math> minutes must be used if the established retention time window is less than 0.03 minutes.</p>	<p>7.6.3 If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. (Recording retention times to three decimal places rather than only two should minimize the instances in which the standard deviation is calculated as 0.000).</p>

---

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
DETECTOR (GC/ECD): SW-846 METHOD 8081

---

TABLE 2, cont'd

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-302-07	METHOD 8081, current revision
QC - Continuing Calibration		
QC - LCS	None	
QC - Accuracy/Precision	None	
QC - MDL	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8081

FIGURE 1

EXAMPLE OF INSTRUMENT RUN LOG

Katahdin Analytical Services, Inc. GC Laboratory Instrument Runlog  
 Instrument: GC08  
 Amount Injected 2ul Method: 608 / 8081 / 8082  
 Reviewed by/ Date: DL 8/31/04 (circle)

Date	Init.	Result File	Sample ID	MI	Y/N	Method	Column	Sequence
6/25/04	LAD	8UP/2-191	WG 8346-3 3545	Y	Y	PSTA/B137A.m	200/231	FW801, II J
		192	WG 8152-1 3510	N				
		193	-2					
		194	-3					
		195	WU 17127					
		196	RF 642A					
		197	WU 1713-1 3545	N	N			TR, DC6 b A/B
		198	EVAL P2806	N	Y			
		199	INDAG 0.05 PPM <sup>P2811</sup>	N	Y			Map. Equip. 9-cell. 2mb. B (ACT)
6/29/04	LAD	200	EVAL P2806	-	Y			
		201	EVAL P2806	N	Y	PSTA/B138A.m		
		202	IND AG 0.05 <sup>P2803</sup> PPM					
		203	0.005 P2857					
		204	0.010 P2858					
		205	0.025 P2859					
		206	0.10 P2860					
		207	0.25 P2861					
		208	0.05 <sup>P2862</sup> PPM					
		209	TOK 1.0 PPM P2845	Y				
		210	TC 0.5 PPM P2844	N	Y			
		211	WG 8339-1 3545	N	Y			
		212	-2					
		213	-3					
		214	WU 1743-1 RE					low Sulf. Conf. meth. A.
		215	WG 8378-1 3510	N	Y			low Sulf.
		216	-2	N	Y			
		217	-3	N	Y			
		218	WU 1725-2					
		219	-4					
		220	-6					

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
 DETECTOR (GC/ECD); SW-846 METHOD 8081

FIGURE 2  
 DATA REVIEW CHECKLIST

Verbal Due Date \_\_\_\_\_ Due Date \_\_\_\_\_

Client:	Primary	Secondary
Method:	Date:	Date:
SDG No:                      Level:	Initials:	Initials:
KAS No:	Approved :	<input type="checkbox"/> Yes

**PRIMARY REVIEW CHECKLIST**

- Highlight Method / project specific information. \_\_\_\_\_
- All needed forms are present . \_\_\_\_\_
- Correct Work Order Number or SDG name (all forms). \_\_\_\_\_
- Correct project name and spelling (all forms). \_\_\_\_\_
- Correct file numbers (all forms). \_\_\_\_\_
- Analysis Date Correct. \_\_\_\_\_
- Extraction Method & Analysis Method Correct. \_\_\_\_\_
- Product list compared to ROAs (compounds & PQLs). \_\_\_\_\_
- Chromatogram reviewed for unlabeled peaks (check product list). \_\_\_\_\_
- Flagging of all ROAs correct ( Florida Flagging  ). \_\_\_\_\_
- All tunes included (level IV) . \_\_\_\_\_
- All log book pages included (Soil weights,TCLP & SPLP). \_\_\_\_\_
- Verify quant results for CLP. \_\_\_\_\_
- Update sample history files. \_\_\_\_\_
- Sign & Date Manual integration ( CLP only ). \_\_\_\_\_
- Sample I.D.'s Truncated ( NARRATE ). YES  Please list KAS # below :

First correction  → Review and replace appropriate SDS Forms  .

Second correction  → Review and replace appropriate SDS Forms  .

TITLE: ANALYSIS OF PESTICIDES BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE  
 DETECTOR (GC/ECD): SW-846 METHOD 8081

FIGURE 3

PQLS FOR METHOD 8081

Parameter/Method	Analyte	Practical Quantitation Level (PQL)	
		Waters (ug/L)	Soils (ug/kg)
Organochlorine Pesticides  SW3510/SW8081A (W) SW3520/SW8081A (W) SW3550/SW8081A(S)	Aldrin	0.05	1.7
	Alpha BHC	0.05	1.7
	Beta BHC	0.05	1.7
	Delta BHC	0.05	1.7
	Gamma BHC (Lindane)	0.05	1.7
	Chlordane	0.50	17
	alpha-Chlordane	0.05	1.7
	gamma-Chlordane	0.05	1.7
	4,4'-DDD	0.10	3.3
	4,4'-DDE	0.10	3.3
	4,4'-DDT	0.10	3.3
	Dieldrin	0.10	3.3
	Endosulfan I	0.05	1.7
	Endosulfan II	0.10	3.3
	Endosulfan Sulfate	0.10	3.3
	Endrin	0.10	3.3
	Endrin Aldehyde	0.10	3.3
	Endrin Ketone	0.10	3.3
	Heptachlor	0.05	1.7
	Heptachlor Epoxide	0.05	1.7
Methoxychlor	0.50	17	
Toxaphene	1.00	33	

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Prepared By: George Brewer Date: 12/97  
 Approved By: \_\_\_\_\_  
 Group Supervisor: George Brewer Date: 01/29/01  
 Operations Manager: John C. Banta Date: 1/29/01  
 QA Officer: Deborah J. Nadeau Date: 1-29-01  
 General Manager: Dennis F. Kufan Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
02 7471A	Format changes, added pollution prevention, other minor changes to sections 7, 8 and QA Table.	GN	1-29-01	1/29/01
03 7471A	Changed Leeman PS200 Automated Mercury Analyzer to Cetac M6100 Mercury analyzer. Revised Sect. 10 to show correct reference material. Removed fig. 2 Revised sect. 4.8, 5.7 and 8.9 to reflect current practises. minor changes through out	LAD	021605	021605

---

**TITLE:           DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA  
METHOD 7471**

---

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

---

I acknowledge receipt of copy \_\_\_\_ of document **SOP CA-611-03**, titled **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_\_ of document **SOP CA-611-03**, titled **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

TITLE:           **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

---

## 1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services, Inc. personnel for the digestion and analysis solid samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in soils, sediments, bottom deposits, and sludges under USEPA Method 7471 (Test Method for Evaluating Solid Wastes, USEPA SW 846, Third Edition).

### 1.1 Definitions

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory reagent grade water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory reagent grade water used to verify calibration accuracy.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

PB - Preparation Blank - Laboratory reagent grade water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

---

**TITLE:           DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA  
METHOD 7471**

---

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

### 1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7471. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7471 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

### 1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Management Plan and Safety Manual and follow appropriate procedures such as wearing safety glasses and

---

**TITLE:       DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA  
METHOD 7471**

---

gloves when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location and use of all safety equipment.

**1.4   Pollution Prevention/Waste Disposal**

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in cold vapor atomic absorption may contain high concentrations of acids, mercury, and other toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested mercury samples and standards and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled "Waste Stream A") that is located in the Metals Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Hazardous Waste Management Plan and Safety Manual and the Department Manager.

---

**2.0   SUMMARY OF METHOD**

The cold vapor atomic absorption technique is based on the absorption of radiation at 253.7 nm by mercury vapor. It relies on the volatility of elemental mercury at room temperature. During preparation, organic mercurials are oxidized and elemental mercury is ionized to  $Hg^{3+}$ . During instrumental analysis, mercuric ions are reduced to elemental mercury by the addition of stannous chloride. Elemental mercury is then aerated from solution and passes through a cell positioned in the path of a mercury spectrophotometer, where absorbance (peak height) is measured as a function of mercury concentration and recorded by the associated computer. The mercury vapor is then swept out of the instrument into an exhaust hood, where it is evacuated from the laboratory.

---

**3.0   INTERFERENCES**

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low bias for analytical results. Certain volatile organic materials will also non-specifically absorb

---

**TITLE:       DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA  
              METHOD 7471**

---

radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Samples that are high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine, which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

---

**4.0    APPARATUS AND MATERIALS**

- 4.1    250 mL Pyrex media bottles with plastic screw caps, for use as digestion vessels.
- 4.2    Water bath capable of maintaining a constant temperature of 95° C.
- 4.3    Analytical balance capable of weighing to 0.01 g.
- 4.4    Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.
- 4.5    Repipettors (adjustable repeating pipettors with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents.
- 4.6    Spirit-filled thermometer, NIST-traceable, covering the range from 20° to 110° C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.
- 4.7    Disposable graduated polystyrene sample cups, 200 mL capacity.
- 4.8    CETAC M6100 Mercury Analyzer and associated peripherals and parts.

Refer to Katahdin SOP CA-629, current revision, "Operation and Maintenance of the CETAC M6100 Mercury Analyzer" for additional required materials.

---

TITLE:           **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

---

## 5.0 REAGENTS

- 5.1 Laboratory reagent grade water – mercury-free water which has been treated by the Culligan Water System and the Millipore Mill-Q<sup>®</sup> Water System and meets the specifications of ASTM Type II water
- 5.2 Concentrated nitric acid (HNO<sub>3</sub>), trace metal grade
- 5.3 Concentrated hydrochloric acid (HCl), trace metal grade
- 5.4 Aqua regia: Prepare an appropriate amount immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO<sub>3</sub> in a heat-proof beaker or flask. Preparation of aqua regia must be performed in a fume hood.
- 5.5 Potassium permanganate solution, 5% w/v: Dissolve 50 g of potassium permanganate in 1 L laboratory reagent grade water. The source reagent should be labeled as suitable for use in mercury determination.
- 5.6 Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in laboratory reagent grade water and dilute to a final volume of 1 L.
- 5.7 Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory reagent grade water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. Reagent should be labeled as suitable for use in mercury determination.
- 5.8 Mercury Stock Standards: Two 10.0 mg/L mercury stock standards, obtained from separate sources, are required. The mercury concentrations of these standards must be certified by the manufacturers as traceable to NIST reference standards.
- 5.9 Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. This intermediate standard is used to prepare calibration standards, matrix spikes, CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard and instructions for its dilution may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard A must be prepared fresh daily, and disposed of appropriately after use. (Note: the concentrations of all stock standards must be certified by the vendors as traceable to NIST reference materials).

---

TITLE: **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

---

- 5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. The source of the stock standard used to prepare Intermediate Mercury Standard B must be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8.0). The identity of the stock standard currently used to prepare this intermediate standard and instructions for its dilution may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard B must be prepared fresh daily, and disposed of appropriately after use.
- 5.11 Solid Reference Material: A soil with a known or empirically-established mercury concentration for use in preparing the laboratory control sample for soils. Solid reference materials should be purchased with certificates listing reference values and quality control acceptance limits. See Figure 3 for an example certificate of analysis for a solid reference material.

---

**6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING**

Soil samples to be analyzed for mercury should be collected and preserved as described in the following table.

Matrix	Container <sup>1</sup>	Collection Volume/Weight	Preservation/Treatment	Holding Time
Solid	P, G	40 g	Cool to 4°C ± 2°	28 days

<sup>1</sup> P = polyethylene, G = glass

---

**7.0 PROCEDURES**

**BOTTLE PREPARATION**

- 7.1 Mercury digestion bottles are reused, and must be cleaned between uses. After the previous contents of the bottles have been discarded, bottles are segregated according to whether the measured mercury concentrations of the previous contents were above the PQL (contaminated bottles) or below the PQL (uncontaminated bottles). Labels are removed from the bottles by wiping with a paper towel saturated with toluene. Both contaminated and uncontaminated bottles

---

**TITLE:           DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA  
METHOD 7471**

---

are then cleaned with Liquinox and water, if necessary, to remove visible grime, and rinsed thoroughly with tap water.

- 7.2    Uncontaminated bottles are then triple-rinsed with laboratory reagent grade water, and are ready for reuse.
- 7.3    Contaminated bottles are placed in a bath containing 10% HCl for at least 12 hours. After acid-leaching, these bottles are triple rinsed with laboratory reagent grade water, and are then ready for reuse.

#### PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

- 7.4    Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer database and print out a copy of the benchsheet. All necessary details of sample preparation (standards preparation information, digestion times, initial weights and final volumes, pertinent observations, etc.) must be recorded on this benchsheet, which will be bound in the Mercury Preparation Logbook. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.
- 7.5    Using a silver paint marker, label clean digestion bottles with the appropriate sample numbers and standard identifications for each sample and standard to be digested.
- 7.6    Using calibrated adjustable pipettes, prepare calibration standards by adding 0 uL, 20 uL, 50 uL, 100 uL, 500 uL, and 1000 uL of Intermediate Mercury Standard A to separate appropriately-labeled digestion bottles. The mercury concentrations of these calibration standards will be, respectively, 0 ug/L (calibration blank), 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L, and 10.0 ug/L. The 0.2 ug/L and 0.5 ug/L standards are analyzed during analysis as the PQL standard and the CCV (refer to Section 8.0), respectively, as well as being used in the creation of the calibration curve.
- 7.7    Using a calibrated adjustable pipette, prepare the initial calibration verification (ICV) standard (refer to Section 8) by adding 600 uL of Intermediate Mercury Standard B to an appropriately labeled digestion bottle. The mercury concentration of the ICV will be 6.0 ug/L.
- 7.8    Prepare an appropriate number of calibration blanks (ICB/CCB) and preparation blanks (PBW) by adding 1.0 mL of laboratory reagent grade water to labeled digestion bottles.
- 7.9    Prepare an appropriate number of laboratory control samples (LCSS) by weighing appropriate masses of solid reference material into labeled digestion bottles. The

---

**TITLE:           DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA  
METHOD 7471**

---

mercury concentration of these LCSSs will depend on the solid reference material used, and the mass of each aliquot. Refer to Figure 4 for an example certificate of analysis for a solid reference material.

- 7.10 Matrix spikes are prepared by adding 100 uL of Intermediate Mercury Std A to each matrix spike sample. The amount of mercury added to each matrix spike increases the final digestate concentration by 1.0 ug/L.
- 7.11 All calibration standards, QC samples, and blanks are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, Steps 7.12 through 7.16 of this SOP.

#### SAMPLE PREPARATION AND DIGESTION

- 7.12 Weigh three approximate 0.2 g portions (a total of approximately 0.6 g) of untreated sample from different parts of the sample container and place them in the bottom of a labeled digestion bottle. The purpose of using three portions is to obtain a representative sample from the sample container.
- 7.13 Add 5 mL of laboratory reagent grade water and 5 mL of aqua regia to each sample, standard, and QC sample. Place bottles in a water bath located in a fume hood and heat for 2 minutes at 95° C. Remove the bottles from the water bath and allow them to cool in a fume hood.
- 7.14 Add 50 mL of laboratory reagent grade water and 15 mL of potassium permanganate solution to each digestion bottle, swirl to mix, and allow to stand for at least 15 minutes. Samples that contain large amounts of oxidizable organic matter may require additional 15 mL aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 15 mL aliquots to samples as necessary until the purple color persists for 15 minutes. If any of the samples requires these additional aliquots of permanganate, note that fact on the mercury preparation benchsheet and accordingly adjust the final volumes recorded on the benchsheet for those samples.

When a persistent purple color has been obtained for all samples, place the digestion bottles in the water bath and heat for 30 minutes at 95° C.

- 7.15 Remove the bottles from water bath and allow them to cool in a fume hood. If any of the samples have become colorless during heating, add additional 15 mL aliquots of potassium permanganate solution as necessary to obtain a persistent purple color and heat for an additional 30 minutes at 95° C. Record any information regarding additional permanganate aliquots on the mercury preparation benchsheet

---

**TITLE:           DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA  
METHOD 7471**

---

and accordingly adjust the final volumes recorded on the benchsheet for the samples affected.

- 7.16 Add 6 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion bottle and swirl to mix. Perform this addition in a fume hood, as chlorine gas may be evolved. This will reduce the excess permanganate, and the sample will change from purple to colorless. Add 50 mL of laboratory reagent grade water to each bottle. Wait at least 30 seconds before proceeding with analysis.

#### INSTRUMENTAL ANALYSIS

- 7.17 Digested mercury samples are analyzed using the CETAC M6100 Mercury Analyzer. Analysis is automated and is controlled by the QuickTrace software running on a dedicated PC. Detailed instructions for setting up the instrument and running samples are given Katahdin SOP CA-629, "Operation and Maintenance of the CETAC M6100 Mercury Analyzer". The following information specifically pertains to analysis of digested samples in accordance with USEPA Method 7471, and should be used in conjunction with the instructions given in Katahdin SOP CA-629.
- 7.18 Instrument operating conditions and quality control acceptance limits are specified in the instrument software in "templates". The template that is used to analyze digested samples in accordance with USEPA Method 7471 is named "SW846-7470-7471".
- 7.19 Prior to analysis, digested samples, standards, and QC samples are decanted into autosampler tubes which are placed in racks on the instrument's autosampler. The "standards" autosampler rack has 10 positions for 25 x 100 mm autosampler tubes (50 mL capacity). Tubes containing the calibration standards, the ICV, the CCV, the ICB/CCB, and the PQL standard are placed in the appropriately labeled positions in this autosampler rack.
- 7.20 Client samples, batch QC samples (preparation blanks and laboratory control samples), and matrix QC samples (duplicates and matrix spikes) are decanted into 17 x 100 mm autosampler tubes (15 mL capacity), which are placed in the one of the "samples" autosampler racks. The "samples" autosampler racks have 60 positions for 17 x 100 mm autosampler tubes. Instructions for filling the "samples" autosampler racks, including recording the rack position of each sample, are contained in Katahdin SOP CA-629, "Operation and Maintenance of the CETAC M6100 Mercury Analyzer".

#### METHOD OF STANDARD ADDITIONS

---

**TITLE:           DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA  
METHOD 7471**

---

7.21 The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

7.21.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume  $V_x$ , are taken. To the first (labeled A) is added a known volume  $V_s$  of a standard analyte solution of concentration  $C_s$ . To the second aliquot (labeled B) is added the same volume  $V_s$  of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration  $C_x$  is calculated:

$$C_x = \frac{S_B V_s C_s}{(S_A - S_B) V_x}$$

where  $S_A$  and  $S_B$  are the analytical signals (corrected for the blank) of solutions A and B, respectively.  $V_s$  and  $C_s$  should be chosen so that  $S_A$  is roughly twice  $S_B$  on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

7.21.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 3. A linear regression program may be used to obtain the intercept concentration.

---

**TITLE:           DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA  
METHOD 7471**

---

7.21.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

- 1) The apparent concentrations from the calibration curve must be linear over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised.
- 2) The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.
- 3) The determination must be free of spectral interference and corrected for nonspecific background interference.

#### DATA REDUCTION AND REPORTING

7.22 Results are obtained in units of ug/L in the digestate. Results that exceed the calibration range of the instrument may not be reported - the sample must be appropriately diluted and reanalyzed. Results for diluted samples must be multiplied by the dilution factor prior to reporting. If additional aliquots of potassium permanganate were added during digestion, the change in digestate final volume must be taken into account in calculating the final result. Mercury results for solid samples are reported in units of ug/g, calculated on a dry weight basis. Calculation of mercury results for solid samples is performed automatically by the Metals reporting database, as follows:

$$\text{Mercury Concentration in Solid (mg/kg dry wt.)} = \frac{(C) \times (DF) \times (FV) \times 100}{(W) \times (TS)}$$

where C = Measured digestate concentration (ug/L)  
DF = Instrument dilution factor  
FV = Digestate final volume (L)  
W = Digested wet sample weight (g)  
TS = Total Solids (%)

7.23 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported as "<PQL".

---

TITLE:           **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

---

## **8.0    QUALITY CONTROL AND ACCEPTANCE CRITERIA**

USEPA Method 7471 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.6 through 7.10 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The supervisor, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

### **INITIAL DEMONSTRATION OF PERFORMANCE**

- 8.1    Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument by each method. This determination requires seven replicate analyses of laboratory reagent grade water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.
  
- 8.2    Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of laboratory reagent grade water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value

---

**TITLE:           DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA  
METHOD 7471**

---

to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

#### ANALYTICAL RUN QC

- 8.3 Instrument calibration - The instrument must be calibrated each time it is set up, and calibration standards must be digested each day that samples are digested. Calibration includes analysis of a calibration blank and five calibration standards with graduated concentrations in the appropriate range. The concentration of one of the calibration standards must be at the Practical Quantitation Level (PQL). Because mercury may be adsorbed onto the walls of glass and plastic containers, the intermediate standard used to prepare calibration standards must be prepared fresh daily. The correlation coefficient for the calibration curve must be at least 0.995. If the calibration curve does not pass this test, analysis must be halted, the problem corrected, and the instrument recalibrated.
- 8.4 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared from a standard source different than that of the calibration standard and at a concentration within the working range of the instrument. The result of the ICV must fall within 90% to 110% of the expected value. If the ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.
- 8.5 The Continuing Calibration Verification (CCV) solution is analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standard used for calibration at a concentration near the mid-point of the calibration curve. Results of the CCVs must fall within 80% to 120% of the expected value. If a CCV fails, associated sample results may not be reported from the run until the problem is corrected and a passing CCV has been analyzed. Also, all samples analyzed after the last passing CCV must be reanalyzed.
- 8.6 A calibration blank is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed. Also, all samples analyzed after the last passing CCB must be reanalyzed.
- 8.7 A standard with a mercury concentration that is at the Practical Quantitation Limit (PQL) is analyzed at the beginning of the run to determine calibration accuracy at

---

**TITLE:           DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA  
METHOD 7471**

---

the reporting limit. Result of the PQL standard should fall within 50% to 150% of the expected values. No corrective action has been established at this time.

#### PREPARATION BATCH QC SAMPLES

- 8.8     A preparation blank (PBS), consisting of laboratory reagent grade water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL). If a preparation blank fails, results may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL, associated sample results that are greater than or equal to ten times the measured preparation blank concentration may be reported with "B" notation. Associated sample results that are below the PQL may be reported without notation.
- 8.9     A laboratory control sample (LCSS), consisting of solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested. The laboratory uses a reference value and statistical acceptance limits for laboratory control samples are supplied by the vendor of the solid reference material.

#### SAMPLE MATRIX QC SAMPLES

- 8.10    Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. Matrix spike recoveries for these samples are calculated as follows:

$$\text{Recovery (\%)} = \frac{(P - S)}{A} \times 100\%$$

where:

P = Spiked sample value  
S = Original sample value  
A = Spike amount

The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, a matrix interference should be suspected and the associated sample result should be flagged on the report of analysis.

---

TITLE:           **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA  
METHOD 7471**

---

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

$$\text{RPD (\%)} = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where:

D<sub>1</sub> = Spike sample result

D<sub>2</sub> = Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

---

## 9.0    **METHOD PERFORMANCE**

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Metals Supervisor and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the current revision of Method 7471 for other method performance parameters and requirements.

---

## 10.0   **APPLICABLE DOCUMENTS/REFERENCES**

Test Methods for Evaluating Solid Wastes, United States Environmental Protection Agency, USEPA SW 846, Third Edition, Final Update III (12/96), Method 7471A

QuickTrace M6100 Mercury Analyzer Operator Manual Version 1.0.1, CETAC Technologies

QuickTrace Mercury Analyzer Software Manual, CETAC Technologies.

TITLE:           **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA  
METHOD 7471**

---

List of Tables and Figures

Table 1	QC Requirements
Table 2	Method Modifications
Figure 1	Example Mercury Preparation Logbook Page
Figure 2	Standard Additions Plot
Figure 3	Example Certificate of Analysis for a Solid Reference Material

**TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

**TABLE 1**  
**QC REQUIREMENTS**

<b>Parameter/ Method</b>	<b>QC Check</b>	<b>Minimum Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>
Mercury/ USEPA 7471	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient $\geq 0.995$ .	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within $\pm 10\%$ of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL.	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within $\pm 50\%$ of true value.	No corrective action required at this time.
	Continuing Calibration Verification (CCV)	At beginning or run, after every 10 samples, and at end of the run	Recovery within $\pm 20\%$ of true value	Repeat calibration and reanalyze all samples analyzed since the last successful CCV.
	Continuing Calibration Blank (CCB)	At beginning or run, after every 10 samples, and at end of the run	Less than PQL.	Repeat calibration and reanalyze all samples analyzed since the last successful CCB.
	Preparation Blank (PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration $\geq$ PQL and $< 10x$ the blank concentration.
	Laboratory Control Sample (LCSS)	One per digestion batch of 20 or fewer samples.	Recovery within vendor-supplied acceptance limits.	Redigest all affected samples.

TITLE: **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

TABLE 1, cont'd

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA 7471	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery $\pm 25\%$ of true value, if sample $> 4x$ spike value.	Flag results.
	Matrix Spike Duplicate Sample (P)	One per digestion batch of 20 or fewer samples.	1) Recovery $\pm 25\%$ of true value, if sample $< 4x$ spike added. 2) RPD 20% for duplicate spikes.	Flag results
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL $<$ PQL	1) Repeat IDL study. 2) Raise PQL.
	Method Detection Limit (MDL) Study	Annually.	Ideally, PQL = at least 3 X the MDL.	1) Repeat MDL study. 2) Raise PQL.

**TITLE:           DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

TABLE 2  
 SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-611-03	USEPA METHOD 7471, current revision
Reagents	Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	Sampling and gas stream switching performed automatically by mercury analyzer.	Sampling and gas stream switching performed manually by analyst.
QC – Calibration Verification	1) Known reference sample (ICV) analyzed daily. 2) Calibration verified after every 10 samples with CCV.	1) Known reference sample analyzed quarterly. 2) Calibration verified after every 20 samples.
QC - Calibration Blanks	Acceptance criteria employed for 7471: $\pm$ PQL	Acceptance criteria stated in 7471: $\pm$ MDL

TITLE: **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK

Katahdin Analytical Services, Inc.

Reagent Information: JT Baker HNO<sub>3</sub>: V42044 JT Baker HCL: N/A JT Baker H<sub>2</sub>SO<sub>4</sub>: V19092 JT Baker KMNO<sub>4</sub>: M2164 JT Baker K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>: M2156 JT Baker NH<sub>2</sub>OH-HCl: M2167 Method: 7470

Standards Information: 1ppm A = mw7971 1ppm B = mw7972 LCSV = 125uL of 1ppm B to 25mL Spike(S/P) = 25uL of 1ppm B to 25mL

Metals Preparation Benchsheet  
REVIEWED  
MSF 1.14.04  
KATAHDIN ANALYTICAL SERVICES  
METALS SECTION

TCLPMS(M) = 200uL of 1ppm A to 100mL  
ICV = 600uL of 1ppm A to 100 mL  
S0.2 = 20uL of 1ppm B to 100 mL  
S0.5 = 50uL of 1ppm B to 100 mL  
S1.0 = 100uL of 1ppm B to 100 mL  
S5.0 = 500uL of 1ppm B to 100 mL  
S10.0 = 1000uL of 1ppm B to 100 mL

Digestion Start Time (@ 95 °C): 13:30 Digestion End Time: 15:30

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LCSWUA13HGW0	UA13HGW0	0.025	L	0.025	L	AQ	HG	EAM	01/13/2004	N/A	N/A	N/A	N/A		
PBWUA13HGW0	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004	N/A	N/A	N/A	N/A		
WU0041-001	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-001P	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-001S	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-002	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-003	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-004	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-005	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-006	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0041-007	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-001	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-002	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-003	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-004	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-005	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-006	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-007	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-008	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-009	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						
WU0051-010	UA13HGW0		L		L	AQ	HG	EAM	01/13/2004						

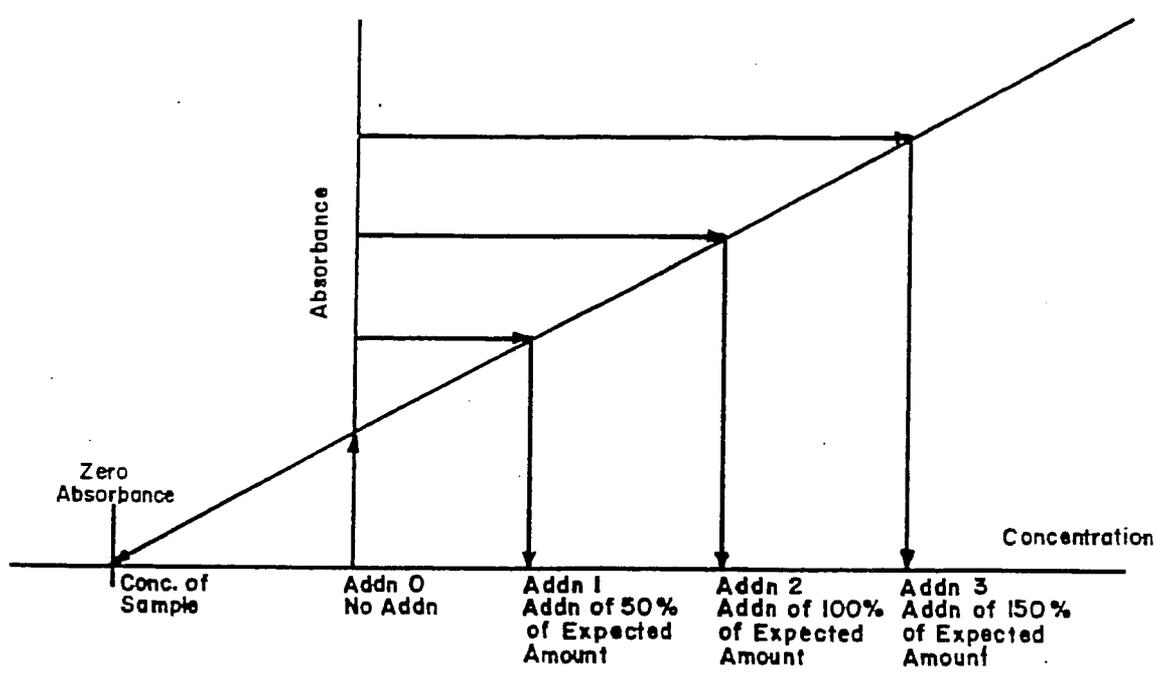
MSF 1.14.04

---

TITLE:       **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

---

FIGURE 2  
STANDARD ADDITIONS PLOT



TITLE: **DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471**

FIGURE 3

EXAMPLE CERTIFICATE OF ANALYSIS FOR A SOLID REFERENCE MATERIAL



*Redd 12/08/00*

**Certification**

PriorityPollutn™/CLP Inorganic Soils - Microwave Digestions

Quality Control Standards

Catalog No PPS-46

Lot No. 245

Method 3051 HNO3 only

Method 3051 HNO3 and HCl

Parameter	Total Concentration <sup>1</sup>	Certified Value <sup>2</sup>	Performance Acceptance Limits™ <sup>3</sup>	Method 3051 HNO3 and HCl	
				Certified Value <sup>2</sup>	Performance Acceptance Limits™ <sup>3</sup>
TRACE METALS PriorityPollutn™ (Catalog No 540)	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg
Aluminum	53800*	5580	3610 - 7550	6110	3260 - 8960
Antimony	198	29.2	D.L. - 93.7	119	32.6 - 206
Arsenic	152	131	97.3 - 164	137	102 - 172
Barium	694*	115	89.0 - 142	127	98.2 - 157
Beryllium	103	93.7	73.5 - 114	92.1	72.2 - 112
Boron	158	106	78.1 - 134	126	92.5 - 159
Cadmium	131	119	91.8 - 147	119	91.3 - 146
Calcium	16100*	11400	8520 - 14300	11400	8520 - 14300
Chromium	135	83.1	66.4 - 99.8	85.2	68.1 - 102
Cobalt	125	118	93.8 - 142	127	101 - 153
Copper	136	115	93.9 - 135	119	97.4 - 140
Iron	26400*	9190	5590 - 12800	10900	6630 - 15200
Lead	172	139	106 - 172	137	105 - 170
Magnesium	5520*	2430	1960 - 2900	2550	2060 - 3040
Manganese	520*	291	235 - 347	320	259 - 382
Mercury	2.60	2.48	1.69 - 3.27	2.48	1.69 - 3.27
Molybdenum	111	88.1	59.0 - 117	108	82.9 - 134
Nickel	186	160	126 - 195	167	131 - 204
Potassium	31000*	2920	2280 - 3560	3040	2370 - 3710
Selenium	105	88.3	65.4 - 111	88.0	65.2 - 111
Silver	132	56.9	11.9 - 102	46.1	31.0 - 61.1
Sodium	12100*	771	523 - 1020	772	524 - 1020
Strontium	294*	76.1	55.2 - 96.9	78.7	57.1 - 100
Thallium	154	137	78.7 - 196	113	64.8 - 162
Tin	117	57.7	D.L. - 129	119	88.8 - 149
Titanium	2900*	82.6	53.9 - 111	115	75.3 - 156
Vanadium	121	65.8	44.9 - 86.6	72.8	49.6 - 95.9
Zinc	100	72.8	11.0 - 135	69.2	53.5 - 84.9

CONTINUED ON BACK

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND GFAA

Prepared By: George Brewer Date: 3/98  
 Approved By: \_\_\_\_\_  
 Group Supervisor: George Brewer Date: 01/24/01  
 Operations Manager: J. C. Burt Date: 1/24/01  
 QA Officer: Dorothy J. Nadeau Date: 1.24.01  
 General Manager: Debra L. Huffman Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3050B	Format changes, added pollution prevention, added MSD, added spiking instruction tables.	GN	1.24.01	1/24/01
02 3050B	Removed all references/procedures devoted to GFAA. Added use of digestates for ICP-MS analysis. Revised standard solution names + concs. in Tables 3 + 4 to reflect current practice.	GN	8.29.02	8.29.02

---

TITLE: **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

---

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

---

I acknowledge receipt of copy \_\_\_ of document **SOP CA-605-02**, titled **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_ of document **SOP CA-605-02**, titled **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

**TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

---

## **1.0 SCOPE AND APPLICATION**

The purpose of this SOP is to describe the Katahdin Analytical Services, Inc. procedure utilized to dissolve solid matrices and solubilize metals from solid samples prior to analysis for metals by ICP-AES and ICP-MS. This SOP applies to samples prepared by EPA Method 3050, with method modifications as summarized in Table 2.

This procedure applies to all solid sample (e.g. sediments, sludges, soils, and ashes) preparations for ICP-AES and ICP-MS analyses. This method is not a total digestion technique for most samples. It is a very strong acid digestion that will dissolve almost all elements that could become "environmentally available". By design, elements bound in silicate structures are not normally dissolved by this procedure as they are not usually mobile in the environment.

### **1.1 Definitions**

ICP-AES – Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICP-MS – Inductively Coupled Plasma Mass Spectrometry.

LCSS – Laboratory Control Sample for Solids – A standard or solid reference material that has been brought through the sample preparation process.

Matrix Spike – An aliquot of a sample to which a known amount of analyte has been added before digestion.

PBS – Preparation Blank for Solids – An aliquot of reagent water that has been brought through the sample preparation process.

### **1.2 Responsibilities**

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of solid samples by USEPA Method 3050 for metals analysis. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Training".

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of solid samples by USEPA Method 3050 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the procedure or irregularities with the samples should also be recorded in the lab notebook and reported to the responsible Supervisor or designated qualified data reviewer.

---

**TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS  
ANALYSIS BY ICP-AES AND ICP-MS**

---

It is the responsibility of the Supervisor to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

### 1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, aprons, dust masks, and shoe protectors, is available in the Metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully, while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever beakers are being heated on a hot plate. Use caution when handling hot beakers.

Personnel are required to read the Katahdin Chemical Hygiene Plan and Safety Manual before performing this procedure, and must be familiar with the general rules for laboratory safety, personal hygiene, housekeeping, and use of protective clothing and equipment.

### 1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Chemical Hygiene Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Chemical Hygiene Plan and Safety Manual.

---

---

**TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

---

## **2.0 SUMMARY OF METHOD**

A representative 1 to 2 g (wet weight) sample is digested with repeated additions of nitric acid and hydrogen peroxide. Hydrochloric acid is added to the initial digestate and the sample is refluxed. The digestate is then filtered and diluted to a final volume of 100 mL.

---

## **3.0 INTERFERENCES**

Interferences are discussed in the applicable analytical SOPs.

---

## **4.0 APPARATUS AND MATERIALS**

- 4.1 100 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning").
- 4.2 Ribbed watch glasses, 75 mm diameter, pre-cleaned as above.
- 4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.
- 4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.
- 4.5 Hot plate, griddle, or other heating source - adjustable and capable of maintaining a temperature of  $95^{\circ}\text{C} \pm 5^{\circ}\text{C}$ . Hot plates must be numbered for easy identification.
- 4.6 Device for measuring hot plate temperature. This may consist of a heat-resistant Erlenmeyer flask containing reagent water in which a thermometer is immersed. The temperature of each hot plate used is measured and recorded each day. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.
- 4.7 Plastic funnels, pre-cleaned as in Section 4.1.
- 4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.
- 4.9 Polyethylene wash bottles for dispensing reagent water and 5%  $\text{HNO}_3$ .
- 4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of

---

**TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

---

5% HNO<sub>3</sub>, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water, again allowing each rinse to drain completely. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

- 4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity.
- 4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, 1:1 HNO<sub>3</sub>, and concentrated HCl.
- 4.13 Analytical balance capable of reading to 0.01 gram.
- 4.14 Spatulas, scoops, or spoons; plastic or stainless steel, rinsed with 5% HNO<sub>3</sub> and reagent water.

---

**5.0 REAGENTS**

- 5.1 Concentrated nitric acid, HNO<sub>3</sub> – trace metals grade.
  - 5.2 Concentrated hydrochloric acid, HCl – trace metals grade.
  - 5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).
  - 5.4 Nitric acid, 1:1. Add a volume of concentrated HNO<sub>3</sub> to an equivalent volume of reagent water and swirl gently to mix.
  - 5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO<sub>3</sub> to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.
  - 5.6 30% hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) - spectrometric grade.
  - 5.7 Multielement spiking solutions (see Table 3 for a list of required spiking solutions).
  - 5.8 Solid reference material – a soil containing all the elements of interest, with empirically established method-specific recoveries and acceptance limits for all analytes. Solid reference materials are purchased with documentation of analysis provided by the vendor. See Figure 4 for an example certificate of analysis for a solid reference material.
-

---

TITLE: **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

---

**6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING**

Samples should be collected in clean plastic or glass containers. Samples must be refrigerated ( $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ ) upon receipt by the laboratory. The holding time for solid samples is 6 months from the date of sample collection.

---

**7.0 PROCEDURE**

The procedure described below is condensed for quick reference in Table 3.

**SAMPLE PREPARATION**

- 7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet (see Figure 2 for an example). Print labels for the digestate containers, and attach them to the polyethylene sample containers that will contain the digestates.
- 7.2 Submerge previously cleaned beakers and watch glasses three times into a 10% nitric acid bath, then rinse three times with reagent water. Label beakers with sample numbers.
- 7.3 Weigh 1 to 2 g of well-mixed sample into a properly cleaned, labeled, and tared Griffin beaker. Record the weight of each sample on the printout of the digestion spreadsheet.
- 7.4 Weigh an appropriate amount of solid reference material to a clean, labeled, and tared Griffin beaker to serve as a laboratory control sample.
- 7.5 Add spike solutions to matrix spike samples (refer to Tables 3 and 4 for spiking instructions).
- 7.6 Add 10 mL of 1:1  $\text{HNO}_3$ , mix the slurry, and cover the beaker with a ribbed watch glass. Place beaker on a hot plate; gently heat the sample to  $95^{\circ}\text{C} \pm 5^{\circ}\text{C}$  and reflux for 10 to 15 minutes without boiling. Remove the beaker from the hot plate and cool the sample.
- 7.7 Add 5 mL of concentrated  $\text{HNO}_3$  to the sample, replace the watch glass, and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by  $\text{HNO}_3$ , repeat this step (addition of 5 mL of concentrated  $\text{HNO}_3$ ) until no brown fumes are given off by the sample, indicating complete reaction by  $\text{HNO}_3$ .

---

**TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS  
ANALYSIS BY ICP-AES AND ICP-MS**

---

- 7.8 Continue heating the sample at  $95^{\circ}\text{C} \pm 5^{\circ}\text{C}$  without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the beaker from the hot plate and cool the sample.
- 7.9 Add 2 mL of reagent water and 3 mL of 30%  $\text{H}_2\text{O}_2$  to the sample, replace the watch glass, and heat gently on the hot plate to start the peroxide reaction. Continue heating until effervescence subsides.
- 7.10 Add an additional 7 mL of 30%  $\text{H}_2\text{O}_2$  in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.
- 7.11 Continue heating the sample at  $95^{\circ}\text{C} \pm 5^{\circ}\text{C}$  without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the beaker from the hot plate and cool the sample.
- 7.12 Add 10 mL of concentrated HCl to the digest from 7.11, replace the watch glass, and reflux at  $95^{\circ}\text{C} \pm 5^{\circ}\text{C}$  for 15 minutes. Remove the beaker from the hot plate and cool the sample.
- 7.13 Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated polystyrene specimen container or graduated polyethylene sample container with attached snap lid. Using a wash bottle, rinse the beaker with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse. Using the graduations on the specimen container or snap-lid container, dilute to 100 mL with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for ICP-AES or ICP-MS analysis.
- 7.14 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) reagent lot numbers, spiking information, and hot plate temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 2.
- 7.15 Reopen the electronic ACCESS spreadsheet for the digestion and transcribe the sample weights from the handwritten, bound copy into the electronic copy. The information in this electronic

---

**TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

---

spreadsheet will later be imported into the ACCESS metals database and used to calculate sample concentrations on a weight basis.

- 7.15 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area. Log the digestion batch into the Internal Custody Log for Metals Digestates (see Figure 3).

#### CALCULATIONS

- 7.16 Analytical results for solid samples are reported on a dry weight basis. Total solids are determined by the Wet Chemistry Group, and are recorded in spreadsheets that are electronically imported into the Access metals database. Final dry weight concentrations are calculated by the Access database as follows:

$$\text{Concentration (mg/kg dry weight)} = (C \times V) / (W \times S)$$

where: C = Measured concentration (mg/L)  
V = Digestate final volume (L)  
W = Sample wet weight (kg)  
S = % Solids/100

---

## 8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 3050 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

- 8.1 At least one preparation blank for soils (PBS) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBS consists of a 1.0 g aliquot of reagent water that is digested using the same reagents as those used to digest associated samples. Refer to the appropriate analytical SOP for PBS acceptance criteria and corrective actions.

---

**TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

---

- 8.2 At least one laboratory control sample for soils (LCSS) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSS consists of an aliquot of a solid reference material for which the concentrations of the analytes of interest have been empirically established (solid-matrix LCSS), or an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations (aqueous-matrix LCSS). The solid reference material should normally be used as the LCSS, unless a particular client or analytical program requires that spiked reagent water be used. The LCSS is digested using the same reagents as those used to digest associated samples. Directions for spiking the aqueous-matrix LCSS are contained in Figure 3. The measured analyte recoveries for the LCSS are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSS recovery acceptance criteria and corrective actions.
- 8.3 Matrix spike samples are processed along with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is fortified with known amounts of all analytes of interest prior to digestion. Matrix spike recoveries are used to assess the biasing effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figure 2. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.
- 8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.
- NOTE: Clients may choose specific samples for matrix spike and duplicate analysis; otherwise, the choice is left to the person performing the digestion.
- 8.5 The quality control measures and frequencies described above are minimum requirements. Individual clients and analytical programs may impose additional QC requirements.

---

**9.0 METHOD PERFORMANCE**

Refer to the applicable instrumental analysis SOP for method performance information.

---

**10.0 APPLICABLE DOCUMENTS/REFERENCES**

"Test Methods for the Evaluation of Solid Waste," United States Environmental Protection Agency, SW-846, Third Edition, Final Update III, 12/96, Method 3050B.

---

**TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

---

LIST OF TABLES AND FIGURES

Table 1	QC Requirements – Method 3050
Table 2	Summary of Method Modifications – Method 3050
Table 3	Preparation of Matrix Spikes and Spiking Solutions
Table 4	Element Concentrations in ICP-AES Matrix Spikes and Their Component Spiking Solutions
Figure 1	Procedure Condensation – Method 3050
Figure 2	Example Page from Metals Sample Preparation Logbook
Figure 3	Example Page from Internal Custody Log for Metals Digestates
Figure 4	Example Certificate of Analysis for Solid Reference Material

**TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

**TABLE 1**  
**QC REQUIREMENTS – METHOD 3050**

Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3050	Preparation Blank for Solids (PBS)	One per prep batch of 20 or fewer samples.	Refer to analytical method.	Refer to analytical method.
	Laboratory Control Sample for Solids (LCSS)	One per prep batch of 20 or fewer samples.	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Duplicate Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Demonstration of analyst proficiency	One-time demonstration by each analyst performing the method.	Must pass all applicable QC for method.	Repeat analysis until able to perform passing QC; document successful performance in personal training file.

**TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

TABLE 2  
 SUMMARY OF METHOD MODIFICATIONS – METHOD 3050

TOPIC	KATAHDIN SOP CA-605-02	METHOD 3050, current revision
Apparatus /Materials	1) Digestion performed in 100 mL Griffin beaker. 2) Graduated disposable plastic cup used to bring digestate to final volume.	1) Digestion performed in 250 mL Griffin beaker. 2) Volumetric flask used to bring digestate to final volume.
Procedure	1) Digestate volume reduced to 5 to 10 mL prior to filtering. 2) Samples are not cooled after the initial addition of H <sub>2</sub> O <sub>2</sub> . 3) After filtration, the filters are rinsed three times with reagent water.	1) Digestate volume reduced to 5 mL prior to filtering. 2) Samples are cooled after the initial addition of H <sub>2</sub> O <sub>2</sub> . 3) After filtration, the filters are rinsed twice with reagent water.

**TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS  
 ANALYSIS BY ICP-AES AND ICP-MS**

TABLE 3

PREPARATION OF MATRIX SPIKES AND SPIKING SOLUTIONS FOR DIGESTION OF SOLID  
 SAMPLES BY USEPA METHOD 3050

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
<b>Matrix Spike for ICP-AES</b>	CLPP-SPK-1	Inorganic Ventures	0.10
	CLPP-SPK-INT1	Lab Prepared (see below)	1.00
	CLPP-SPK-INT2	Lab Prepared (see below)	1.00

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
<b>CLPP-SPK-INT1</b>	QCP-CICV-3	Inorganic Ventures	10.0
	1000 mg/L Sb	High Purity Standards	5.0
	10000 mg/L K	High Purity Standards	10.0
	10000 mg/L Na	High Purity Standards	7.5
	10000 mg/L Mg	High Purity Standards	5.0
	10000 mg/L Ca	High Purity Standards	2.5
<b>CLPP-SPK-INT2</b>	2007ICS-1	Inorganic Ventures	10.0
	1000 mg/L Sr	High Purity Standards	5.0
	1000 mg/L Sn	High Purity Standards	5.0
	10000 mg/L Si	High Purity Standards	5.0

**TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

TABLE 4

ELEMENT CONCENTRATIONS IN ICP-AES MATRIX SPIKES AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY METHOD 3050

Element	CONCENTRATION IN SOLUTION, mg/L						
	Matrix Spike (ICP-AES)	CLPP- SPK-1	CLPP- SPK-4	CLPP- SPK-INT1	CLPP- SPK-INT2	QCP-CICV-3 SPK-3	2007ICS-1
Aluminum	2.000	2000					
Antimony	0.500		100	50			
Arsenic	2.000		40	200		500	
Barium	2.000	2000					
Beryllium	0.050	50					
Boron	0.500				50		500
Cadmium	0.050		50	5		250	
Calcium	2.500			250			
Chromium	0.200	200					
Cobalt	0.500	500					
Copper	0.250	250					
Iron	1.000	1000					
Lead	0.500		20	50		500	
Magnesium	5.000			500			
Manganese	0.500	500					
Molybdenum	0.300				30		300
Nickel	0.500	500					
Potassium	25.000			2500			
Selenium	2.000		10	200		500	
Silicon	5.230				523		230
Silver	0.050	50					
Sodium	7.500			750			
Strontium	0.500				50		
Thallium	2.000		50	200		500	
Tin	0.500				50		
Titanium	1.000				100		1000
Vanadium	0.500	500					
Zinc	0.500	500					

---

**TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

---

FIGURE 1

PROCEDURE CONDENSATION – METHOD 3050

1. Prepare and print out ACCESS spreadsheet. Label plastic sample containers.
2. Rinse beakers and watch glasses 3 times in 10% HNO<sub>3</sub> bath.
3. Rinse beakers and watch glasses 3 times with reagent water.
4. Label beakers with sample numbers.
5. Weigh 1 to 2 g of well-mixed sample into tared beaker. Record sample weights.
6. Add spike solutions to matrix spike samples.
7. Add 10 mL 1:1 HNO<sub>3</sub> to samples and cover with watch glasses.
8. Reflux for 10 to 15 minutes at 95° ± 5° C. without boiling. Cool samples.
9. Add 5 mL conc. HNO<sub>3</sub>, cover beakers, and reflux for 30 minutes.
10. Repeat Step 9 as necessary until digestion is complete.
11. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
12. Cool sample and add 3 mL 30% H<sub>2</sub>O<sub>2</sub> and 2 mL reagent water. Heat gently until effervescence subsides.
14. Cool samples and add 7 mL of 30% H<sub>2</sub>O<sub>2</sub> in 1 mL aliquots. Heat gently until effervescence subsides.
15. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
16. Add 10 mL conc. HCl and reflux for 10 to 15 minutes at 95° ± 5° C.
17. Cool sample and filter into graduated specimen container. Bring to volume with reagent water and transfer to labeled polyethylene bottle.
18. Enter sample weights into ACCESS spreadsheet.

**TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

FIGURE 2

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK

Katahdin Analytical Services, Inc.

Metals Preparation Benchsheet

Reagent Information:

JT Baker HNO<sub>3</sub>: T31060 JT Baker HCL: T31035 Ashland H<sub>2</sub>O<sub>2</sub>: 004A008E

Method: 3050H

LCSS: E.R.A. Priority Pollutant/CLP Lot: S-824  
LOT # 243

Standards Information:

I.V. CLPP-SPK-1: 5895 0.1 ml

CLPP-SPK-INT1: MW5426 1.0

CLPP-SPK-INT2: MW5463 1.0

N/A

N/A Hot Plate No.: 6

Temp.: 99 °C

REVIEWED

Edm 12-8-00

KATAHDIN ANALYTICAL  
METALS SECTION

Spiking Witnessed by: WITNESS N/A

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Texture	Final Color	Final Clarity	Artifacts	Bottle
LCSSQL081CS0	QL081CS0	<u>0.50</u>	g	<u>0.1</u>	L	SL	IC	DCP	12/08/2000	N/A	N/A	N/A	N/A	<u>N/A</u>	<u>N/A</u>
PBSQL081CS0	QL081CS0	<u>1.00</u>	g		L	SL	IC	DCP	12/08/2000	N/A	N/A	N/A	N/A		
WQ3697-001	QL081CS0	<u>0.52</u>	g		L	FP	IC	DCP	12/08/2000	<u>N/A</u>	<u>N/A</u>	<u>N/A</u>	<u>N/A</u>		
WQ3697-002	QL081CS0	<u>0.59</u>	g		L	FP	IC	DCP	12/08/2000						
WQ3705-011	QL081CS0	<u>1.13</u>	g		L	SL	IC	DCP	12/08/2000						
WQ3705-021	QL081CS0	<u>1.03</u>	g		L	SL	IC	DCP	12/08/2000						
WQ3705-022	QL081CS0	<u>1.00</u>	g		L	SL	IC	DCP	12/08/2000						
WQ3719-003	QL081CS0	<u>1.17</u>	g		L	SL	IC	DCP	12/08/2000						
WQ3719-003P	QL081CS0	<u>1.20</u>	g		L	SL	IC	DCP	12/08/2000						
WQ3719-003S	QL081CS0	<u>1.19</u>	g		L	SL	IC	DCP	12/08/2000						
WQ3720-001	QL081CS0	<u>2.56</u>	g		L	FP	IC	DCP	12/08/2000						
WQ3728-001	QL081CS0	<u>1.02</u>	g		L	SL	IC	DCP	12/08/2000						
WQ3729-001	QL081CS0	<u>1.30</u>	g		L	SL	IC	DCP	12/08/2000						

DCP 12/8/00

DCP 12/8/00

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS

FIGURE 3

EXAMPLE PAGE FROM INTERNAL CUSTODY LOG FOR METALS DIGESTATES

Katahdin Analytical Services, Inc.  
Internal Custody Record for Metals Digestates

	Removed By			Returned By			Comments
	Initials	Date	Time	Initials	Date	Time	
QC BATCH ID: Q119ICW0	1/17/01	12-20-00	1140	Edm	1-2-01	8:20	<del>REMOVED</del>
Logged In By:							
Log-In Date:							
Log-In Time:							
QC BATCH ID: Q119IC50	1/17/01	12-20-00	1140	Edm	1-2-01	8:20	
Logged In By:							
Log-In Date:							
Log-In Time:							
QC BATCH ID: Q120ICW0	1/17/01	12-21-00	1130	Edm	1-2-01	8:20	
Logged In By:							
Log-In Date:							
Log-In Time:							
QC BATCH ID: Q120IC50	1/17/01	12-21-00	1130	Edm	1-2-01	8:20	
Logged In By:							
Log-In Date:							
Log-In Time:							

TITLE: **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND ICP-MS**

FIGURE 4

EXAMPLE CERTIFICATE OF ANALYSIS FOR SOLID REFERENCE MATERIAL



ENVIRONMENTAL  
RESOURCE ASSOCIATES  
ARVADA, COLORADO 1-800-372-0122

*Rec'd 12/08/00*

**Certification**

PriorityPollutnT™/CLP Inorganic Soils - Hot Plate Digestions

Quality Control Standards

Catalog No PPS-46

Lot No. 245

Parameter	Total Concentration <sup>1</sup>	Method 3050 ICP-OES/FLAA		Method 3050 ICP-MS/GFAA	
		Certified Value <sup>2</sup>	Performance Acceptance Limits™ <sup>3</sup>	Certified Value <sup>2</sup>	Performance Acceptance Limits™ <sup>3</sup>
TRACE METALS PriorityPollutnT™ (Catalog No 540)	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg
Aluminum	53800*	8830	3780 - 13900	9100	5980 - 12200
Antimony	198	68.9	18.8 - 119	8.10	D.L. - 27.7
Arsenic	152	136	101 - 171	136	101 - 171
Barium	894*	124	95.3 - 152	132	102 - 163
Beryllium	103	95.3	74.7 - 116	96.1	75.0 - 117
Boron	158	115	79.2 - 151	119	69.0 - 169
Cadmium	131	118	90.4 - 145	121	93.0 - 149
Calcium	16100*	11500	8590 - 14400	11600	8670 - 14500
Chromium	135	89.3	71.3 - 107	89.7	71.6 - 108
Cobalt	125	110	87.2 - 132	121	93.2 - 148
Copper	136	117	95.7 - 138	118	90.4 - 146
Iron	26400*	13700	8340 - 19100	11500	7000 - 16000
Lead	172	138	105 - 170	144	110 - 179
Magnesium	5520*	3040	2150 - 3930	3000	2420 - 3590
Manganese	520*	341	272 - 409	319	258 - 381
Mercury	2.60	2.48	1.69 - 3.27	2.48	1.69 - 3.27
Molybdenum	111	94.1	72.0 - 116	82.6	63.2 - 102
Nickel	186	156	122 - 190	161	117 - 204
Potassium	31000*	3430	2670 - 4190	3290	2570 - 4010
Selenium	105	87.6	64.9 - 110	95.0	70.4 - 120
Silver	122	119	88.8 - 150	87.1	47.3 - 127
Sodium	12100*	853	578 - 1130	809	549 - 1070
Strontium	294*	83.4	60.5 - 106	80.4	58.4 - 103
Thallium	154	139	79.6 - 199	144	82.4 - 205
Tin	117	96.3	72.1 - 121	29.8	13.3 - 46.3
Titanium	2900*	276	86.2 - 465	160	103 - 218
Vanadium	131	79.1	54.0 - 104	71.3	48.7 - 93.9
Zinc	100	66.0	42.9 - 89.1	72.1	49.2 - 95.0

CONTINUED ON BACK

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

Prepared By: George Brewer Date: 7/98

Approved By: \_\_\_\_\_

Group Supervisor: George Brewer Date: 01/23/01

Operations Manager: John C. Banta Date: 1/23/01

QA Officer: Deborah J. Nadeau Date: 1.23.01

General Manager: Deborah F. Nadeau Date: 1/23/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 6010B	Format changes, added pollution prevention, expanded procedure and QC sections. Added tables.	DN	1.23.01	1/23/01
02 6010B	Calibration begins with analysis of SO (cal. blank) followed by SI (Mixed Cal. Std.) Changes to SECTION 7.5 and Table 8 to reflect this. Made changes to element concs. in Tables 3, 4, 5, 6 to reflect current practices.	DN	10.21.02	10.21.02
03 6010B	Added MN-IEC to standards run. Changed frequency of LRS. Changed concentration of HNO <sub>3</sub> in calibration blank. CRI changed from three separate solutions to one. Changed CRI vendor.	MRC	04.15.04	04.15.04
04	updated ICV, CCV, ICB, PQL chk std. PBW, PBS, MS & MSD acceptance criteria updated Table 1	LAD	05/06	05/06

---

TITLE:           **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

---

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

---

I acknowledge receipt of copy \_\_\_ of document **SOP CA-608-04**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_ of document **SOP CA-608-04**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

TITLE:           **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

---

## **1.0 SCOPE AND APPLICATION**

Inductively coupled plasma atomic-emission spectroscopy (ICP-AES) determines trace elements, including metals, in solution. The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services, Inc. personnel to analyze aqueous and solid samples for trace metals by USEPA Method 6010 (Test Methods for Evaluating Solid Waste, Physical/ Chemical Methods, USEPA SW846).

Sample types that may be analyzed using these methods include drinking waters, ground waters, aqueous samples, TCLP, SPLP and EP Toxicity extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes. The following elements may be analyzed under this SOP: Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Si, Ag, Na, Sn, Sr, Tl, Ti, V, and Zn.

All samples, except filtered ground water samples, analyzed under USEPA Method 6010 require digestion prior to analysis. USEPA Methods 3005, 3010, and 3050 describe appropriate digestion procedures for samples to be analyzed by ICP-AES under EPA Method 6010. Refer to current revisions of Katahdin SOPs CA-604 and CA-605, current revisions, for sample digestion procedures.

### **1.1 Definitions**

Analytical Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

CRI - Contract Required detection limit sample for ICP - A low concentration standard used to verify calibration accuracy near the low end of the calibration range.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-AES - Inductively Coupled Plasma Atomic Emission Spectroscopy.

---

TITLE:           **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

---

ICS - Interference Check Sample - Two standards (ICSA and ICSAB) used to verify the effectiveness of interelement correction and background correction. Solution ICSA contains only interferences (Al, Ca, Fe, and Mg) at high concentrations (200 to 500 mg/L); solution ICSAB contains interferences at the same concentrations as well as analytes at low (20 mg/L or less) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LRS - Linear Range Standard - A high-concentration standard used to determine the upper reporting limit of the ICP calibration.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferences.

## 1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP analysis by EPA Method 6010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in ICP analysis by Method 6010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in

---

TITLE:           **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

---

the lab notebook and reported to the group supervisor or designated qualified data reviewer responsible for this data.

It is the responsibility of the Group Supervisor to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

### 1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Chemical Hygiene Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Safety glasses should be worn when changing or adjusting argon tanks.

### 1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes from ICP analysis should be disposed of in a manner appropriate to the hazards they present. Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Chemical Hygiene Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

---

TITLE:           **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

---

## **2.0   SUMMARY OF METHOD**

This method describes multielemental determinations by ICP-AES using simultaneous optical systems and radial or axial viewing of the plasma. The basis of the method is the measurement of atomic emission from sample atoms entrained in an argon plasma by optical spectroscopy. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where thermal excitation of entrained atoms and ions

occurs. Characteristic atomic-line and ionic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emitted lines are monitored by photomultiplier tubes. Photocurrents from the photomultiplier tubes are measured simultaneously by a computer system. Element concentrations of unknown samples are quantitated by comparison of sample emission intensities to emission intensities of standards of known concentration. A background correction technique is used to compensate for variable background contribution to the determination of trace elements. Background is measured adjacent to the analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, has been determined by the complexity of the spectrum adjacent to the analytical line. The position used must be relatively free of spectral interference and must reflect the same change in background intensity as occurs at the analyte wavelength. Physical interferences are corrected through the use of an internal standard (yttrium) that is automatically added to all samples and standards prior to nebulization. The possibility of additional interferences (noted in Section 3) must be recognized and appropriate corrections applied.

---

## **3.0   INTERFERENCES**

Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as spectral interferences, physical interferences, and chemical interferences.

Spectral interferences can be categorized as 1) overlap of a spectral line from another element; 2) unresolved overlap of molecular band spectra; 3) background contribution from continuous or recombination phenomena; and 4) background from stray light from the line emission of high concentration elements. The first of these effects is compensated by utilizing the computer correction of raw data, requiring the monitoring and measurement of the interfering element (interelement correction). The second effect is controlled by choosing analytical wavelengths that are free from overlapping molecular emission spectra. The third and fourth effects are usually compensated by a background correction adjacent to the analyte line. Uncorrected spectral interferences may be detected through examination of serial dilution and matrix spike data.

---

TITLE:           **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

---

Physical interferences are generally considered to be effects associated with sample nebulization and transport processes. Such properties as changes in viscosity and surface tension can cause significant inaccuracies, especially in samples that may contain high dissolved solids and/or acid concentrations. Matrix matching of standards and samples and the use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem that can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow rate causing instrumental drift. Regular cleaning of nebulizer tips and dilution of samples with high dissolved solids contents are used to control this problem. Physical interferences are also corrected by this laboratory through the use of an internal standard. Uncorrected physical interferences may be detected through examination of serial dilution and matrix spike data. Instrument drift caused by the salting up of nebulizer tips may also be detected by looking for oriented drift in calibration verification standards analyzed regularly throughout the run.

Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not pronounced with the ICP technique; however, if observed they can be minimized by careful selection of operating conditions (i.e., incident power, observation position, etc.), by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element. Uncorrected chemical interferences may be detected through examination of serial dilution data.

---

#### **4.0 APPARATUS AND MATERIALS**

- 4.1 Computer-controlled inductively-coupled plasma atomic emission spectrometer (plasma viewed radially or axially) equipped for internal standardization, and capable of performing automatic background correction and interelement correction.
- 4.2 Computer-controlled autosampler.
- 4.3 Argon gas supply – high purity.
- 4.4 Volumetric glassware of suitable precision and accuracy.
- 4.5 Automatic pipets of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

Refer to the appropriate instrument-specific SOP for additional required equipment.

---

TITLE:           **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

---

## **5.0 REAGENTS**

- 5.1 Hydrochloric acid, concentrated (HCl) – spectroscopic grade.
- 5.2 Nitric acid, concentrated (HNO<sub>3</sub>) – spectroscopic grade.
- 5.3 Reagent water, trace metals free.
- 5.4 Calibration blank – reagent water containing HCl (5% v/v) and HNO<sub>3</sub> (5% v/v). Calibration blank solution is prepared in large volumes (up to 20 liters) and stored in a carboy. Calibration blank solution is used in establishing the analytical curve, and in all initial and continuing calibration blank determinations. This solution is also used to flush the system between standards and samples. Intermediate and working standards are prepared by diluting stock standards and intermediate standards with calibration blank solution so that all standards and blanks are acid matrix-matched to sample digestates.
- 5.5 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals, and supplied by the vendors with certificates of purity and analysis. Refer to Tables 3 and 4 for a listing of stock standards required, and to Table 7 for element concentrations in stock standards.
- 5.6 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 4 for a listing of intermediate standards required and for preparation instructions. Refer to Table 6 for element concentrations in intermediate standards.
- 5.7 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument and to perform all necessary QC checks. Refer to Table 3 for a listing of working standards and for preparation instructions. Refer to Table 5 for element concentrations in working standards.
- 5.8 25 mg/L yttrium internal standard solution – add 2.5 mL 10000 mg/L yttrium stock standard (source: High Purity Standards) to a 1000 mL volumetric flask half filled with calibration blank solution. Bring to volume with calibration blank solution.

---

## **6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING**

Samples to be analyzed for trace metals by ICP should be collected and preserved as described in the following table.

---

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

---

Matrix	Container <sup>1</sup>	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO <sub>3</sub> to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO <sub>3</sub> to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months

<sup>1</sup> P = polyethylene or , G = glass

---

## 7.0 PROCEDURES

- 7.1 Background correction positions, dynamic linear ranges, and interelement correction factors must be established for each analytical wavelength used on each instrument prior to beginning sample analysis. Refer to Section 8 of this SOP and to the appropriate instrument operation SOP (current revisions of Katahdin SOPs CA-609 or CA-612).
- 7.2 Ignite the plasma and allow the instrument to aspirate calibration blank solution (refer to the appropriate instrument operation SOP). Wait 30 to 60 minutes for the instrument to become thermally stable before proceeding with analysis.
- 7.3 Profile the instrument as described in the appropriate instrument operation SOP.
- 7.4 Sample analysis will be performed through the use of an autosampler. Using the instructions given in the appropriate instrument operation SOP, prepare an autosampler table listing all standards and samples to be analyzed, in the appropriate order. Print out the autosampler table and fill the autosampler racks with tubes containing samples and standards in the positions listed in the autosampler table.
- 7.5 Initiate sample analysis using the autosampler, as described in the appropriate instrument operation SOP. Analysis must proceed in the sequence described in Table 8 to ensure that all necessary quality control samples are analyzed at the appropriate frequencies. A minimum of two replicate integrations is required for all standards and samples. Analysis always begins with a calibration blank solution (S0) followed by the analysis of a multielement calibration standard (S1 in Table 3) to calibrate the instrument. The system is flushed with calibration blank for two minutes between each sample and standard, and each sample and standard is aspirated for one minute prior to the beginning of emission measurements.

---

TITLE:           **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

---

- 7.6 Calibration is followed by analysis of interelement correction standards, which are used to update the interelement correction (IEC) factors prior to continuing with analysis. The solution "AL\_IEC" contains 500 mg/L aluminum, and the IEC factors for aluminum are automatically updated after analysis of this solution. The solution "FE\_IEC" contains 200 mg/L iron, and the IEC factors for iron are automatically updated after analysis of this solution. The solution MN\_IEC contains 20 mg/L Manganese and the IEC factors for manganese are automatically updated after analysis of this solution.
- 7.7 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.
- 7.8 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples, and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.
- 7.9 Interference check standard solutions ICSA and ICSAB must be analyzed at the beginning of each run to verify the accuracy of the IEC factors. Refer to Section 8 and Table 1 for additional information.
- 7.10 A practical quantitation limit standard (PQL) must be analyzed at the beginning of each run to determine the accuracy of the calibration at the reporting limit. Refer to Section 8 and Table 1 for additional information.
- 7.11 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a QC sample (ICV, ICB, CCV, CCB, ICSA, or ICSAB) for that element must not be reported. The sample must be reanalyzed for the element in question.
- 7.12 All samples that exceed the linear calibration range must be diluted and reanalyzed. This includes samples with interfering elements that exceed the calibration ranges, because accurate quantitation of interfering elements is necessary for reliable interelement correction. For example, if a sample has been submitted to the laboratory for lead analysis, and the measured aluminum concentration of that sample exceeds the calibration range for aluminum, it must be diluted sufficiently to bring aluminum within the linear calibration range and the lead result must be reported from that dilution analysis.
- 7.13 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is accomplished

**TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

---

automatically by entering the dilution factor in the autosampler table prior to initiation of analysis.

- 7.14 All analyses are performed using yttrium as an internal standard to compensate for enhancement or depression of the analytical signal due to matrix effects. Yttrium solution is pumped at a constant rate through one channel of the peristaltic pump. Samples and standards are pumped through a second channel of the pump. The tubing carrying the internal standard is connected to the tubing carrying samples and standards downstream from the pump, and mixing of the two streams is accomplished in a mixing coil downstream from the connection, prior to nebulization.

For each sample or standard, the computer that controls the spectrometer divides the detected emission signal for each element by the detected yttrium emission signal prior to quantitation, thus normalizing all emission signals to that of yttrium.

---

## **8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA**

USEPA Method 6010 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are

described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new

reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

### **INITIAL DEMONSTRATION OF PERFORMANCE**

- 8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of a reagent water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on

---

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

---

performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

- 8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of a reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the seven replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.
- 8.3 The upper limit of the linear dynamic range (LDR) must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing succeeding higher standard concentrations of the analyte until the observed analyte concentration is no more than 10% below the stated concentration of the standard. Determined LDRs must be documented and kept on file. The LDR which may be used for the analyses of samples should be judged by the analyst from the resulting data. Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified annually or whenever, in the judgement of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.
- 8.4 The alkali and alkaline earth metals may have non-linear response curves due to ionization and self-absorption effects. These curves may be used for quantitation of samples if the effective range is checked and if the second order curve fit has a correlation coefficient of 0.995 or better. Third order fits are not acceptable. Non-linear response curves must be revalidated and recalculated every six months.

#### ANALYTICAL RUN QC SAMPLES

- 8.5 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared by combining compatible elements from a standard source different than that of the calibration standard and at concentrations within the linear working range of the instrument. The results of the ICV must fall within 90% to 110% of the expected values, with relative standard deviation <5% from replicate integrations. If the ICV fails, result for the failing elements may not be reported from the run unless the ICV recovery is greater than 110% and the sample result is less than the PQL.

---

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

---

- 8.6 Continuing Calibration Verification (CCV) solutions are analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standards used for calibration at concentrations near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results for the failing elements may not be reported from the run unless the CCV recovery is greater than 110% and the sample result is less than the PQL. Also, for failing elements, all samples analyzed after the last passing CCV must be reanalyzed.
- 8.7 Calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for a CCB a ICB is greater than the PQL, sample results that are less than the PQL or greater than or equal to ten times the measured CCB concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.
- 8.8 Interference check solutions ICSA and ICSAB (refer to Section 1.1) are analyzed at the beginning of each run to verify interelement correction factors and background correction. ICSA contains interferent elements (Al, Ca, Fe, and Mg) only, at concentrations of 200 mg/L to 500 mg/L. Results for interfering elements in the ICSA must fall within 80% to 120% of the expected values. Results for unspiked elements in ICSA must fall within  $\pm$  PQL if the PQL is greater than 0.01 mg/L, within  $\pm 2xPQL$  if the PQL is less than or equal to 0.01 mg/L. ICSAB contains interferent elements at concentrations of 200 mg/L to 500 mg/L, and analytes at concentrations of 20 mg/L or less. Results for all elements (interferents and analytes) in ICSAB must fall within 80% to 120% of the expected values. If the ICSA or ICSAB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICSA or ICSAB has been analyzed.
- 8.9 A Practical Quantitation Limit (PQL) Check Standard is analyzed at the beginning of each run, after the ICV and ICB samples. Element concentrations in this solution are at the laboratories practical quantitation

---

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

---

limit. Element recoveries for the PQL check Standard must fall between 50-150% of the expected values. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run, unless the PQL Check Standard recovery is greater than 150% and the samples results are less than the PQL.

#### PREPARATION BATCH QC SAMPLES

- 8.10 Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spike sample or matrix spike sample duplicate.
- 8.11 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL, associated sample results that are less than the PQL or greater than or equal to ten times the measured preparation blank concentration may be reported.
- 8.12 A laboratory control sample (LCS), consisting of spiked reagent water or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested with the following exception. If the LCS fails high, samples results less than the PQL may be reported.

#### SAMPLE MATRIX QC SAMPLES

- 8.13 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, the associated sample result must be flagged on the report of analysis.

**TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

The relative percent difference between sample duplicate, matrix spiked duplicate or LCS duplicate, is calculated as follows:

$$\text{RPD (\%)} = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where:

D<sub>1</sub> = sample result

D<sub>2</sub> = duplicate sample result

A control limit of 20% RPD is applied to duplicate analysis if the original sample result is greater than 50X the IDL. If the matrix spike duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

- 8.12 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$\text{Difference (\%)} = \frac{|L-S|}{S} * 100\%$$

where:

L = Serial dilution result (corrected for dilution)

S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

**9.0 METHOD PERFORMANCE**

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Metals Supervisor and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the current revision of Method 6010 for other method performance parameters and requirements.

TITLE:           **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

---

## **10.0 APPLICABLE DOCUMENTS/REFERENCES**

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846 Updates I, II, IIA, and IIB, Revised December 1996., Method 6010B.

---

### List of Tables & Figures

Table 1	QC Requirements
Table 2	Summary of Method Modifications
Table 3	Preparation of Calibration and Quality Control Standards
Table 4	Preparation of Intermediate Standards
Table 5	Element Concentrations in Working Standards
Table 6	Element Concentrations in Intermediate Standards
Table 7	Element Concentrations in Stock Standards
Table 8	Required Analytical Sequence

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

TABLE 1  
QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010	Initial Calibration, minimum 1 point plus a calibration blank.	Daily prior to sample analysis.		
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within $\pm$ 10% of true value, and RSD < 5%.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL.	1) Do not use results if $\geq$ PQL and < 10x CCB level. 2) Investigate and correct problem.
	Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within $\pm$ 10% of true value, and RSD < 5%.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Continuing Calibration Blank (CCB)	After every 10 samples and at end of the run.	Absolute value of CCB < PQL.	1) Do not use results if $\geq$ PQL and < 10x CCB level. 2) Investigate and correct problem.
	CRDL Standard for ICP (CRI)	At the beginning of a sample run, after every 20 samples, and at the end of the run.	Recovery within 50% - 150% of true value.	Terminate analysis, correct problem, recalibrate, and reanalyze all analytical samples analyzed since last good CRI.
	Practical Quantitation Level Check Standard (PQL)	At beginning or run.	Recovery within $\pm$ 50% of true value.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Interference Check Solution A (ICSA)	Before beginning a sample run.	For Al, Ca, Fe, and Mg, recovery within $\pm$ 20% of true value. For analytes not spiked, $\pm$ PQL, or, if PQL < 0.01 mg/L, $\pm$ 2x PQL.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Interference Check Solution AB (ICSAB)	Before beginning a sample run.	Recovery of each analyte within $\pm$ 20% of true value.	1) Do not use results for failing elements. 2) Investigate and correct problem.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1, CONTINUED

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010, continued	Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration $\geq$ PQL and $<10x$ the blank concentration.
	Laboratory Control Sample (LCSW/LCSS)	One per digestion batch of 20 or fewer samples.	Recovery within $\pm 20\%$ of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery $\pm 25\%$ of true value, if sample $< 4x$ spike added.	Flag results.
	Matrix Spike Duplicate Sample (P)	One per digestion batch of 20 or fewer samples.	1) Recovery $\pm 25\%$ of true value, if sample $< 4x$ spike added. 2) RPD $\leq 20\%$ for duplicate spikes.	Flag results
	Serial Dilution (L)	One per digestion batch.	If original sample result is at least $50x$ IDL, 5-fold dilution must agree within $\pm 10\%$ of the original result.	Flag result or dilute and reanalyzed sample to eliminate interference.
	Instrument Detection Limit (IDL) Study	Quarterly.	PQL $> 2-3 * \text{the IDL}$	1) Repeat IDL study. 2) Raise PQL.
	Method Detection Limit (MDL) Study	Annually.	M PQL $> 2-3 * \text{the MDL}$	1) Repeat MDL study. 2) Raise PQL.
	Linear Range Study	Annually .	Run succeedingly higher stds until recovery <u>not</u> within $\pm 10\%$ . Use highest passing concentration as upper limit of linear range.	Only accept data to highest passing concentration until next linear range study.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 2  
 SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-608-04	METHOD 6010, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6010: $\pm$ PQL	Acceptance criteria stated in 6010: mean blank value $\pm$ 3 standard deviations

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 3

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Calibration Standard (STD1 or S1) (add 0.6 mL conc. HCl to maintain acid strength)	ICP-MIX-A	Lab Prepared (see Table 4)	4.0
	ICP-MIX-B	Lab Prepared (see Table 4)	4.0
	CLPP-ICS-A	Inorganic Ventures	2.0
	10000 mg/L K	High Purity Standards	1.0
	10000 mg/L Na	High Purity Standards	1.0
	10000 mg/L Si	High Purity Standards	0.2
	1000 mg/L Ag	High Purity Standards	0.05
Initial Calibration Verification (ICV)	CLPP-ICS-B4	Inorganic Ventures	1.0
	1000 mg/L Si	Inorganic Ventures	1.0
	10000 mg/L K	Inorganic Ventures	0.1
	10000 mg/L Na	Inorganic Ventures	0.2
	10000 mg/L Al	High Purity Standards	0.2
	10000 mg/L Ca	High Purity Standards	0.2
	10000 mg/L Fe	High Purity Standards	0.2
	10000 mg/L Mg	High Purity Standards	0.2
	2007ICS-1	Inorganic Ventures	0.1
	QCP-CICV-3	Inorganic Ventures	0.1
	1000 mg/L Sn	Inorganic Ventures	0.05
1000 mg/L Sr	Inorganic Ventures	0.05	
Practical Quantitation Limit Sample (PQL)	PQL-INT	Lab Prepared (see Table 4)	1.0
AL_IEC	10000 mg/L Al	Inorganic Ventures	5.0
	10000 mg/L Ca	Inorganic Ventures	5.0
	10000 mg/L Mg	Inorganic Ventures	5.0
FE_IEC	10000 mg/L Fe	Inorganic Ventures	2.0
	10000 mg/L Ca	Inorganic Ventures	5.0
MN_IEC	1000 mg/L Mn	High-Purity Standards	2.0
Contract Required Detection Limit Standard for ICP (CRI)	CRDL	High Purity Standards	0.1
Interference Check Solution A (ICSA)	CLPP-ICS-A	Inorganic Ventures	10.0
Interference Check Solution AB (ICSAB)	CLPP-ICS-A	Inorganic Ventures	10.0
	CLPP-ICS-B4	Inorganic Ventures	1.0
	ICSAB-INT	Lab Prepared (see Table 4)	5.0

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 3, cont'd

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Continuing Calibration Verification (CCV)	ICP-MIX-A	Lab Prepared (see Table 4)	2.0
	ICP-MIX-B	Lab Prepared (see Table 4)	2.0
	CLPP-ICS-A	Inorganic Ventures	1.0
	10000 mg/L K	High Purity Standards	0.5
	10000 mg/L Na	High Purity Standards	0.5
	1000 mg/L Ag	High Purity Standards	0.025
	10000 mg/L Si	High Purity Standards	0.1

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 4  
PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
ICP-MIX-A	1000 mg/L Sb,As,Cd	High Purity Standards	5.0 each
	1000 mg/L Pb,Mo,Se	High Purity Standards	5.0 each
	1000 mg/L Sr,Ti,Ti	High Purity Standards	5.0 each
ICP-MIX-B	1000 mg/L Ba,Be,B	High Purity Standards	5.0 each
	1000 mg/L Cr,Co,Cu	High Purity Standards	5.0 each
	1000 mg/L Mn,Ni,Sn	High Purity Standards	5.0 each
	1000 mg/L V,Zn	High Purity Standards	5.0 each
PQL-INT	1000 mg/L B,Mo,Sn,Sr	High Purity or Inorganic Ventures	1.0 each
	10000 mg/L K	High Purity or Inorganic Ventures	1.0
	1000 mg/L Ni	High Purity Standards	0.4
	1000 mg/L Co	High Purity Standards	0.3
	1000 mg/L Cu,V,Zn	High Purity Standards	0.25 each
	10000 mg/L Si	High Purity Standards	0.2
	1000 mg/L Cr,Ti,Ti,Ag	High Purity Standards	0.15 each
	1000 mg/L Cd,Se	High Purity Standards	0.1 each
	10000 mg/L Al	High Purity Standards	0.3
	10000 mg/L Na	High Purity or Inorganic Ventures	1.0
	1000 mg/L As,Sb	High Purity Standards	0.08 each
	1000 mg/L Ba,Be,Mn,Pb	High Purity Standards	0.05 each
	10000 mg/L Ca,Mg	High Purity Standards	0.05 each
10000 mg/L Fe	High Purity Standards	0.1	
ICSAB-INT	10000 mg/L K, Na	High Purity or Inorganic Ventures	4.0 each
	10000 mg/L B,Mo,Sr,Sn,Ti	High Purity Standards	1.0 each
	10000 mg/L Si	High Purity Standards	0.4

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 5  
 ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L									
	STD1	ICV	PQL	AL_IEC	FE_IEC	MN_IEC	ICSA	ICSAB	CCV	CRI
Aluminum	100	50	0.3	500			500	500	50	
Antimony	2	0.6	0.008					0.6	1.0	0.12
Arsenic	2	0.6	0.008					0.1	1.0	0.02
Barium	2	0.5	0.005					0.5	1.0	
Beryllium	2	0.5	0.005					0.5	1.0	0.01
Boron	2	0.5	0.1					0.5	1.0	
Cadmium	2	1.25	0.01					1.0	1.0	0.01
Calcium	2	50	0.05	500	500		500	500	50	
Chromium	2	0.5	0.015					0.5	1.0	0.02
Cobalt	2	0.5	0.03					0.5	1.0	0.1
Copper	2	0.5	0.025					0.5	1.0	0.05
Iron	40	50	0.1		200		200	200	20	
Lead	2	0.55	0.005					0.05	1.0	0.006
Magnesium	100	50	0.05	500			500	500	50	
Manganese	2	0.5	0.005			20		0.5	1.0	0.03
Molybdenum	2	0.3	0.1					0.5	1.0	
Nickel	2	1.0	0.04					1.0	1.0	0.08
Potassium	100	50	1.0					20	50	
Selenium	2	0.55	0.01					0.05	1.0	0.01
Silicon	20	10.23	0.2					10	10	
Silver	0.5	0.2	0.015					0.2	0.25	0.02
Sodium	100	50	1.0					20	50	
Strontium	2	0.5	0.1					0.5	1.0	
Thallium	2	0.6	0.015					0.1	1.0	0.02
Tin	2	0.5	0.1					0.5	1.0	
Titanium	2	1.0	0.015					0.5	1.0	
Vanadium	2	0.5	0.025					0.5	1.0	0.1
Zinc	2	1.0	0.025					1.0	1.0	0.04

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

TABLE 6

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L			
	ICP-MIX-A	ICP-MIX-B	PQL-INT	ICSAB-INT
Aluminum			30	
Antimony	50		0.8	
Arsenic	50		0.8	
Barium		50	0.5	
Beryllium		50	0.5	
Boron		50	10	10
Cadmium	50		1.0	
Calcium			5.0	
Chromium		50	1.5	
Cobalt		50	3.0	
Copper		50	2.5	
Iron			10	
Lead	50		0.5	
Magnesium			5.0	
Manganese		50	0.5	
Molybdenum	50		10	10
Nickel		50	4.0	
Potassium			100	400
Selenium	50		1.0	
Silicon			20	40
Silver			1.5	
Sodium			100	40
Strontium	50		10	10
Thallium	50		1.5	
Tin		50	10	10
Titanium	50		1.5	10
Vanadium		50	2.5	
Zinc		50	2.5	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 7  
 ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L				
	2007 ICS-1	CLPP-ICS-A	CLPP-ICS-B4	CRDL	QCP-CICV-3
Aluminum		5000			
Antimony			60	120	
Arsenic			10	20	500
Barium			50		
Beryllium			50	10	
Boron	500				
Cadmium			100	10	250
Calcium		5000			
Chromium			50	20	
Cobalt			50	100	
Copper			50	50	
Iron		2000			
Lead			5	6	500
Magnesium		5000			
Manganese			50	30	
Molybdenum	300				
Nickel			100	80	
Potassium					
Selenium			5	10	500
Silicon	230				
Silver			20	20	
Sodium					
Strontium					
Thallium			10	20	500
Tin					
Titanium	1000				
Vanadium			50	100	
Zinc			100	40	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 8  
REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	S0 (Calibration Blank)	Initial calibration
2	S1 (Calibration Standard)	Initial calibration
3	AL_IEC	Update IECs for aluminum
4	FE_IEC	Update IECs for iron
5	MN_IEC	Update IECs for manganese
6	ICV (Initial Calibration Verification)	Check calibration accuracy
7	ICB (Initial Calibration Blank)	Check calibration accuracy
8	CRI (Contract Required Detection Limit Standard for ICP)	Check calibration accuracy near CRDL
9	PQL (Practical Quantitation Level Sample)	Check calibration accuracy near PQL
10	ICSA (Interference Check Solution A)	Verify accuracy of IEC factors
11	ICSAB (Interference Check Solution AB)	Verify accuracy of IEC factors
12	CCV (Continuing Calibration Verification)	Check calibration stability
13	CCB (Continuing Calibration Blank)	Check calibration stability
14 – 23	Analyze up to 10 samples	
24	CCV (Continuing Calibration Verification)	Check calibration stability
25	CCB (Continuing Calibration Blank)	Check calibration stability
...	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	

TITLE: ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP ANALYSIS OF TOTAL OR DISSOLVED METALS

Prepared By: George Brewer Date: 11/97

Approved By:

Group Supervisor: George Brewer Date: 01/19/01

Operations Manager: John C. Brunton Date: 1/22/01

QA Officer: Deborah J. Madreau Date: 1-22-01

General Manager: Dennise F. Keenan Date: 1/22/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3010A	Format changes, added pollution prevention, block digester; revised database references; revised and added tables.	DN	1-22-01	1/22/01
02 3010A	Added wording allowing use of digestates for ICP-MS analysis. Added use of block digester as primary heating source & adjusted volumes. Revised standard solution names & concs. in Figures 3 & 4.	DN	8-29-02	8-29-02
03	Added Uranium to spiking solutions for LCS & MS/D. Removed the Internal Custody Record for Metals Digestates figure and reference.	LAD	04/06	04/06

---

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

---

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

---

I acknowledge receipt of copy \_\_\_\_ of document **SOP CA-604-03**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_\_ of document **SOP CA-604-03**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

**TITLE: ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

---

## **1.0 SCOPE AND APPLICATION**

The purpose of this SOP is to describe the procedure utilized by Katahdin Analytical Services, Inc. personnel to solubilize metals in aqueous samples, wastes that contain suspended solids, and mobility-procedure extracts prior to analysis by inductively coupled plasma atomic emission spectroscopy (ICP) and inductively coupled plasma mass spectrometry (ICP-MS). This SOP applies to samples prepared by EPA Method 3010, with the method modifications mentioned in Table 2.

1.1 Definitions - none.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of aqueous samples by EPA Method 3010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of aqueous samples using EPA Method 3010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their work in the appropriate lab notebook. Any deviations from the method or irregularities with the samples should also be recorded in the lab notebook and reported to the Supervisor or designated qualified data reviewer responsible for these data.

It is the responsibility of the Supervisor to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, rubber aprons, dust masks, and rubber shoe protectors, is available in the metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

---

**TITLE: ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

---

Hood sashes should be lowered as far as possible whenever beakers are being heated in the hood. Use caution when handling hot beakers.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Chemical Hygiene Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

**1.4 Pollution Prevention/Waste Disposal**

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Any other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Chemical Hygiene Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision.

---

**2.0 SUMMARY OF METHOD**

The aqueous sample is refluxed with nitric acid in a covered digestion vessel. Additional nitric acid is added until the color of the digestate has stabilized. After the digestate has been evaporated to a low volume, it is refluxed with hydrochloric acid and diluted to the appropriate final volume with reagent water.

Samples may be concentrated (i.e. final digestate volume less than initial sample volume) during digestion if lower detection limits are required. Volumes of reagents and spiking standards must be added in proportion to the final volume of the digestate. Because

---

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

---

concentration of samples during digestion increases the concentrations of dissolved solids and may exacerbate analytical interferences, concentration factors greater than 5 are not recommended.

---

### **3.0 INTERFERENCES**

Interferences are discussed in the applicable analytical SOPs.

---

### **4.0 APPARATUS AND MATERIALS**

- 4.1. 250 mL and 400 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning") for digestion using a hot plate. If digestion will be performed using a block digester, 70ml graduated, polyethylene block digester tubes (with attached snap caps) will be used instead of glass beakers.
- 4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter and 100 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40mm diameter disposable polyethylene watch glasses are used.
- 4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.
- 4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.
- 4.5 Hot plate, block digester, or other heating source - adjustable and capable of maintaining a temperature of 90-95<sup>o</sup>C. Hot plates must be numbered for easy identification.
- 4.6 Device for measuring hot plate temperature. This may consist of a heat-resistant 100ml beaker containing reagent water in which a thermometer is immersed. When using a block digester, a digestion tube containing reagent water in which a thermometer is immersed may be used. The temperature of one hot plate is measured each day, on a rotating basis. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.
- 4.7 Plastic funnels, pre-cleaned as in Section 4.1.

---

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

---

- 4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.
- 4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO<sub>3</sub>.
- 4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO<sub>3</sub>, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.
- 4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity. These are not necessary when using the block digester since the final digestates are stored in the digestion tubes.
- 4.12 Repipettors (adjustable repeating pipettors with reservoirs) for dispensing concentrated nitric acid and 1:1 HCl.

---

**5.0 REAGENTS**

- 5.1 Concentrated nitric acid, HNO<sub>3</sub> – trace metals grade.
- 5.2 Concentrated hydrochloric acid, HCl – trace metals grade.
- 5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).
- 5.4 Hydrochloric acid, 1:1. Add a volume of concentrated hydrochloric acid to an equivalent volume of reagent water and swirl gently to mix.
- 5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO<sub>3</sub> to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.
- 5.6 Multi-element spiking solutions (as listed in Figure 3).

---

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

---

## **6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING**

Samples to be analyzed for dissolved metals should be filtered through a 0.45 um membrane filter and preserved as soon as possible after collection. Samples to be analyzed for total metals should be preserved, unfiltered, as soon as possible after collection. Aqueous samples are preserved by acidification with nitric acid to a pH of <2.

---

## **7.0 PROCEDURES**

- 7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet and print labels for the digestate containers. Print out a copy of the spreadsheet. Attach labels to the polyethylene sample containers that will contain the digestates.
- 7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digester do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.
- 7.3 If digestion is performed using a block digester, the sample aliquot may be measured in the digestion vessel using the graduations on the digestion tubes. Measure 50 ml of well-mixed sample into a 70 ml block digestion tube. A larger sample aliquot may be used (up to 250 mL) if concentration of the sample during digestion is desired. Sample volumes larger than 50 mL may be digested in 250 mL beakers. Measure aliquot of well-mixed sample into a graduated specimen cup and transfer into a properly cleaned 250 mL beaker. Sample volumes of more than 50ml may not be digested using the 70ml block digester tubes. The volumes of reagents and spiking solutions used must be adjusted in proportion to the final digestate volume. The reagent and spiking solution volumes listed below are based on a final volume of 50 mL.
- 7.4 Add spike solutions to matrix spike samples and laboratory control samples (refer to Figure 3 for spiking instructions).
- 7.5 Use a repipetter to add 1.5 mL of concentrated HNO<sub>3</sub> (per 50 mL final volume) to the sample. Cover with a ribbed watch glass and place on heatsource. Heat cautiously, without boiling the sample, and evaporate to a low volume (10 - 15 mL).

**NOTE:** Do not allow any portion of the bottom of the digestion vessel to go dry during any part of the digestion. If a sample is allowed to go to dryness, low

---

**TITLE: ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

---

recoveries may result. Should this occur, discard the digestate and re-prepare the sample.

- 7.6 Cool the sample and add another 1.5 mL aliquot (per 50 mL final volume) of concentrated HNO<sub>3</sub>. Cover and resume heating, increasing the temperature until a gentle reflux action occurs.
- 7.7 Continue heating, adding additional acid as necessary, until the digestate is light in color or does not change in appearance with continued refluxing.
- 7.8 Evaporate digestate to a low volume (10 - 15 mL).
- 7.9 Cool the sample and use a repipetter to add 5 mL (per 50 mL final volume) of 1:1 HCl. Cover the sample and resume heating, refluxing for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.
- 7.10 Allow the sample to cool.
- 7.11 If the digestate contains visible particulate material, it must be filtered. Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated plastic specimen container or block digester digestion tube. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container or digestion tube, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse.  
  
If the digestion was performed using hot plates and the digestate does not contain particulate material, simply decant the digestate into a clean graduated specimen container (or graduated sample container with attached snap lid), rinse the beaker with reagent water, and add the rinsates to the container.  
  
If the digestion was performed using a block digester and the digestate contains no visible particulate material, the digestate may be brought to final volume and stored in the digestion tube without decanting or rinsing.
- 7.12 Using the graduations on the specimen container, snap-lid container or digestion tube, dilute to the required final volume with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container or digestion tube has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for analysis.
- 7.13 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a

---

**TITLE: ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

---

corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) reagent lot numbers, spiking information, initial and final volumes, and hot plate temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 1.

- 7.14 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.
- 7.15 A condensation of the procedure described above is included in this SOP as Table 3. A controlled copy of this table may be posted in the metals preparation laboratory for reference by the analyst.

---

**8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA**

- 8.1 At least one preparation blank for waters (PBW) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBW consists of an aliquot of reagent water that is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the PBW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the PBW must also be concentrated). Refer to the appropriate analytical SOP for PBW acceptance criteria and corrective actions.
- 8.2 At least one laboratory control sample for waters (LCSW) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSW consists of an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations, and is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the LCSW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the LCSW must also be concentrated). Directions for spiking the LCSW are contained in Figures 3 and 4. The measured analyte recoveries for the LCSW are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSW recovery acceptance criteria and corrective actions.
- 8.3 Matrix spiked samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is spiked with known amounts of all analytes of interest. Matrix spike recoveries are used to assess the effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are

---

**TITLE: ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

---

contained in Figures 3 and 4. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

- 8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

NOTE: Clients may choose specific samples for matrix spike and matrix spike duplicate analysis; otherwise, the choice is left to the person performing the digestion. The sample volumes available may restrict the choice of samples used for matrix spike and duplicate digestion. Field blank samples should not be chosen for matrix spike and matrix spike duplicate analysis.

- 8.5 The quality control measures and frequencies described above are minimum requirements. They are summarized for reference in Table 1. Individual clients and analytical programs may impose additional QC requirements.

---

**9.0 METHOD PERFORMANCE**

Refer to the applicable analytical SOPs for method performance information.

---

**10.0 APPLICABLE DOCUMENTS/REFERENCES**

"Test Methods for the Evaluation of Solid Waste," United States Environmental Protection Agency, SW-846, Third Edition, Final Update III, 12/96, Method 3010A.

---

**LIST OF TABLES AND FIGURES**

Table 1	QC Requirements
Table 2	Summary of Method Modifications
Table 3	Procedure Condensation
Figure 1	Example Page From Metals Sample Preparation Logbook
Figure 2	Preparation of Matrix Spikes, LCSs, and Spiking Solutions: Method 3010
Figure 3	Element Concentrations in Matrix Spikes, LCSs, and Spiking Solutions: Method 3010

**TITLE: ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 1  
 QC REQUIREMENTS

Analytical Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3010	Preparation Blank for Waters (PBW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Laboratory Control Sample for Waters (LCSW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Matrix Spike Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Matrix Spike Duplicate Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 2  
 SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-604-03	EPA METHOD 3010, current revision
Apparatus/Materials	1) Disposable plastic specimen cup used to measure sample volume.  2) Digestion performed in 250 mL, 400 mL Griffin beaker, or 70ml digestion tube to facilitate evaporation.  3) Ribbed watch glass used throughout digestion to reduce contamination.	1) Graduated cylinder used to measure sample volume.  2) Digestion performed in 150 mL Griffin beaker.  3) Ribbed and non-ribbed watch glasses alternated in digestion.
Procedures	1) Digestate may be analyzed for antimony and silver.  2) Sample aliquots larger or smaller than 100 mL may be used.  3) Sample evaporated to 10 - 15 mL.	1) Digestate may not be analyzed for antimony and silver.  2) Requires sample aliquot of 100 mL.  3) Sample evaporated to 5 mL.

---

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

---

TABLE 3

PROCEDURE CONDENSATION: EPA METHOD 3010

1. If performing digestion on a hot plate, rinse glass beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with reagent water. If performing digestion with block digester, polyethylene digestion tubes do not require precleaning.
3. Label digestion vessels with sample numbers.
4. Mix sample well, measure 50 mL (or smaller or larger aliquot) into a polyethylene digestion tube. If using glass beakers, measure aliquot into graduated specimen container, and transfer to appropriate digestion vessel.
5. Add spike solutions to matrix spike samples and LCSW (refer to Figure 3 of this SOP).
6. Add 1.5 mL (per 50 mL final volume) concentrated  $\text{HNO}_3$  to sample.
7. Cover with a ribbed watch glass.
8. Place on heating device (hotplate or block digester) and evaporate to 10 - 15 mL.
9. Cool sample and add another 1.5 mL (per 50 mL final volume) concentrated  $\text{HNO}_3$ .
10. Resume heating until gentle reflux action occurs.
11. Continue heating, adding additional  $\text{HNO}_3$  as necessary until digestion is complete.
12. Evaporate to 10 - 15 mL.
13. Cool sample and add 5 mL (per 50 mL final volume) 1:1 HCl. Resume heating and reflux gently for 15 minutes.
14. Cool sample and filter (if necessary) or decant into a graduated polyethylene digestion tube. Rinse beaker with reagent water and filter or decant rinsate into specimen container.
13. Dilute to appropriate final volume with reagent water.
14. Cap sample container and shake gently to mix.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 1

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK

Katahdin Analytical Services, Inc. Metals Preparation Benchsheet

Reagent Information: JT Baker HNO<sub>3</sub>: C20022 JT Baker HCL: C07046 Ashland H<sub>2</sub>O<sub>2</sub>: NA Method: 3010

Standards/Spiking Information: I.V. CLPP-SPK-1 (ID/Vol): MS1362 10.05 ml N/A : N/A  
 CLPP-SPK-INT1 (ID/Vol): MW9925 10.5 ml Hot Plate No.: A  
 CLPP-SPK-INT2 (ID/Vol): MW9930 10.5 ml Temp.: 95 °C  
C-5 Spike (ID/Vol): MS1532 10.05 ml

Spiking Witnessed by: N/A

REVIEWED  
En 06-27-06  
KATAHDIN ANALYTICAL  
METALS SECTION

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LC2WWF271CW1	WF271CW1	0.05	L	0.05	L	AQ	IC	DJJ	06/27/2006	N/A	N/A	N/A	N/A	N/A	N/A
LCSWWF271CW1	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006	N/A	N/A	N/A	N/A		
PBWFF271CW1	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006	N/A	N/A	N/A	N/A		
WW3165-001	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-002	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-003	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-004	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-005	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-006	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-007	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-008	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-009	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-010	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-011	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-012	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-013	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-014	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-015	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-016	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-017	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-018	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-019	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-020	WF271CW1		L		L	AQ	IC	DJJ	06/27/2006						

DJJ 6/27/06

**TITLE: ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 2

PREPARATION OF MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 50 mL Final Volume (mL)
<b>Laboratory Control Sample (LCSW) and Matrix Spike</b>	CLPP-SPK-1	Inorganic Ventures	0.050
	CLPP-SPK-INT1	Lab Prepared (see below)	0.50
	CLPP-SPK-INT2	Lab Prepared (see below)	0.50
	1000 mg/L Uranium Standard	Inorganic Ventures	0.050

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
<b>CLPP-SPK-INT1</b>	QCP-CICV-3	Inorganic Ventures	10.0
	1000 mg/L Sb	High Purity Standards	5.0
	10000 mg/L K	High Purity Standards	10.0
	10000 mg/L Na	High Purity Standards	7.5
	10000 mg/L Mg	High Purity Standards	5.0
	10000 mg/L Ca	High Purity Standards	2.5
<b>CLPP-SPK-INT2</b>	2007ICS-1	Inorganic Ventures	10.0
	1000 mg/L Sr	High Purity Standards	5.0
	1000 mg/L Sn	High Purity Standards	5.0
	10000 mg/L Si	High Purity Standards	5.0

**TITLE: ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 3

ELEMENT CONCENTRATIONS IN MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Element	CONCENTRATION IN SOLUTION, mg/L								
	Matrix Spike	LCSW	CLPP-SPK-1	CLPP-SPK-4	CLPP-SPK-INT1	CLPP-SPK-INT2	QCP-CICV-3	2007 ICS-1	1000 mg/L U
Aluminum	2.000	2.000	2000						
Antimony	0.500	0.500		100	50				
Arsenic	2.000	2.000		40	200		500		
Barium	2.000	2.000	2000						
Beryllium	0.050	0.050	50						
Boron	0.500	0.500				50		500	
Cadmium	0.050	0.050		50	5		250		
Calcium	2.500	2.500			250				
Chromium	0.200	0.200	200						
Cobalt	0.500	0.500	500						
Copper	0.250	0.250	250						
Iron	1.000	1.000	1000						
Lead	0.500	0.500		20	50		500		
Magnesium	5.000	5.000			500				
Manganese	0.500	0.500	500						
Molybdenum	0.300	0.300				30		300	
Nickel	0.500	0.500	500						
Potassium	25.000	25.000			2500				
Selenium	2.000	2.000		10	200		500		
Silicon	5.230	5.230				523		230	
Silver	0.050	0.050	50						
Sodium	7.500	7.500			750				
Strontium	0.500	0.500				50			
Thallium	2.000	2.000		50	200		500		
Tin	0.500	0.500				50			
Titanium	1.000	1.000				100		1000	
Uranium	1.000	1.000							1000
Vanadium	0.500	0.500	500						
Zinc	0.500	0.500	500						

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Prepared By: Peter Lemay Date: 4/98  
 Approved By: \_\_\_\_\_  
 Group Supervisor: Peter Lemay Date: 1/15/01  
 Operations Manager: John C. Banta Date: 1/15/01  
 QA Officer: Deborah J. Nadeau Date: 1.22.01  
 General Manager: Dennis P. Kufan Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8082	Format changes, added pollution prevention, minor changes to sections 7, 8 and Table 1	DL	1.22.01	1/02/01
02 8082	Revised sections 7.3.1, 7.4.5 and 7.6.1 to be compliant with South Carolina requirements.	DL	5.23.01	5.23.01
03 8082	Changed to practice of reporting highest value. Other minor changes to sections 7.5.2, 7.7.3 + to Table 2.	DL	5.21.02	5.21.02
04 8082	Revised SOP to indicate Turbochrom is being used as instrument control + data collection software. Included target-related definitions. Changes to sections 7.7.3, 7.7.4 and 7.8.	MRC	08.20.04	08.20.04
05 8082	Changed 7.5.2 to reflect alternating CV Changed Table 2 Sect. 7.3.1 New checklist added wording to sect. 8	LAD	020305	020305



---

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

---

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

---

I acknowledge receipt of copy \_\_\_ of document **SOP CA-329-06**, titled **ANALYSIS OF AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_ of document **SOP CA-329-06**, titled **ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

---

## **1.0 SCOPE AND APPLICATION**

This SOP describes all aspects of the analysis of extracts of solid and aqueous samples for PCBs by EPA Method 8082 as performed by Katahdin Analytical Services, Inc. including sample analysis, data review, standard preparation and instrument calibration.

It is applicable to the following compounds: Aroclor-1242, Aroclor-1254, Aroclor-1221, Aroclor-1232, Aroclor-1248, Aroclor-1260 and Aroclor-1016. Extracts are analyzed by Gas Chromatography-Electron Capture Detector (GC-ECD)

### **1.1 Definitions**

**ANALYTICAL BATCH:** 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

**METHOD BLANK (laboratory reagent blank):** An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; for soil samples, muffled sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

**CALIBRATION CHECK:** Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

**CALIBRATION STANDARD (WORKING STANDARD):** A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

**LABORATORY CONTROL SAMPLE (LCS):** A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

**MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD):** Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

**STANDARD CURVE (CALIBRATION CURVE):** A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

---

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

---

**STOCK STANDARD SOLUTION:** A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

**SURROGATES:** Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

**KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS):** A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

**PE NELSON TURBOCHROM:** A data acquisition system that is used to collect chromatographic data. The system can also be used to archive raw data files.

**TARGET:** A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

**TARGET DB:** An oracle database used to store and organize all Target data files.

**QUICKFORMS:** A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

## 1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of PCBs by method 8082. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis by method 8082 to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

---

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

---

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

**1.3 Health and Safety**

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

**1.4 Pollution Prevention/Waste Disposal**

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

---

**2.0 SUMMARY OF METHOD**

**2.1** Method 8082 provides gas chromatographic conditions for the detection of PPB concentrations of certain PCBs. Prior to the use of this method, appropriate sample extraction techniques must be used. Both neat and diluted organic liquids (Method 3580, waste dilution) may be analyzed by direct injection. A 2 to 5 ul aliquot of sample is

---

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

---

injected into a gas chromatograph (GC) using the direct injection technique, and compounds in the GC effluent are detected by an electron capture detector (ECD).

- 2.2 The sensitivity of Method 8082 usually depends on the concentration of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, Method 8082 may also be performed on samples that have undergone the following cleanups: Method 3660 - Sulfur Cleanup and Method 3665 - Sulfuric Acid Cleanup.

---

**3.0 INTERFERENCES**

Interferences by phthalate esters can pose a problem in PCB determinations when using the electron capture detector. Common flexible plastics contain various amounts of phthalates. Care has to be taken to avoid using any plastic materials during the extraction process. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

---

**4.0 APPARATUS AND MATERIALS**

4.1 Gas chromatograph

4.1.1 GC Hewlett Packard 5890 series I or II connected to the Turbochrom data system, or equivalent.

4.1.2 Columns - Instruments are configured with a pre-column originating from the injection port, which is connected to a deactivated glass Y splitter that connects two different columns to two detectors. The most commonly used columns are: RTX-35 30M x 0.53 mm ID, RTX-5 30M x 0.53 MM ID, or RTX-1701 30M x 0.53 mm ID. Equivalent columns can be used.

4.1.3 Detectors: Electron capture detectors (ECD).

4.2 Volumetric flasks, class A: sizes as appropriate with the ground-glass stoppers.

4.3 Syringes: various sizes for preparing standards and injecting samples on the instrument.

4.4 Vials: various sizes and types including crimp tops.

4.5 Balances: Analytical, 0.0001 g

4.6 Refrigerator for storage of extracts and standards.

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

---

## **5.0 REAGENTS**

### 5.1 Solvents

5.1.1 Hexane: pesticide quality or equivalent for diluting samples and standards.

### 5.2 Standards

5.2.1 Stock standard solutions: Solutions purchased from suppliers like Restek or other acceptable retailers. Expiration dates are one year from date of opening vial or sooner if manufacturers date is less. Upon receipt, all standards are logged into the appropriate logbook with the date of receipt, expiration date, source, lot number, solvent and concentration of compounds. Standard solutions are stored at 4°C in polytetrafluoroethylene (PTFE)-sealed containers in the dark.

5.2.2 Calibration standards: Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in standards prep logbook. The concentrations of the working PCB calibration standards are 0.05 ug/ml, 0.10 ug/ml, 0.25 ug/ml, 1.0 ug/ml, 2.5 ug/ml, and 10.0 ug/ml. The standards also contain the surrogates Tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB) at the respective concentrations: 0.001 ug/ml, 0.002 ug/ml, 0.005 ug/ml, 0.020 ug/ml, 0.050 ug/ml, and 0.20 ug/ml.

---

## **6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING**

Extracts must be stored under refrigeration and analyzed within 40 days of extraction.

---

## **7.0 PROCEDURES**

### 7.1 Extraction

Refer to the appropriate SOPs for the correct extraction procedure. In general, water samples are extracted using methods 3510 or 3520 while solid samples use methods 3540, 3545, or 3550.

### 7.2 Instrument conditions

Refer to the instrument logbook for the current column and conditions.

Typical conditions are: Makeup flow: 60 ml/min Ar/Methane

---

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

---

Column flow: 6 ml/min  
Injector Temp: 200  
Detector Temp: 300  
Oven Ramp: 160(0) - 5/min - 260(10)  
Run time: 30 min  
Injection size: 2 ul

### 7.3 Calibration

7.3.1 The GC system is calibrated using the external standard calibration procedure. Six-point calibration standards of Aroclor 1660 (Aroclor 1016 and Aroclor 1260), Aroclor 1242, Aroclor 1248 and Aroclor 1254 are prepared along with mid-point calibration standards of Aroclor 1221 and Aroclor 1232. If Aroclor 1221 or Aroclor 1232 are suspected, then a six-point curve of the respective aroclor will be analyzed prior to the analysis and quantitation of the sample.

Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. Three to five characteristic peaks from each aroclor are used to calibrate a curve. The Target system will calculate a peak height for all three to five peaks in each aroclor. A separate calibration curve for each of the three to five peaks can be prepared in Target using the peak height against the concentration of the standard. A non-linear calibration applying a second order polynomial (quadratic fit) equation is used to prepare the curve. In order to be used for quantitative purposes, the correlation coefficient must be greater than or equal to 0.990. The quadratic equation is:

$$y = ax^2 + bx + c$$

where: y = Instrument response  
b = Slope of the line  
x = Concentration of the calibration standard  
c = The intercept

Please note that a non-linear calibration model may not be allowable for certain stated, federal programs, or clients. South Carolina does not allow non-linear calibration work originating in their state. In these cases, a linear calibration model must be used. The linear equation is:

$$y = bx + c$$

where: y = Instrument response  
b = Slope of the line  
x = Concentration of the calibration standard  
c = The intercept

---

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

---

The calibration curve is calibrated the same way as the second order polynomial equation except that a five-point calibration standard mix is used.

7.3.2 The working calibration curve must be verified on each 12 hour shift that samples are to be analyzed by injecting the mid-point calibration standard. If the response for any analyte varies from the expected response by more than  $\pm 15\%$ , a new calibration curve must be prepared for that analyte. The average result for 5 peak heights of the PCB are used for quantitation.

7.4 Retention time windows

7.4.1 Three injections are made of all the PCBs throughout the course of a 72 hour period.

7.4.2 A major peak from the envelope is chosen and a standard deviation is calculated using the three retention times for that peak.

7.4.3 Plus or minus three times the standard deviation of the retention times for each standard is used to define the retention time window; however, the experience of the analyst should weight heavily in the interpretation of chromatograms. The analyst should use the retention time window, but should primarily rely on pattern recognition.

7.4.4 Retention time windows are calculated for each standard on each GC column and whenever a new GC column is installed. The data is kept on file in the laboratory.

7.4.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being too narrow. The windows are:  $\pm 0.07$  for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of  $\pm 0.03$  minutes must be used if the established retention time window is less than 0.03 minutes.

7.5 Gas chromatographic analysis

7.5.1 Shake samples and let them sit for one minute before vialing for analysis.

---

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

---

- 7.5.2 All instrument injections are performed using the direct injection technique with an autosampler set for 2-5 ul injection volumes.
- 7.5.3 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration as listed in section 7.3 followed by sample extracts interspersed with mid-concentration calibration standards. Before any samples are analyzed the instrument must be calibrated by analyzing a six-point calibration or a 1.0ppm concentration standard (CV-calibration verification standard) for Aroclor 1660, Aroclor 1242, Aroclor 1248 and Aroclor 1254. If a CV is run, the calculated concentration must not exceed a difference of  $\pm 15\%$ . Each sample analysis must be bracketed with an acceptable initial calibration or an opening CV and an ending CV for each 12-hour shift. The closing CV for Aroclor 1660 is a 0.25ppm concentration standard. All other aroclors at the closing of the run remain at 1.0ppm concentration. If a second window of samples is run immediately after the closing CVs, the concentration of Aroclor 1660 at the completion of this window would be 1.0ppm. The calibration standard must also be injected at intervals of not less than once every twenty samples (after every 10 samples is recommended) and at the end of the analysis sequence. If the CV fails, the instrument is checked for any obvious problems and maintenance is performed if deemed necessary. Another CV is analyzed or the instrument is recalibrated and then samples are injected. All samples that were injected after the standard exceeding the criterion must be reinjected to avoid errors in quantitation, if the initial analysis indicated the presence of the specific target analyte that exceeded the criterion.
- 7.5.4 Absolute retention time windows are established every 12 hour shift for each analyte using the mid-point of the window of that day **if** after analyzing the mid-point it is determined that one or more of the analytes fall outside of the previously established absolute retention time window. The daily retention time window equals the mid-point  $\pm$ three times the standard deviations.
- 7.5.5 The identification of PCBs is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes. An analyte is tentatively identified when a peak from a sample falls within the daily retention time window. Each tentative identification must be confirmed using a second GC column of dissimilar stationary phase or using another technique such as GC/MS. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.
- 7.5.5.1 An additional criterion is applied for the identification and quantitation of PCBs. Identification is based on the characteristic fingerprint

---

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

---

retention time and shape of the major peaks. Major peaks are defined as those peaks in the Aroclor standard that are at least 25% of the height of the largest Aroclor peak. The sample chromatogram is compared to the individual Aroclor standard chromatograms. Once the Aroclor pattern has been identified, a concentration is then calculated in Target.

7.5.5.1.1 Three to five aroclor concentrations are calculated using the peak heights of the three to five characteristic peaks of the aroclor. These three to five concentrations are then averaged to determine the concentration of that aroclor.

7.5.6 When samples are analyzed from a source known to contain specific Aroclors, the results from a single-column analysis may be confirmed on the basis of a clearly recognizable Aroclor pattern.

7.5.7 If the response for an analyte exceeds the calibration range of the system, the sample must be diluted and reanalyzed.

7.5.8 If peak detection and identification are prevented due to interferences, the hexane extract may need to undergo a cleanup. The extract may be subjected to a sulfur cleanup (method 3660) and/or a sulfuric acid cleanup (method 3665).

7.5.8.1 **Note:** Samples routinely receive a sulfuric acid clean up. However, for samples from a known site with a clean matrix, a sulfuric acid clean up may not be performed. Whenever a sample receives a cleanup, the associated QC must also be subjected to the same cleanup(s) and reanalyzed.

7.5.9 When a GC system is determined to be out of control because either a CV cannot pass or a six point calibration does not meet the correlation coefficient criteria, instrument maintenance is likely necessary. Routine instrument maintenance may involve changing the septum, replacing the liner, clipping the pre-column, or replacing the column. This information is recorded in the instrument run log (Figure 1). When an instrument requires more severe maintenance like replacing the ECD or an electronic board, this information is written in the instrument maintenance logbook.

## 7.6 Calculations

7.6.1 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration after the file is processed through the appropriate calibration method. Aroclor quantitation is accomplished by the method described in

---

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

---

section 7.5.4.1.1. However, if a sample contains more than one aroclor, a peak common to both analytes must not be used to quantitate either compound.

7.6.2 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

7.6.2.1 Water:

$$\text{Concentration (ug/L)} = (C) (Vt)/(Vs)$$

7.6.2.2 Soil/Sediment:

$$\text{Concentration (mg/kg)} = (C) (Vt)/(Ws) (D)$$

where, C = concentration calculated by Turbochrom in ug/ml  
Vt = Volume of total extract including any instrument dilutions  
Vs = Volume of sample extracted  
Ws = Weight of sample extracted  
D = Decimal total solids

7.7 Data Review

7.7.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed samples. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- ◆ QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.
- ◆ Surrogate recovery
- ◆ Chromatography: cleanups, manual integration.
- ◆ Target compound detection: quantitation, confirmation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next workday. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.8.

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

---

7.7.2 Surrogate recovery

All recoveries must meet the most recently laboratory established acceptance limits, which are listed on the GC Laboratory Surrogate Acceptance Limit sheet.

The sample is evaluated for recoveries of the two surrogates. If the recovery of one surrogate is within the acceptance limit, and the second is out, the data is narrated. If the surrogate recoveries are high for both and the sample contains less than the PQL for all target analytes, the data is narrated. If the surrogate recoveries are low and may be attributable to matrix interference or a matrix effect, the data is narrated. If the surrogate recoveries are low and the sample concentration is less than the PQL for all target analytes and there is no apparent matrix effect, reextract the sample.

7.7.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries. If the chromatogram indicates interferences, then a cleanup may be needed. See section 7.5.7.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an "m" qualifier will automatically be printed on the quantitation report summary. For specific procedures on how to manually integrate, refer to Katahdin SOP QA-811, Manual Integration, current revision.

7.7.4 Target Compound Detection

The chromatogram from channel A is evaluated with that from channel B. If a target analyte is present on both channels and the concentration is within the calibration range, and the quantitation from both chromatograms agrees within  $\pm 50\%$ , the analyte is considered present in the sample. The higher of the two concentrations is reported.

---

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

---

In order to avoid reporting false positives, identified peaks on a chromatogram may need to be undetected electronically in Target. The possible scenarios are: If an analyte is present on one column but its concentration is below the PQL, if an analyte is present on one column but does not confirm on the other channel, if an analyte is present on both columns but the concentrations differ by more than 50%, or if an analyte is present but its retention time is  $\pm 0.04$  minutes or more than the retention time of the analyte in the preceding CV.

#### 7.8 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the Department Manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

---

### 8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions

---

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

---

may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

- 8.1 For each analytical batch (up to 20 samples), a method blank, laboratory control sample (LCS), matrix spike and matrix spike duplicate are analyzed. They are carried through all stages of the sample preparation and analysis steps.
- 8.2 Spike concentrations: The LCS and the MS/MSD are spiked at the same concentration with Aroclor 1660. The spike concentrations are:

	<u>WATER ug/L</u>	<u>SOILS mg/kg</u>
Aroclor 1660	5.0	0.17

The surrogate spike concentrations in the final extract are:

	<u>WATER ug/ml</u>	<u>SOILS ug/ml</u>
Tetrachloro-m-xylene(TCX)	0.10	0.10
DCB	0.10	0.10

- 8.3 LCS and MS/MSD acceptance criteria and Corrective Action: All QC samples are calculated for percent recovery of the spiked analyte. The recoveries are compared to laboratory established acceptance limits. The LCS acceptance limits for PCBs are established for both water and soil matrices. The MS/MSD acceptance limits for PCBs use the respective matrix LCS acceptance limits. Separate limits for MS/MSD pairs are not calculated because of the varying matrices involved. In addition many of the MS/MSD data points cannot be used (i.e. recoveries not calculable due to a matrix effect).

If any spike compound in the laboratory control sample falls outside of the established recovery acceptance limit window, the QC sample is considered to be out of control and any sample that is associated should be evaluated with other QC elements to determine the corrective action. If the recovery is high and the associated samples do not contain the specific compound(s), the data can possibly be accepted with narration. In other cases, the associated samples must be extracted.

If a spike compound is outside of the acceptance limits in the matrix spike sample but is acceptable in the LCS, the data is considered acceptable. The cause of the failure is possibly attributable to matrix interference. However, if the compound fails in both the LCS and the MS/MSD, the result for that analyte is suspect and may not be reported for regulatory compliance purposes.

---

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

---

- 8.4 Surrogate acceptance criteria and Corrective Action: Surrogate recoveries are calculated on all samples, blanks and spikes. The recoveries are compared to laboratory established acceptance limits.

When a sample has a surrogate that falls outside of the laboratory established acceptance limit window, the problem should be investigated. If the recovery looks like it is affected by the sample matrix, the sample may be reinjected to confirm matrix interference. When a sample has no detectable surrogate recovery, the sample should be reextracted.

- 8.5 CAR: Whenever data is not acceptable because of a failing LCS or surrogate recovery, a corrective action report (CAR) must be initiated as soon as possible to document resolution.

---

**9.0 METHOD PERFORMANCE**

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the current revision of Method 8082 for other method performance parameters and requirements.

---

**10.0 APPLICABLE DOCUMENTS/REFERENCES**

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition. Final Update III, dated December, 1996.

---

**LIST OF FIGURES**

Table 1 - QC Requirements  
Table 2 - Summary of Method Modifications  
Figure 1 - Instrument Run Log  
Figure 2 - Review Checklist  
Figure 3 – PQLs

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

TABLE 1  
QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS	One per prep batch of twenty or fewer samples	Statistically derived limits	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank, QC and the remaining samples.
CCV	After every 20 samples; If calibration curve previously analyzed, analyze daily before samples.	± 15% D	(1) Evaluate the samples: If the %D >+15% and sample results are <PQL, narrate. If %D >±15% only on one channel, narrate. If %D >±15% for closing CV, and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples back to last acceptable CV.
Matrix Spike\ Matrix Spike Duplicate	One for every set of 20 samples	Same as for LCS	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep samples and QC.

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

TABLE 1, cont'd

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Sample Duplicate	One sample duplicate per ten samples if requested	RPD $\leq$ 20	(1) If lab QC in criteria and matrix interference suspected, flag data or narrate (2) Otherwise, reanalyze
6pt calibration of Aroclor 1660, 1242, 1248, 1254 and mid-point cal of other Aroclors	Initial cal prior to sample analysis	6 pt calibration - correlation coefficient $\geq$ 0.990	(1) Repeat Initial calibration (2) If single pt cal Aroclor is identified in analysis of sample, 6-pt calibration run of identified compound with reanalysis of sample.
Demonstration of analyst proficiency – 4 replicates	Once per analyst initially and annually thereafter	P&A meet method criteria	Repeat P&A study
MDL study	Once per year	Ideally, PQL = at least 3xMDL	Repeat MDL study

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

TABLE 2  
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-329-06	METHOD 8082, current revision
Procedures	<p>7.4.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being too narrow. The windows are: <math>\pm 0.07</math> for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of <math>\pm 0.03</math> minutes must be used if the established retention time window is less than 0.03 minutes.</p> <p>5.2.2 Calibration standards: Prepared through the dilution of the stock standards ... The standards also contain the surrogates Tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB) at the respective concentrations: 0.001 ug/ml, 0.002 ug/ml, 0.005 ug/ml, 0.02 ug/ml, 0.05 ug/ml, and 0.25 ug/ml.</p> <p>7.3.1. The GC system is calibrated using the external standard calibration procedure. Six-point calibration standards of Aroclor 1660 (Aroclor 1016 and Aroclor 1260), Aroclor 1242, Aroclor 1248 and Aroclor 1254 are prepared along with mid-point calibration standards of Aroclor 1221 and Aroclor 1232. If Aroclor 1221 or Aroclor 1232 are suspected, then a six point curve of the respective Aroclor will be analyzed prior to the analysis and quantitation of the sample.</p>	<p>7.5 refers to method 8000B section 7.6.3: If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. (Recording retention times to three decimal places rather than only two should minimize the instances in which the standard deviation is calculated as 0.000).</p> <p>5.9.1 When PCBs are to be determined as Aroclors, Decachlorobiphenyl is used as a surrogate.</p> <p>7.4.3.2 Standards of the other five Aroclors are necessary for pattern recognition. These standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1016/1260 mixture in section 7.3.4.1 has been used to describe the detector response. The standards for these five Aroclors should be analyzed before the analysis of any samples, and may be analyzed before or after the analysis of the five 1016/1260 standards in sec 7.3.4.1.</p>

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

TABLE 2, cont'd

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-329-06	METHOD 8082, current revision
Procedures cont'd	7.5.3 Absolute retention time windows are established every 12 hour shift for each analyte using the mid-point of the window of that day <b>if</b> after analyzing the mid-point it is determined that one or more of the analytes fall outside of the previously established absolute retention time window.	7.5 refers to method 8000B section 7.6.5: Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift.
Apparatus/Materials		
Reagents		
Sample Preservation and handling		
QC - Spikes		
QC - LCS		
QC – Accuracy/ Precision		
QC - MDL	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

---

FIGURE 1

EXAMPLE OF INSTRUMENT RUN LOG

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

---

FIGURE 2

DATA REVIEW CHECKLIST

**TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/  
 ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**

FIGURE 3

PQLs for Method 8082

METHODS	ANALYTE	Practical Quantitation Level (PQL)	Practical Quantitation Level (PQL)
		(ug/L)	(ug/kg)
SW3540/SW 8082 (S)	PCB-1016	0.50	17
<b>SW3510/SW 8082 (W) #</b>	PCB-1221	0.50	17
<b>SW3520/SW 8082 (W)</b>	PCB-1232	0.50	17
<b>SW3550/SW 8082 (S) *</b>	PCB-1242	0.50	17
SW3545/SW 8082 (S)	PCB-1248	0.50	17
	PCB-1254	0.50	17
	PCB-1260	0.50	17

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Prepared By: George Brewer Date: 01/01

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: Joh C. Buntin Date: 1/29/01

QA Officer: Dorothy J. Kadeau Date: 1-29-01

General Manager: Deanna F. Huffman Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
00 7470A	NA	DN	1-29-01	1/29/01
01	Revised Sect. 4, 5 and 7 to reflect current practice. Revised Sect. 8 to reflect current QC limits. Revised sect. 10 to reflect current Applicable Documents and references. Removed figure 2. Update table 1 to reflect current QC limits. Minor changes throughout	CAJ	02-16-05	02-16-05

---

**TITLE:           DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY  
USEPA METHOD 7470**

---

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

---

I acknowledge receipt of copy \_\_\_ of document **SOP CA-615-01**, titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_ of document **SOP CA-615-01**, titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

TITLE:       **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY  
USEPA METHOD 7470**

---

## 1.0   SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services, Inc. personnel for the digestion and analysis aqueous samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in groundwaters, aqueous wastes, and mobility-procedure extracts under USEPA Method 7470 (Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods, SW-846, 2nd edition, 1982 (revised 1984), 3rd edition, 1986, and Updates I, II, IIA, and III 1996, Office of Solid Waste and Emergency Response, U.S. EPA.

### 1.1   Definitions

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

PB - Preparation Blank - Laboratory grade reagent water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

---

**TITLE:           DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY  
                  USEPA METHOD 7470**

---

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

## 1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7470. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7470 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

## 1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Rubber gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

---

**TITLE:           DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY  
USEPA METHOD 7470**

---

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Management Plan and Safety Manual and follow appropriate procedures such as wearing safety glasses and gloves when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location and use of all safety equipment.

**1.4   Pollution Prevention/Waste Disposal**

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in cold vapor atomic absorption may contain high concentrations of acids, mercury, and other toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested mercury samples and standards and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled "Waste Stream A") that is located in the Metals Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Hazardous Waste Management Plan and Safety Manual and the Department Manager.

---

**2.0   SUMMARY OF METHOD**

The cold vapor atomic absorption technique is based on the absorption of radiation at 253.7 nm by mercury vapor. It relies on the volatility of elemental mercury at room temperature. During preparation, organic mercurials are oxidized and elemental mercury is ionized to  $\text{Hg}^{3+}$ . During instrumental analysis, mercuric ions are reduced to elemental mercury by the addition of stannous chloride. Elemental mercury is then aerated from solution and passes through a cell positioned in the path of a mercury spectrophotometer, where absorbance (peak height) is measured as a function of mercury concentration and recorded by the associated computer. The mercury vapor is then swept out of the instrument into an exhaust hood, where it is evacuated from the laboratory.

---

**3.0   INTERFERENCES**

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold

---

**TITLE:           DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY  
                  USEPA METHOD 7470**

---

vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low bias for analytical results. Certain volatile organic materials will also non-specifically absorb radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate and potassium persulfate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Sea waters, brines, and industrial effluents high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

---

**4.0    APPARATUS AND MATERIALS**

- 4.1    40 mL VOA vials, for use as digestion vessels.
- 4.2    250 mL Pyrex media bottles with plastic screw caps, for use in digesting calibration standards.
- 4.3    Water bath capable of maintaining a constant temperature of 95° C.
- 4.4    Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.
- 4.5    Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents
- 4.6    Spirit-filled thermometer, NIST-traceable, covering the range from 20° to 110° C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.
- 4.7    Disposable graduated polystyrene sample cups, 200 mL capacity
- 4.8    CETAC M-6100 automated mercury analyzer and associated peripherals and parts

---

**TITLE:           DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY  
USEPA METHOD 7470**

---

4.9     Disposable graduated dose cups, 30 mL capacity

Refer to Katahdin SOP CA-629, current revision, "Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer" for additional required materials.

---

**5.0     REAGENTS**

- 5.1     Laboratory grade reagent water – mercury-free water meeting the specifications of ASTM Type II water
- 5.2     Concentrated sulfuric acid, trace metals grade
- 5.3     Concentrated nitric acid, trace metals grade
- 5.4     Concentrated hydrochloric acid, trace metal grade
- 5.5     Potassium permanganate solution, 5% w/v: Dissolve 50 g of potassium permanganate in 1 L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.
- 5.6     Potassium persulfate solution, 5% w/v: Dissolve 50g of potassium permanganate in 1L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.
- 5.7     Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in laboratory grade reagent water and dilute to a final volume of 1 L.
- 5.8     Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory grade reagent water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. Reagent should be labeled as suitable for use in mercury determination.
- 5.9     Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. This intermediate standard is used to prepare calibration standards, matrix spikes, CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard A must be prepared fresh monthly ,and disposed of appropriately after use. (Note: the concentrations of all stock standards must be certified by the vendors as traceable to NIST reference materials).

---

**TITLE:           DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY  
USEPA METHOD 7470**

---

5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. The source of the stock standard used to prepare Intermediate Mercury Standard B must be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard B must be prepared fresh monthly, and disposed of appropriately after use.

---

**6.0   SAMPLE COLLECTION, PRESERVATION AND HANDLING**

Aqueous samples to be analyzed for mercury should be collected and preserved as described in the following table.

<b>Matrix</b>	<b>Container<sup>1</sup></b>	<b>Collection Volume/ Weight</b>	<b>Preservation/ Treatment</b>	<b>Holding Time</b>
Aqueous (total)	P, G	250 mL	HNO <sub>3</sub> to pH < 2	28 days
Aqueous (dissolved)	P, G	250 mL	HNO <sub>3</sub> to pH < 2	28 days

<sup>1</sup> P = polyethylene or , G = glass

---

**7.0   PROCEDURES**

**BOTTLE PREPARATION**

7.1 Mercury digestions are performed in two different types of vessels. Calibration standards, the Initial Calibration Verification (ICV) standard, and the Initial/Continuing Calibration Blank (ICB/CCB) are prepared in 250 mL Pyrex media bottles. Large bottles are used to provide sufficient volumes of these standards to allow for multiple reanalyses when required. Field samples, Method Blanks, and Laboratory Control Samples are digested in 40 mL VOA vials. These smaller vials provide enough digestate to allow one or two reanalyses when required, but reduce the amounts of samples consumed and waste generated.

---

**TITLE:           DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY  
                  USEPA METHOD 7470**

---

VOA vials are reused if the samples they have contained have no measurable mercury above the PQL. After the previous contents of the vials have been discarded, these vials are segregated according to whether the measured mercury concentrations of the previous contents were above the PQL (contaminated vials) or below the PQL (uncontaminated vials). Labels are removed from the vials by wiping with a paper towel saturated with toluene. Uncontaminated vials are rinsed with laboratory grade reagent water. Contaminated vials are discarded.

The Pyrex media bottles in which standards are prepared are emptied, rinsed, and reused. Each of these bottles is permanently marked with the concentration of the standard it contains.

#### PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

- 7.2 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS Metals database and print out a copy of the sample prep bench sheet. All necessary details of sample preparation (standards preparation information, digestion times, initial and final volumes, pertinent observations, etc.) must be recorded on this spreadsheet, which will be bound in the Mercury Preparation Logbook. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.
- 7.3 Using a silver paint marker, label clean VOA vials with the appropriate sample numbers and standard identifications for each sample and standard to be digested.
- 7.4 Use a bottle-top dispenser to add 100 mL of laboratory grade reagent water to 6 standards digestion bottles (250 mL media bottles). Using calibrated adjustable pipettes, prepare calibration standards by adding 0 uL, 20 uL, 50 uL, 100 uL, 500 uL, and 1000 uL of Intermediate Mercury Standard A to separate appropriately-labeled media bottles containing 100 mL of laboratory grade reagent water. The mercury concentrations of these calibration standards are, respectively, 0 ug/L (calibration blank), 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L, and 10.0 ug/L. The 0.2 ug/L and 0.5 ug/L standards are analyzed after calibration as the PQL standard and the CCV (refer to Section 8.0), respectively, as well as being used in the creation of the calibration curve.
- 7.5 Add 100 mL of laboratory grade reagent water to the media bottle labeled "ICV". Using a calibrated adjustable pipette, prepare the Initial Calibration Verification standard (refer to Section 8) by adding 600 uL of Intermediate Mercury Standard B to the water in this bottle, and record the bottle number in the Mercury Preparation Logbook. The mercury concentration of the ICV is 6.0 ug/L.

---

**TITLE:           DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY  
                  USEPA METHOD 7470**

---

- 7.6    Prepare an appropriate number of preparation blanks (PBW) by adding 25 mL of laboratory grade reagent water to labeled vials.
- 7.7    Prepare an appropriate number of laboratory control samples (LCSW) by adding 125 uL of Intermediate Mercury Standard A to labeled digestion vials containing 25 mL of laboratory grade reagent water. The mercury concentration of each LCSW is 5.0 ug/L.
- 7.8    Matrix spikes are prepared by adding 25 uL of Intermediate Mercury Std A to 25 mL aliquots of samples. The concentration of mercury added to each matrix spike is 1.0 ug/L.
- 7.9    All QC samples and blanks are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, sections 7.10 through 7.13 of this SOP. The volumes of reagents added to the standards prepared in the media bottles are four times those listed in sections 7.10 through 7.13.

**SAMPLE PREPARATION AND DIGESTION**

- 7.10   Using a graduated disposable dosecup, transfer 25 mL of sample, or an aliquot diluted to 25 mL, to a digestion vial. Add 1.25 mL of concentrated sulfuric acid and 0.625 mL of concentrated nitric acid, swirling to mix after each addition. Add 3.75 mL of potassium permanganate solution, swirl to mix, and allow to stand for at least 15 minutes. Samples that contain large amounts of organic substances may require additional 3.75 mL aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 3.75 mL aliquots to samples as necessary until the purple color persists for 15 minutes. If any of the samples require these additional aliquots of potassium permanganate solution, record the additional volume used for each sample on the mercury preparation benchsheet.
- 7.11   Add 2 mL of potassium persulfate solution to each sample. Cap the vials and place them in a preheated water bath. Monitor the temperature of the bath with a spirit thermometer throughout the digestion. The temperature of the water bath will fall below 95° C upon addition of the digestion vials. After the temperature of the bath has risen back to 95° C, continue heating the samples at 95° C for two hours.
- 7.12   Remove bottles from the water bath and allow to cool to room temperature. If the purple permanganate color has failed to persist after digestion in any of the samples, add additional 3.75 mL aliquots of potassium permanganate solution as required to the samples, and record these additions in the mercury preparation benchsheet. Heat the samples that required additional permanganate in the water bath at 95° C for an additional two hours. Remove the bottles from the water bath and allow to cool to room temperature. If the purple color fails to persist after the

---

**TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

---

second heating step, consult the Department Manager for advice on how to proceed.

- 7.13 Add 1.5 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion vial and swirl to mix. This will reduce the excess permanganate, and the sample will change from purple to colorless. Wait at least 30 seconds before proceeding with analysis.

#### INSTRUMENTAL ANALYSIS

- 7.14 Digested mercury samples are analyzed using the CETAC M-6100 Automated Mercury Analyzer. Analysis is automated and is controlled by the QuickTrace Mercury Analyzer software running on a dedicated PC. Detailed instructions for setting up the instrument and analyzing samples are given Katahdin SOP CA-629, "Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer".

#### METHOD OF STANDARD ADDITIONS

- 7.15 The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.
- 7.15.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume  $V_x$ , are taken. To the first (labeled A) is added a known volume  $V_s$  of a standard analyte solution of concentration  $C_s$ . To the second aliquot (labeled B) is added the same volume  $V_s$  of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration  $C_x$  is calculated:

$$C_x = \frac{S_B V_s C_s}{(S_A - S_B) V_x}$$

where  $S_A$  and  $S_B$  are the analytical signals (corrected for the blank) of solutions A and B, respectively.  $V_s$  and  $C_s$  should be chosen so that  $S_A$  is roughly twice  $S_B$  on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

---

**TITLE:           DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY  
                  USEPA METHOD 7470**

---

7.15.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 3. A linear regression program may be used to obtain the intercept concentration.

7.15.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

- 1) The apparent concentrations from the calibration curve must be linear over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised.
- 2) The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.
- 3) The determination must be free of spectral interference and corrected for nonspecific background interference.

#### DATA REDUCTION AND REPORTING

7.16 Results are obtained in concentration units (ug/L) from the instrument. Electronic instrument data files are imported into the Metals ACCESS database for data reduction. Sample preparation information (initial sample volumes and final digestate volumes) are entered directly into the Metals ACCESS database to allow calculation of final results for reporting. Results are calculated as follows:

$$\text{Mercury concentration (ug/L)} = \frac{\text{MC} \times \text{DF} \times \text{IV}}{\text{FV}}$$

---

**TITLE:           DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY  
                  USEPA METHOD 7470**

---

Where:           MC = Measured mercury concentration (ug/L)  
                  DF = Dilution factor at instrument  
                  IV = Initial sample volume (mL)  
                  FV = Final digestate volume (mL)

- 7.17 Results that exceed the calibration range of the instrument may not be reported - the sample must be appropriately diluted and reanalyzed. Results for diluted samples should be multiplied by the dilution factor prior to reporting. If additional aliquots of potassium permanganate were added during digestion, the resulting dilution must be corrected for before reporting.
- 7.18 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported to the PQL and flagged with a "U" qualifier.

---

## **8.0    QUALITY CONTROL AND ACCEPTANCE CRITERIA**

USEPA Method 7470 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.4 through 7.8 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

---

**TITLE:           DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY  
                  USEPA METHOD 7470**

---

**INITIAL DEMONSTRATION OF PERFORMANCE**

- 8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument by each method. This determination requires seven replicate analyses of a laboratory grade reagent water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.
- 8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of laboratory grade reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

**ANALYTICAL RUN QC**

- 8.3 Instrument calibration - The instrument must be calibrated each time it is set up, and calibration standards must be digested each day that samples are digested. Calibration includes analysis of a calibration blank and five calibration standards with graduated concentrations in the appropriate range. The concentration of one of the calibration standards must be at the Practical Quantitation Level (PQL). The intermediate standards used for preparing the calibration standards are prepared at least once per month in 2% nitric acid. Because mercury may be adsorbed onto the walls of glass and plastic containers, the calibration standards must be prepared fresh daily. The correlation coefficient for the calibration curve must be at least 0.995. If the calibration curve does not pass this test, analysis must be halted, the problem corrected, and the instrument recalibrated.
- 8.4 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared from a standard source different than that of the calibration standard and at a concentration within the working range of the instrument. The result of the ICV must fall within 90% to 110% of the expected value. If the ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.
- 8.5 The Continuing Calibration Verification (CCV) solution is analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standard used for calibration at a concentration near the mid-point of the calibration curve. Results of the CCVs must fall within 80% to 120% of the expected value. If a CCV fails, associated sample results may

---

**TITLE:           DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY  
                  USEPA METHOD 7470**

---

not be reported from the run until the problem is corrected and a passing CCV has been analyzed. Also, all samples analyzed after the last passing CCV must be reanalyzed.

- 8.6 A calibration blank is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed. Also, all samples analyzed after the last passing CCB must be reanalyzed.
- 8.7 A standard with a mercury concentration that is at the Practical Quantitation Limit (PQL) is analyzed at the beginning of the run to determine calibration accuracy at the reporting limit. Result of the PQL standard should fall within 50% to 150% of the expected values. No corrective action has been established at this time.

#### PREPARATION BATCH QC SAMPLES

- 8.8 A preparation blank (PBW), consisting of laboratory grade reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL). If a preparation blank fails, results may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. Associated sample results that are below the PQL may be reported without notation.
- 8.9 A laboratory control sample (LCSW), consisting of spiked reagent carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless laboratory-generated statistical limits are available. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested.

#### SAMPLE MATRIX QC SAMPLES

- 8.10 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. Matrix spike recoveries for these samples are calculated as follows:

$$\text{Recovery (\%)} = \frac{(P - S)}{A} \times 100\%$$

where:

P = Spiked sample value

---

TITLE:           **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY  
USEPA METHOD 7470**

---

S = Original sample value  
A = Spike amount

The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, a matrix interference should be suspected and the associated sample result should be flagged on the report of analysis.

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

$$\text{RPD (\%)} = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where:

D<sub>1</sub> = Spike sample result  
D<sub>2</sub> = Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

---

## 9.0    **METHOD PERFORMANCE**

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the current revision of USEPA Method 245.1 for other method performance parameters and requirements.

---

## 10.0   **APPLICABLE DOCUMENTS/REFERENCES**

(Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods, SW-846, 2nd edition, 1982 (revised 1984), 3rd edition, 1986, and Updates I, II, IIA, and III 1996, Office of Solid Waste and Emergency Response, U.S. EPA, Method 7470

TITLE:           **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY  
USEPA METHOD 7470**

---

QuickTrace M-6100 Mercury Analyzer – Operator Manual, Version 1.0.1, CETAC  
Technologies, 2003.

QuickTrace Mercury Analyzer – Software Manual, Version 1.0, Cetac Technologies, June  
2002

---

#### List of Tables and Figures

Table 1	QC Requirements
Table 2	Method Modifications
Figure 1	Example Mercury Preparation Logbook Page
Figure 2	Standard Additions Plot

TITLE: **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

TABLE 1  
QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA 7470	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient $\geq 0.995$ .	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within $\pm 10\%$ of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL.	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within $\pm 50\%$ of true value.	No corrective action required at this time.
	Continuing Calibration Verification (CCV)	At beginning or run, after every 10 samples, and at end of the run	Recovery within $\pm 20\%$ of true value	Repeat calibration and reanalyze all samples analyzed since the last successful CCV.
	Continuing Calibration Blank (CCB)	At beginning or run, after every 10 samples, and at end of the run	Less than PQL.	Repeat calibration and reanalyze all samples analyzed since the last successful CCB.
	Preparation Blank (PBW)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration $\geq$ PQL and $< 10x$ the blank concentration.
	Laboratory Control Sample (LCSW)	One per digestion batch of 20 or fewer samples.	Recovery within $\pm 20\%$ of true value.	Redigest all affected samples.

TITLE: **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

TABLE 1, cont'd  
 QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA 245.1	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery $\pm 25\%$ of true value, if sample > 4x spike value.	Flag results.
	Matrix Spike Duplicate Sample (P)	One per digestion batch of 20 or fewer samples.	1) Recovery $\pm$ 25% of true value, if sample < 4x spike added. 2) RPD 20% for duplicate spikes.	Flag results
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < PQL	1) Repeat IDL study. 2) Raise PQL.
	Method Detection Limit (MDL) Study	Annually.	Ideally, PQL = at least 3 X the MDL.	1) Repeat MDL study. 2) Raise PQL.

**TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

TABLE 2  
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-615-01	USEPA METHOD 7470
Reagents	Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	1) Sampling and gas stream switching performed automatically by mercury analyzer. 2) Working Mercury standard prepared monthly in 2% nitric; calibration standards prepared fresh daily.	1) Sampling and gas stream switching performed manually by analyst. 2) Working Mercury standard prepared fresh daily and acidity maintained at 0.15% nitric.
QC – Calibration Verification	1) Known reference sample (ICV) analyzed daily. 2) Calibration verified after every 10 samples with CCV.	1) Known reference sample analyzed quarterly. 2) Calibration verified after every 20 samples.
QC - Calibration Blanks	Acceptance criteria employed for 245.1: $\pm$ PQL	Acceptance criteria stated in 245.1: $\pm$ MDL

TITLE: **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK

Katahdin Analytical Services, Inc. Metals Preparation Benchsheet

Reagent Information: Method: 7470

JT Baker HNO3: Y42044    JT Baker HCL: M/A    JT Baker H2SO4: Y19092    REVIEWED

JT Baker KMNO4: mrl64    JT Baker K2S2O8: mrl56    JT Baker NH2OH-HCl: mrl167

Standards Information: MJE 1.14.04  
KATAHDIN ANALYTICAL  
METALS SECTION

1ppm A = mw7971    TCLPMS(M) = 200uL of 1ppm M/A to 100mL    S0.5 = 50uL of 1ppm B to 100 mL

1ppm B = mw7972    ICV = 600uL of 1ppm A to 100 mL    S1.0 = 100uL of 1ppm B to 100 mL

LCSW = 125uL of 1ppm B to 25mL    S0.2 = 20uL of 1ppm B to 100 mL    S5.0 = 500uL of 1ppm B to 100 mL

Spike(S/P) = 25uL of 1ppm B to 25mL    S10.0 = 1000uL of 1ppm B to 100 mL

Digestion Start Time (@ 95 °C): 13:30    Digestion End Time: 15:30

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LCSWUA13HGWO	UA13HGWO	<u>0.025</u>	L	<u>0.025</u>	L	AQ	HG	EAM	01/13/2004	N/A	N/A	N/A	N/A		
PBWUA13HGWO	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004	N/A	N/A	N/A	N/A		
WU0041-001	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						
WU0041-001P	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						
WU0041-001S	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						
WU0041-002	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						
WU0041-003	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						
WU0041-004	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						
WU0041-005	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						
WU0041-006	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						
WU0041-007	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						
WU0051-001	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						
WU0051-002	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						
WU0051-003	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						
WU0051-004	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						
WU0051-005	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						
WU0051-006	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						
WU0051-007	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						
WU0051-008	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						
WU0051-009	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						
WU0051-010	UA13HGWO		L		L	AQ	HG	EAM	01/13/2004						

MJE 1.14.04

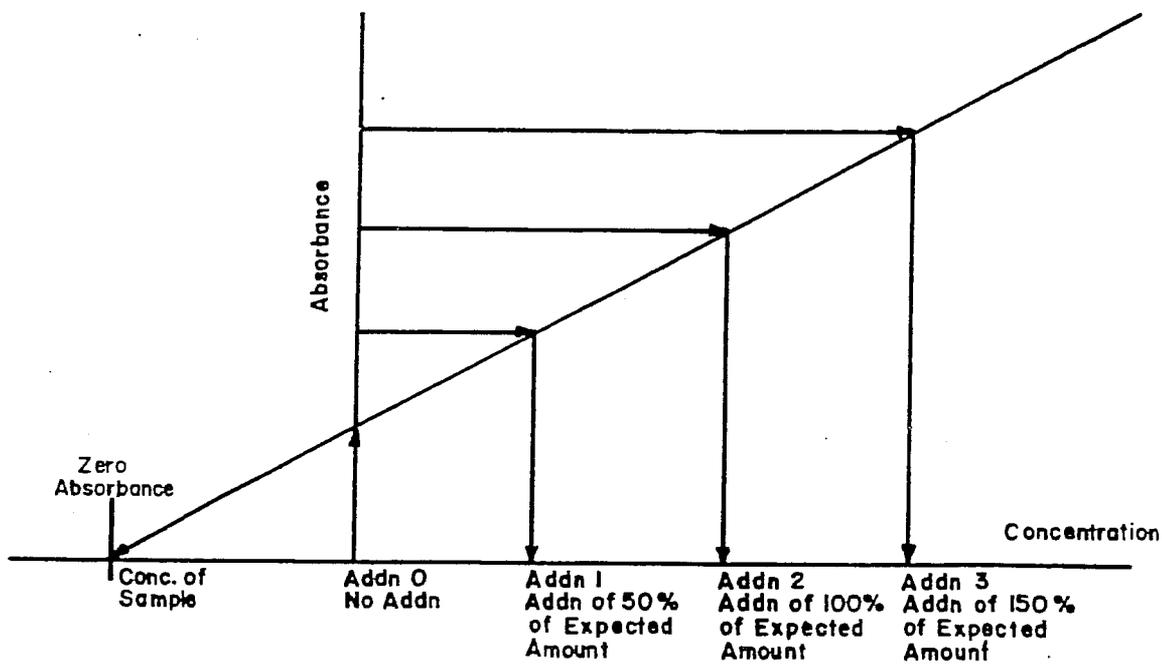
Digestion performed by: Jan    On: 01-13-04

---

TITLE:       **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY  
              USEPA METHOD 7470**

---

FIGURE 2  
STANDARD ADDITIONS PLOT



**TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

Prepared By: Michael Thomas Date: 07-24-00

Approved By:

Department Manager: [Signature] Date: 6-23-06

Operations Manager: [Signature] Date: 6-23-06

QA Officer: [Signature] Date: 6-23-06

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03	Changes to sect. 5.5 : Figures 3 : 4 to reflect current spike solutions and concentrations Replaced cover page. original cover page filed with SOP CA502-02	LAD	04/06	04/06
04	Added definitions, added waste information added LCS/D, added SIM LCS/D, MS/D, updated Table 1, added use of narrow range pH paper. Minor changes throughout to reflect current practice.	LAD	09/07	09/07

---

**TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

---

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

---

I acknowledge receipt of copy \_\_\_ of document **SOP CA-502-04**, titled **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_ of document **SOP CA-502-04**, titled **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

**TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

---

## **1.0 SCOPE AND APPLICATION**

The purpose of this SOP is to describe procedures utilized by Katahdin Analytical personnel in the preparation of all non-CLP aqueous samples for analysis of extractable semivolatile organic compounds.

The goal of this procedure is to ensure uniformity involving the preparation of samples for subsequent SVOA analysis by GC/MS. This SOP is applicable to the extraction portion of EPA Method 625, as well as EPA Methods 3510 (modified separatory funnel extraction) and 3520 (continuous liquid-liquid extraction), current revisions.

### **1.1 Definitions**

**METHOD BLANK** (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

**LABORATORY CONTROL SAMPLE (LCS)**: A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

**MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)**: Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

**SURROGATES**: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

### **1.2 Responsibilities**

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to

---

**TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

---

Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their department follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

### 1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDS's for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

### 1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards

---

**TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

---

are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware, disassembly of CLLEs after extraction, etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction aqueous samples are considered either N-Hi or N-Low waste and should be disposed of in the corresponding satellite waste accumulation area nearest the point of generation. Sodium sulfate used for sample drying should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

---

## **2.0 SUMMARY OF METHOD**

For aqueous samples extracted by CLLE, a one liter aliquot of sample is adjusted to  $\text{pH} \leq 2$  and extracted with methylene chloride using a continuous liquid-liquid extractor. The pH is then adjusted to  $\text{pH} \geq 11$  and the sample is extracted again with methylene chloride. A modified separatory funnel extraction may also be used. If this procedure is used, the sample aliquot is first adjusted to  $\text{pH} \geq 11$  and then to  $\text{pH} \leq 2$ . The methylene chloride extract is dried and concentrated to a volume of 1.0 mL. Specifically, for Method 625, separate base/neutral and acid fractions are produced, with the base/neutral fraction extracted first and the acid fraction second; each is dried and concentrated to 1.0 mL.

---

## **3.0 INTERFERENCES**

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC/MS analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in the total ion current profiles (TICPs). Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or teflon or other non-phthalate plastic substitute.

**TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

---

Special care should be taken to ensure that clean glassware and apparatus are used and pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be needed to minimize interferences.

---

#### **4.0 APPARATUS AND MATERIALS**

Brand names and catalog numbers are included for illustration purposes only.

- 4.1 Continuous liquid-liquid extractors - including body, 500 mL round bottom flask and Alhin condensers and equipped with Teflon or glass connecting joints requiring no lubrication (Hershberg-Wolf Extractor, Ace Glass Company, Vineland, NJ, P/N 6841-10 or equivalent).
- 4.2 Glass powder funnels.
- 4.3 Fluted filter paper, 18.5cm diameter.
- 4.4 Concentrator tube - Kuderna-Danish, 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test.
- 4.5 Evaporation flask - Kuderna-Danish, 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with neck clips.
- 4.6 Snyder column - Kuderna-Danish, three- or four-ball macro (Kontes K-503000-0121 or equivalent).
- 4.7 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.
- 4.8 Vials - Glass, 1.8 mL capacity, with polytetrafluoroethylene (PTFE)-lined screw top and 12 mL with Teflon-lined caps.
- 4.9 2 L separatory funnel, equipped with Teflon stopper and stopcock; Nalgene Teflon FEP separatory funnels may also be used.
- 4.10 Organic Free Boiling Chips - approximately 10/40 mesh, Teflon or silicon carbide (or equivalent). Rinsed with methylene chloride.

---

TITLE: **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

---

- 4.11 Water bath - heated, with concentric ring cover, capable of temperature control ( $\pm 20^{\circ}\text{C}$ ). The bath should be used in a hood.
- 4.12 Nitrogen evaporation apparatus.
- 4.13 Wide range pH test strips, pH 0-14, Whatman CF Type.
- 4.14 Glass rods for stirring samples.
- 4.15 Amber bottles or other appropriate containers for collection of extracts from separatory funnel extraction.
- 4.16 5  $\frac{3}{4}$ " Pasteur pipets.
- 4.17 Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.
- 4.18 Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.

---

**5.0 REAGENTS**

All reagent and solvent lots must be checked for possible contamination. Refer to the current version of Katahdin SOP CA-105, Reagent and Solvent Handling, for further details. The extraction staff is responsible for submitting samples to the GC or GC/MS sections for appropriate analysis. All information concerning preparation of the reagent/solvent lot sample will be recorded in the Organic Extraction Log (Figure 1) and acceptance or rejection of these lots must be recorded in the solvent/reagent lot check logbook (Figure 2). All reagents and solvents must be free (<PQL) of any target compounds.

- 5.1 Laboratory Reagent Grade Water - defined as water in which an interferent is not observed at or above the PQL of each parameter of interest. Deionized water filtered through activated charcoal.
- 5.2 Sodium sulfate - granular. Bake at  $400^{\circ}\text{C}$  for 4 hours (may be done by vendor). Purify by rinsing three times with pesticide grade methylene chloride. Allow residual methylene chloride to evaporate before each use. Cool in a desiccator and store in a glass bottle with a Teflon-lined cap.
- 5.3 Sulfuric acid solution (1:1  $\text{H}_2\text{SO}_4$  :  $\text{H}_2\text{O}$ ) - slowly add 500 mL of  $\text{H}_2\text{SO}_4$  (sp gr 1.84) to 500 mL reagent water.
- 5.4 Acetone, methanol, methylene chloride - pesticide residue analysis grade or equivalent.

---

**TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

---

5.5 Standard Preparation - For all standard preparations, see current revision of the following Katahdin Analytical SOPs:

- "Standards Preparation, Documentation and Traceability", (CA-106, current revision)
- "Balance Calibration," (CA-102, current revision)

5.5.1. Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound	Conc.
phenol-d <sub>6</sub>	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-d <sub>5</sub>	50 ug/mL
p-terphenyl-d <sub>14</sub>	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5.2 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d <sub>10</sub>	2.0 ug/mL
2-Methylnaphthalene-d <sub>10</sub>	2.0 ug/mL
Pyrene-d <sub>10</sub> .	2.0 ug/mL
2,4-Dibromophenol	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5.3 SVOA Matrix Spike/Lab Control Samples Spiking Solution - the matrix spike/LCS solution consists of the compounds listed in Figure 3.

---

**TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

---

Prepare a spiking solution that contains each of the base/neutral compounds listed in Figure 3 at 50 ug/mL in methanol and the acid compounds at 100 ug/mL in methanol. Matrix spike/LCS standards are stored in the freezer (-10°C to -20°C) located in the storage area.

5.5.4 Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 2 ug/mL for base/neutral. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL of methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.5.5 Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution – Prepare a spiking solution in methanol that contains the compounds listed in Figure 4 at concentrations of 100 ug/ml. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

---

**6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING**

Continuous liquid-liquid (Method 3520) and/or separatory funnel (Method 3510) extractions for semivolatiles must be started within seven days of date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific. If sampling date is unknown, the hold time is counted from one day prior to date received.

---

**7.0 PROCEDURES**

The internal chain-of-custody must be signed when removing and replacing samples in storage locations. The sample preparation/extraction log must be filled out with the necessary information as extraction of each sample is completed.

**7.1 CONTINUOUS LIQUID-LIQUID EXTRACTION (Method 3520)**

7.1.1 Set up the CLLE apparatus. All glassware should be pre-rinsed three times with methylene chloride in order to eliminate any contamination factors.

7.1.2 Add approximately 500 - 600 mL of methylene chloride to the CLLE body. Label each flask with the following: sample number (or QC identification number), analyte (SVOA), matrix (Aq), extraction date.

---

**TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

---

- 7.1.3 A method blank and a laboratory control sample (LCS) must be prepared for each daily extraction batch of twenty samples or fewer (if a work order consists of more than twenty samples, a new batch must be started on a separate page with its own method blank and LCS). To prepare method blank and LCS, add 1 L reagent water to a CLLE body. Be sure that no water leaks into the round bottom flask. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. This blank and LCS are carried through the entire extraction and analytical procedure.
- 7.1.4 Mark the sample level (meniscus) on the sample bottle with a wax crayon so that the volume can be measured (this may be done prior to removal from the walk-in cooler). Transfer the sample to a CLLE body, being sure that no water leaks into the round bottom flask.
- 7.1.5 If the batch requires a MS/MSD, transfer two 1 L portions of the sample selected/designated for MS/MSD to CLLE bodies for preparation of a matrix spike/matrix spike duplicate if required. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis. An MS/MSD is required if requested by the client or per 20 samples or every 14 days, whichever occurs first. (Refer to the logbook page, "date QC expires"). If extra MS/MSD aliquots of sample are unavailable a laboratory control sample duplicate (LCSD) may be substituted.
- 7.1.6 Check the pH of each sample with wide range pH paper by removing a couple of sample drops with a clean disposable pipet or on the tip of a stirring rod. Adjust the pH of the samples (including method blank, LCS/LCSD, and MS/MSD) to  $\leq$  pH 2 with 1:1 H<sub>2</sub>SO<sub>4</sub> after addition of surrogates and spikes and prior to attaching Allihn condensers (Step 7.1.11). Stir with a glass stirring rod and check pH by tapping the glassrod onto wide range pH paper. The pH must be  $\leq$  2. If the pH test strip does not clearly indicate the pH is less than 2, narrow range pH paper must be used.
- 7.1.7 For each sample, rinse the original sample container with approximately 30 mL of methylene chloride. Add this rinse to the CLLE body.
- 7.1.8 Determine the initial volume of the samples. Measure the initial volume of the samples by filling the sample bottles with water up to the level marked earlier. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.
- 7.1.9 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL base/neutral and acid (SVOA) surrogate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record surrogate spike volume and identification

---

**TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

---

- code in extraction logbook. Thoroughly rinse syringe with acetone before and after each use.
- 7.1.9.1 If the request is for SVOA, use the SVOA Surrogate Solution (sect. 5.5.1).
- 7.1.9.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5.2).
- 7.1.9.3 If the request is for SIM-SVOA, use the SIM surrogate solution as well as SVOA surrogate solution. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.
- 7.1.10 To LCS/LCSD and MS/MSD add 1.0 mL base/neutral and acid (SVOA) matrix spike/LCS spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with methanol before and after each use.
- 7.1.10.1 If the request is for SVOA -  
add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3).
- 7.1.10.2 If the request is for SIM -  
add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3) and  
add 1.0 mL of Base/Neutral (SIM) Matrix Spike/ Lab Control Sample Spike Solution (sect 5.5.4).
- 7.1.10.3 If the request is for SVOA Appendix IX, use the SVOA Appendix IX Spiking solution as well as the SVOA spiking solution -  
add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3) and  
add 1.0 mL of Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution (sect 5.5.5).
- 7.1.11 Attach cooling water Allihn condensers, after first rinsing each 45/50 joint with methylene chloride. Turn on the heating mantles and allow the samples to extract for  $22 \pm 2$  hours. Turn off the mantles and let samples cool.
- 7.1.12 Detach condensers and verify that the pH is still  $\leq 2$  in the same manner mentioned in 7.1.6. If the pH has changed, more acid should be added to make the pH  $\leq 2$  and the sample extracted for several more hours.

---

TITLE: **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

---

7.1.13 Upon completion of acid extraction, allow the sample to cool. Detach condensers and add enough 10N NaOH to adjust the pH to  $\geq 11$  with stirring. Use glass stirring rods to stir and check the pH of each sample in the same manner mentioned in 7.1.6.

7.1.14 Re-attach Allihn condensers, turn on heating mantles, and allow samples to extract for  $22 \pm 2$  hours. Turn off mantles and allow samples to cool.

Proceed to Step 7.4 for sample extract concentration procedures.

7.2 SEPARATORY FUNNEL EXTRACTION (Modified Method 3510)

If an emulsion prevents acceptable recovery or client history indicates samples may demonstrate matrix interference, then samples should be extracted by continuous liquid-liquid extraction (CLLE).

7.2.1 Rinse all glassware, including teflon separatory funnels, three times with methylene chloride prior to use.

7.2.2 Label 2 L separatory funnels and amber collection bottles clearly. Each label should include: sample number (or QC indicator number), analyte (SVOA), matrix (Aq), extraction date.

7.2.3 A method blank and a laboratory control sample (LCS) must be prepared for every 20 samples or with each extraction batch, whichever is more frequent. To prepare method blank and LCS, add 1 L reagent water to a separatory funnel. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. This blank and LCS are carried through the entire extraction and analytical procedure.

7.2.4 Mark the sample level (meniscus) on the sample bottle with a wax crayon so that the volume can be measured (this may be done prior to removal from the walk-in cooler). Transfer the sample to a separatory funnel.

7.2.5 If the batch requires a MS/MSD, transfer two 1 L portions of the sample selected/designated for MS/MSD to separatory funnels for preparation of a matrix spike/matrix spike duplicate if required. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis. An MS/MSD is required if requested by the client or per 20 samples or every 14 days, whichever occurs first. (Refer to the logbook page, "date QC expires"). If extra MS/MSD aliquots of sample are unavailable, a laboratory control sample duplicate (LCSD) may be substituted.

---

**TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

---

- 7.2.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL base/neutral and acid (SVOA) surrogate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with acetone before and after each use.
- 7.2.6.1 If the request is for SVOA, use the SVOA Surrogate Solution.
- 7.2.6.2 If the request is for SIM, use the SIM Surrogate Solution.
- 7.2.6.3 If the request is for SIM-SVOA, use the SIM surrogate solution as well as SVOA surrogate solution. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.
- 7.2.7 To LCS/LCSD and MS/MSD add 1.0 mL base/neutral and acid (SVOA) matrix spike/LCS spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in the extraction logbook. Thoroughly rinse syringe with methanol before and after each use.
- 7.2.7.1 If the request is for SVOA, use the SVOA Spiking Solution.
- 7.2.7.2 If the request is for SIM, use the SIM Spiking solution.
- 7.2.7.3 If the request is for SVOA Appendix IX, use the SVOA Appendix IX Spiking solution as well as the SVOA spiking solution
- 7.2.8 For each sample, rinse the original sample container with 60 mL of methylene chloride. Add this rinse to the separatory funnel.
- 7.2.8 Determine the initial volume of each sample. Measure the initial volume of the sample by filling the sample bottle with water up to the level marked earlier. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.
- 7.2.10 Adjust the pH of the samples (including method blank, LCS/LCSD, and MS/MSD) to  $\text{pH} \geq 11$  with 10N NaOH after addition of surrogates and spikes. Stir with a glass stirring rod and check pH by tapping the glass stirring rod onto wide range pH paper. The pH must be  $\geq 11$ . If the pH test strip does not clearly indicate the pH is greater than 11, narrow range pH paper must be used.

---

TITLE: **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

---

- 7.2.11 Add 60 mL of methylene chloride directly to the method blank and LCS/LCSD separatory funnels.
- 7.2.12 Extract the samples by shaking the funnel for two minutes, venting often, but gently, in a hood to release pressure. A mechanical shaker may be used, where samples are shaken for 3 minutes. Following each shake, allow phases to separate for at least 10 minutes. Drain the methylene chloride layer into an amber collection bottle.
- 7.2.13 If an emulsion forms, mechanical techniques must be employed to achieve maximum separation. Such means include swirling, centrifugation, and draining through a small separatory funnel. In certain instances, transferring the entire sample into a continuous liquid-liquid extractor (CLLE) may be the only alternative. If any such techniques are used, they must be noted in the extractions logbook, and the batch transferred to a CLLE batch with its own batch ID.
- 7.2.14 Add a second 60 mL aliquot of methylene chloride to the separatory funnel and extract for the second time (see 7.2.12 – 7.2.13). Collect the methylene chloride layer in the same amber collection bottle.
- 7.2.15 Repeat the extraction for a third time as described in 7.2.14.
- 7.2.16 Following the third shake, using a glass stirring rod, check the pH to ensure that it has remained at  $\geq 11$ . If the pH has changed back to neutral range, it must be readjusted to  $\geq 11$  and the sample must be extracted at least one more time, adding the methylene chloride to the same amber bottle, that was previously used. If the pH has remained at a value  $\geq 11$ , the pH is then adjusted to  $\leq 2$  with 1:1 H<sub>2</sub>SO<sub>4</sub>. Add enough 1:1 H<sub>2</sub>SO<sub>4</sub> to adjust the pH to  $\leq 2$  with stirring. Use glass stirring rods to stir.
- 7.2.17 Add 60 mL methylene chloride and extract the samples three times in the same manner described in 7.2.11 – 7.2.13. Collect the methylene chloride layer in the same amber collection bottle used to collect the acid fraction.

Proceed to Section 7.4 for extract concentration procedures.

7.3 625 BN/A EXTRACTION (Either Method 3510 or 3520 may be used.)

This extraction procedure is used, where extraction will be followed by 625 GC/MS analysis rather than 8270 GC/MS. Either a separatory funnel extraction technique or a continuous liquid-liquid extraction technique (described above in 7.1 and 7.2) can be used for this procedure. There are two main differences in this procedure from those described in sections 7.1 and 7.2: (1) the pH adjustments articulated in section 7.1

---

TITLE: **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

---

will be reversed when 3520 CLLE is used; and (2) the collection of base and acid fractions are collected in separate containers and concentrated separately.

7.3.1 For extraction using a separatory funnel technique (Method 3510), follow the general procedure as described in 7.2. **NOTE:** The pH adjustments are made in the same manner as described in section 7.2 with the first adjustment to  $\geq 11$ , followed by extraction of the samples three times in methylene chloride. The methylene chloride layer is collected in an amber jar. Label this as the base/neutral fraction. After the third shake, the pH is adjusted to  $\leq 2$ , followed by extraction of the samples three times in methylene chloride. The methylene chloride layer is collected in a separate amber jar. Label this the acid fraction.

7.3.2 For extraction using a continuous liquid-liquid technique (Method 3520), follow the general procedure as described in 7.1. **However; the pH adjustments are reversed from those described in section 7.1.** Following the first  $22 \pm 2$  hours extraction at  $\text{pH} \geq 11$ , the round bottom flask with the base/neutral fraction is removed, covered, and stored. A new round bottom flask with 200 mL methylene chloride is attached to the CLLE body and the pH is adjusted to  $\leq 2$ . Extraction continues at this pH for an additional  $22 \pm 2$  hours.

#### 7.4 CONCENTRATING THE EXTRACTS

For Methods 3510 and 3520, the combined fractions are concentrated to a final volume of 1.0 mL. For Method 625, the base/neutral fraction and acid fraction extracts are routinely concentrated separately to 1.0 mL. Figure 4 shows an EPA Region I memo which discusses GC/MS analysis of the separate base/neutral and acid extracts versus GC/MS analysis of combined extracts from Method 625.

7.4.1 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride. Add two boiling chips to the K-D prior to final rinse. Also rinse the assembled funnels, filter paper, and granular sodium sulfate used for drying the extracts.

7.4.2 Transfer the methylene chloride extract to a K-D concentrator setup through a short stem funnel filled with 1-2 inches of sodium sulfate in fluted filter paper. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with  $\sim 2 - 3$  mL of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with  $\sim 15$  mL of methylene chloride and allow to drain

---

TITLE: **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

---

- 7.4.3 Transfer the label from the collection bottle or round bottom flask (for CLLE) to a K-D. Remove the funnel and attach a 3- or 4-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.
- 7.4.4 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches  $\approx$  6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with  $\approx$  1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with  $\approx$  1 mL methylene chloride.
- 7.4.5 Reduce the methylene chloride extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with  $\approx$ 1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N<sub>2</sub> sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.
- 7.4.6 Reduce each extract to slightly less than 1 mL and then, using a 5  $\frac{3}{4}$ " pasteur pipet, transfer the final extract and label to a 1.8 mL vial with PTFE-lined cap.
- 7.4.7 Using methylene chloride for a quantitative transfer, adjust the final volume of each extract to 1 mL. Use the 1 mL oil-filled reference vial for volume comparison.
- 7.4.8 Store in refrigerator until GC/MS analysis.

## 8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)

**TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

---

- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of semivolatiles for quality control acceptance criteria.

---

**9.0 METHOD PERFORMANCE**

Refer to the applicable analytical SOP.

---

**10.0 APPLICABLE DOCUMENTS/REFERENCES**

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Methods 3510 and 3520 (current revisions), SW-846 Third Edition, Updates I, II, IIA, and IIB, Revised January 1995, US EPA.

Code of Federal Regulations (40 CFR), Part 136, Appendix A, Rev. July 1, 1995, Method 625.

---

**LIST OF TABLES AND FIGURES**

- Table 1 Summary of Method Modifications
- Figure 1 Example of Semivolatiles Logbook Page
- Figure 2 Example of Solvent/Reagent Lot Check Logbook Page
- Figure 3 LCS/Matrix Spike Component List
- Figure 4 Appendix Ix LCS/Matrix Spike Component List
- Figure 5 Copy of EPA Region I Memo Regarding Combination of Extracts for Method 625

**TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-502-03	METHOD 3510, current revision
Apparatus/Materials	<ol style="list-style-type: none"> <li>1) 250 mL amber bottle or flask</li> <li>2) 1.0 mL syringe</li> <li>3) short stem funnels</li> </ol>	<ol style="list-style-type: none"> <li>1) 250 mL Erlenmeyer flask</li> <li>2) 5.0 mL syringe</li> <li>3) drying columns</li> </ol>
Reagents		
Sample preservation/handling		
Procedures	<ol style="list-style-type: none"> <li>1) extract collection in amber bottle or Erlenmeyer flask</li> <li>2) Add surrogate/spike to sample in CLLE</li> <li>3) Extract for 3 minutes on mechanical shaker</li> <li>4) extract three times at pH <math>\geq</math> 11, then extract three times at pH <math>\leq</math> 2.</li> <li>5) extract dried using Na<sub>2</sub>SO<sub>4</sub> in short stem funnels</li> <li>6) Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer</li> <li>7) water bath temp 75-85 deg C</li> <li>8) no apparatus height specification for concentration on water bath</li> <li>9) sample removed from water bath when volume reaches ~6 mL</li> <li>10) N bath temp no higher than 39 deg C</li> </ol>	<ol style="list-style-type: none"> <li>1) extract collection in Erlenmeyer flask</li> <li>2) Add surrogate/spike directly to sample bottle</li> <li>3) Extract by shaking vigorously for 1 - 2 minutes with periodic venting</li> <li>4) extract three times at pH <math>\leq</math> 2, then extract three times at pH <math>\geq</math> 11.</li> <li>5) extract dried using Na<sub>2</sub>SO<sub>4</sub> in drying columns</li> <li>6) Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer</li> <li>7) water bath temp 15-20 deg C above solvent boiling temp</li> <li>8) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min</li> <li>9) sample removed from water bath when volume reaches 1 mL</li> <li>10) N bath temp 35 deg C</li> </ol>
QC - Spikes	<ol style="list-style-type: none"> <li>1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL</li> </ol>	<ol style="list-style-type: none"> <li>1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL</li> </ol>
QC - LCS	<ol style="list-style-type: none"> <li>1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL</li> </ol>	<ol style="list-style-type: none"> <li>1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL</li> </ol>

**TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

TABLE 1, continued

SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)

TOPIC	KATAHDIN SOP CA-502-03	METHOD 3520, current revision
Apparatus/Materials	1) short stem funnels	1) drying columns
Reagents		
Sample preservation/handling		
Procedures	<ol style="list-style-type: none"> <li>1) Add surrogate/spike to sample in CLLE</li> <li>2) Add approximately 500 - 600 mL of methylene chloride to the CLLE body</li> <li>3) CLLE for 22 ± 2 hours</li> <li>4) Extract dried using Na<sub>2</sub>SO<sub>4</sub> in short stem funnels</li> <li>5) Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer</li> <li>6) water bath temp 75-85 deg C</li> <li>7) no apparatus height specification for concentration on water bath</li> <li>8) sample removed from water bath when volume reaches ~6 mL</li> <li>9) N bath temp no higher than 39 deg C</li> </ol>	<ol style="list-style-type: none"> <li>1) Add surrogate/spike directly to sample bottle</li> <li>2) Add 300 - 500 mL of methylene chloride to the distilling flask of the extractor</li> <li>3) CLLE for 18 - 24 hours</li> <li>4) Extract dried using Na<sub>2</sub>SO<sub>4</sub> in drying columns</li> <li>5) Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer</li> <li>6) water bath temp 15-20 deg C above solvent boiling temp</li> <li>7) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min</li> <li>8) sample removed from water bath when volume reaches 1 mL</li> <li>9) N bath temp 35 deg C</li> </ol>
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL

**TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

TABLE 1, continued

SUMMARY OF METHOD MODIFICATIONS (METHOD 625)

<b>TOPIC</b>	<b>KATAHDIN SOP CA-502-03</b>	<b>METHOD 625</b>
Apparatus/Materials	See above tables for 3510 and 3520	See above tables for 3510 and 3520
Reagents		
Sample preservation/ handling		
Procedures	1) See above tables for 3510 and 3520 2) For 625 analysis, a new round bottom flask with 200 mL methylene chloride is attached to the CLLE body for second pH extraction	1) See above tables for 3510 and 3520 2) For 625 analysis, attach a clean distilling flask containing 500 mL of methylene chloride to the continuous extractor for second pH extraction
QC - Spikes	See above tables for 3510 and 3520	See above tables for 3510 and 3520
QC - LCS	See above tables for 3510 and 3520	See above tables for 3510 and 3520

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 1

EXAMPLE OF SEMIVOLATILES LOGBOOK PAGE

SM/SV SEP

Katahdin Analytical Services, Inc.  
Organic Extraction Log

Protocol: SW846/EPA Analytical Method: SW846-8270  
 Analysis: Semivolatile Organics EPA-825 or CLP  
 Date QC Started: 9-25-07 Extraction Technique: CLLE-1520  
 QC Expiration Date: 10 SEP-15-10  
 SURROGATE ID: SV-5V2163 Matrix: AQUEOUS  
 SURROGATE ID: SM-SV2171 SPIKE ID: SV-SV2166  
SV-SV2173 SPIKE ID: SM-SV2172  
 CLEAN-UP: GC screen, GPC, Florisil, Acid Wash, Other: pH Adjustments - 1st extraction: 2/1  
 Solvent Lot # Mech. E30666 2nd extraction: 3/2  
 All volumes in milliliters unless otherwise indicated. Have all samples been adjusted for pH? Yes

Case	Date	Lab	Sample ID	Volume	Sur. Vol.	Extr. Vol.	Flask Vol.	Date	Tray	Lab	Comments
1	9-25-07	AF	MS SAS373-7A	1000	1000	NR	1000	9-26-07	D4	GN	SV only
2			-4 S	1050					D5		SV/SV
3			-6A-7A	1040					D6		SV/SV
4			-8A-9A	1030					D7		emulsion
5			-10A-11A	1040					D8		emulsion
6			-12A-13A	1040					D9		
7			-14A-15A	1050					D10		emulsion
8			-16A-17A	970					E1		
9			-18A-19A	1050					E2		*

SM/SV SEP

Case	Date	Lab	Sample ID	Volume	Sur. Vol.	Extr. Vol.	Flask Vol.	Date	Tray	Lab	Comments
9											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											

TITLE: **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

---

FIGURE 2  
SOLVENT/REAGENT LOT CHECK LOGBOOK

SOLVENT:

LOT#:

DATE RECEIVED:

DATE CONCENTRATED:

CONCENTRATED BY:

PREP METHOD:

TRAY LOCATION:

ANALYZED BY:

PASS/FAIL:

TITLE: **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 3

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Benzyl alcohol
1,1-Biphenyl	Bis (2-chloroethoxy) methane
1,2,4-Trichlorobenzene	Bis (2-chloroethyl) ether
1,2-Dichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl phthalate
3,3'-Dichlorobenzidine	Dimethyl phthalate
3-Nitroaniline	Di-n-butylphthalate
4-Bromophenylphenyl ether	Di-n-octyl phthalate
4-Chloroaniline	Fluoranthene
4-Chlorophenylphenyl ether	Fluorene
4-Nitroaniline	Hexachlorobenzene
Acenaphthene	Hexachlorobutadiene
Acenaphthylene	Hexachlorocyclopentadiene
Acetophenone	Hexachloroethane
Aniline	Indeno (1,2,3-cd) pyrene
Anthracene	Isophorone
Atrazine	Naphthalene
Azobenzene	Nitrobenzene
Benzaldehyde	N-Nitrosodimethylamine
Benzidine	N-Nitroso-di-n-propylamine
Benzo (a) Anthracene	N-Nitrosodiphenylamine
Benzo (a) pyrene	Phenanthrene
Benzo (b) fluoranthene	p-toluidine
Benzo (ghi) perylene	Pyrene
Benzo (k) fluoranthene	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

**TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**

FIGURE 4

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepon
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitrobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

---

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

---

FIGURE 5

COPY OF EPA REGION I MEMO  
REGARDING COMBINATION OF EXTRACTS FOR METHOD 625

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION I  
Environmental Services Division  
60 Westview Street, Lexington, MA 02173-3185

MEMORANDUM

DATE: March 6, 1995

SUBJ: Analysis of Combined  
Extracts from Method 625

FROM: Arthur E. Clark, Chemist,  
Quality Assurance Office (EQA)

TO: James O'Dell, ATP Coordinator,  
QA Research Division, EMSL-CI

I am writing to request that you support the position within the Region I ESD that the extracts from EPA method 625 samples may be combined and analyzed together as long as the analysts can document their ability to do so successfully. You and I have discussed this previously. We have now been asked by a treatment authority to state our position formally.

As you know, method 625 for semivolatile organics requires that separate extractions be performed for the acid and base/neutral compounds. However, the method does not state that the extracts must be analyzed separately. Indeed, it is common practice among commercial laboratories to combine the two extracts for analysis. The RCRA and CERCLA Contract Lab Program protocols combine the extracts for analysis. If a program believes that combined analysis will compromise their objectives, the fractions should be analyzed separately. But, when neither fraction is compromised by the other, combined analysis is acceptable; the lab must prove that it can perform the analysis successfully.

One of our guidance documents<sup>1</sup> states that method 625 requires that the extracts be analyzed separately, citing two sections and two tables in the method. However, although they give instructions for analyzing the two fractions, they do not make any statement requiring separate analysis. The intent of the guidance document is to provide guidance for analyzing semivolatile samples when interferences are present.

While analyzing the acid and base/neutral extracts separately is one way to decrease interferences, we do not think that separate analysis should be imposed as an across-the-board requirement. There are many samples which can be analyzed successfully with the extracts combined.

I have spoken with William Telliard, Chief, Analytical Methods Staff, Engineering and Analysis Division, OST. While he does not support blanket approval for combined extract analysis, he believes that it is acceptable as long as the analysts document their ability to do so successfully.

<sup>1</sup>Guidance on Evaluation, Resolution, and Documentation of Analytical Problems Associated with Compliance Monitoring, EPA 821-B-93-001, p. 35 (June 1993).

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

Prepared By: Mike Thomas Date: 8/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: JCB Date: 10/25/00

QA Officer: Dorah J. Nadeau Date: 10-23-00

General Manager: Deann F. Kufan Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10-23-00	
02	Addition of SPE Procedure. Minor changes throughout. Added wording to sections 6 and 8.	LAD	013105	013105
03	Added separate QC for Pest. and PCB. Updated concentration procedure to reflect current practices. Changes in wording for clarification. Update Logbook page.	LAD	04/06	04/06

---

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

---

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

---

I acknowledge receipt of copy \_\_\_\_\_ of document **SOP CA-515-03**, titled **PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_\_\_ of document **SOP CA-515-03**, titled **PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

---

## 1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel for the preparation of aqueous samples prior to analysis for pesticides/PCBs by GC/ECD. It includes extraction of water samples by separatory funnel, continuous liquid-liquid, and solid phase extraction methods (EPA Methods 3510, 3520, and 3535A, current revisions).

### 1.1 Definitions

### 1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the extraction of aqueous samples for pesticides/PCBs analysis. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training".

It is the responsibility of all Katahdin personnel involved in the preparation of aqueous samples for pesticides/PCBs analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for the data.

It is the responsibility of the Supervisor to oversee that members of their group follow this SOP, that their work is properly documented and to initiate periodic review of the associated logbooks.

### 1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

---

analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

#### 1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Management Plan and Safety Manual.

---

## 2.0 SUMMARY OF METHOD

Pesticides/PCBs are extracted from aqueous samples using methylene chloride and separatory funnel, continuous liquid-liquid apparatus or Automated Extractor System (SPE), following EPA Methods 3510, 3520 and 3535A. The methylene chloride is exchanged with hexane for the final extract. Method detection limit studies must be performed annually for pesticides/PCBs using both extraction methods, if the extraction lab wishes to use either or both techniques. Method 3510 (separatory funnel) is generally preferred for pesticides/PCBs since organochlorine pesticides may dechlorinate if under elevated pH conditions for an extended period of time. (Section 3.2, Method 3510B, Rev. 2, 9/94)

---

## 3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates which are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves which have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

---

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

---

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be needed to minimize interferences.

---

#### 4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with the solvent to be used for extraction.

- 4.1 Separatory Funnel - 2000 mL capacity, Nalgene Teflon FEP separatory funnels with Nalgene Tefzel® screw-cap closures (or equivalent)
- 4.2 Concentrator tube - 10 mL, graduated
- 4.3 Evaporative flask - Kuderna-Danish, 500 mL capacity attached to concentrator with neck clips
- 4.4 Snyder column - Kuderna-Danish, three ball macro
- 4.5 Graduated cylinders - 100 mL, 1000 mL, or 2000 mL
- 4.6 Short Stem Funnels
- 4.7 250 mL amber collection bottles with Teflon-lined caps
- 4.8 12 mL and/or 16 mL glass vials with Teflon-lined caps
- 4.9 Continuous liquid-liquid extractors (CLLE) including body, 500 mL flat bottom boiling flask and Alhin condensers
- 4.10 Fluted Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)
- 4.11 Nitrogen evaporation apparatus.
- 4.12 Boiling chips - approximately 10/40 mesh, Teflon or selenized carborundum, 12 mesh (or equivalent).

---

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

---

- 4.13 Water bath - eight position concentric ring bath or equivalent, equipped with a calibrated thermometer.
- 4.14 Vials, 40 mL with PTFE – lined screw caps.
- 4.15 Glass separatory funnels, 125 mL.
- 4.16 Horizon SPE-DEX 4790 Automated Extractor System.
- 4.17 C-18 Speedisk.

---

**5.0 REAGENTS**

- 5.1 Laboratory reagent grade water - water in which an interferent is not observed at or above the PQL for any parameter of interest (carbon filtered ASTM Type II water or equivalent)
- 5.2 Sodium Hydroxide (10N) – Purchased from vendor, “Baker-analyzed”, or equivalent
- 5.3 Sodium Sulfate (ACS) - Granular, anhydrous. Bake at 400°C for 4 hours (may be done by vendor). Purify by rinsing three times with pesticide grade methylene chloride. Allow residual methylene chloride to evaporate before use. Stored in a Teflon capped glass bottle.
- 5.4 Sulfuric Acid Solution (1:1) - Add 500 mL concentrated sulfuric acid (certified ASC grade or better) slowly to 500 mL laboratory reagent grade water. Prepare as needed and store in a ground glass stoppered bottle.
- 5.5 Methylene Chloride (MeCL<sub>2</sub>) - Pesticide grade or better. Lot must be verified by concentrating 300-400 mL to 1.0 mL and evaluating by GC/MS.
- 5.6 Hexane - Pesticide grade or better. Lot must be verified by concentrating approximately 20-30 mL to 1.0 mL and evaluating by GC/ECD.
- 5.7 Methanol – ACS or HPLC grade, residue less than 1 mg/L.
- 5.8 Pesticide/PCB Surrogate spiking solution - Prepare a solution of decachlorobiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1.0 ug/mL ea in acetone. Store the solution at –10 to -20 °C in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

---

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

---

- 5.9 Pesticide Matrix Spike/Lab Control Sample spiking solution - Prepare a matrix spiking solution in pesticide grade methanol that contains all target analytes listed below:

ANALYTE	ug/mL
4,4'-DDT	0.5
4,4'-DDD	0.5
4,4'-DDE	0.5
Aldrin	0.5
Dieldrin	0.5
Endrin	0.5
Endrin Aldehyde	0.5
Endrin Ketone	0.5
Endosulfan I	0.5
Endosulfan II	0.5
Endosulfan Sulfate	0.5
alpha-BHC	0.5
beta-BHC	0.5
delta-BHC	0.5
gamma-BHC (Lindane)	0.5
Heptachlor	0.5
Heptachlor epoxide	0.5
Methoxychlor	0.5
alpha-Chlordane	0.5
gamma-Chlordane	0.5

- 5.10 PCB Matrix Spike/Lab Control Sample spiking solution - Prepare a matrix spiking solution in pesticide grade acetone that contains 5.0ug/ml ea of Aroclor® 1016/1260 mix (Restek catalog# 32039).
- 5.11 Store the spiking solutions at -10 to -20 °C in a Teflon sealed container. The solutions must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

---

**6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING**

Samples are collected in 1 L amber bottles and held at 4 (±2) °C until time of extraction.

---

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

---

Holding time for extraction of aqueous samples for Methods 3510 and 3520 is 7 days from date of sample collection, although the analyst should be aware that actual holding times employed might be project/program specific.

---

## 7.0 PROCEDURES

### SEPARATORY FUNNEL SAMPLE EXTRACTION

If an emulsion prevents acceptable recovery or client history indicates samples may demonstrate matrix interference, then samples should be extracted by continuous liquid-liquid extraction (CLLE).

- 7.1 Rinse all glassware three times with methylene chloride prior to use.
- 7.2 Label a 2 L Teflon separatory funnel and a 250 mL amber collection bottle clearly. Label should include laboratory sample number, matrix, analyte, and extraction date. Be sure that the detachable stopcocks are secured to the separatory funnels before adding samples.
- 7.3 Mark the sample level on the side of the sample bottle for later measurement of initial volume. Transfer the contents of the sample bottle to a 2 L separatory funnel.
- 7.4 Transfer 1 L of laboratory reagent grade water to a 2 L separatory funnel. This serves as a method blank for the extraction batch. A method blank must be prepared for every daily extraction batch of twenty or fewer samples.
- 7.5 Transfer 1 L of laboratory reagent grade water to a 2 L separatory funnel for each analysis to be performed (pesticide and/or PCB). This will serve as a Laboratory Control Sample (LCS). An LCS is required for every daily extraction batch of twenty or fewer samples and each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.
- 7.6 A matrix spike/matrix spike duplicate (MS/MSD) is to be prepared as requested by a client or, at a minimum, one pair per 20 samples or every 14 days and each analysis (refer to the logbook page, "date QC expires"). Transfer two additional 1 L aliquots of sample to 2 L separatory funnels for a matrix spike and matrix spike duplicate (MS/MSD) for each analysis. Note: Sufficient sample volume should be available without depleting all remaining sample aliquots.

---

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

---

- 7.7 Check the pH of the samples. If it is not between pH 5 and 9, adjust the pH with 10N sodium hydroxide or 1:1 sulfuric acid solution. Note the addition of NaOH or H<sub>2</sub>SO<sub>4</sub> in the extraction logbook.
- 7.8 Using a gas-tight syringe, add 1.0 mL of surrogate spiking solution to all samples the blank, LCS/LCSD(s) and MS/MSD(s), if performed.
- 7.9 Using a gas-tight syringe, add 1.0 mL of pesticide or PCB matrix spiking solution to the appropriate LCS, LCSD, MS and MSD if performed.
- 7.10 To each empty sample bottle add 60 mLs of methylene chloride, rinse the bottle and transfer the solvent into the appropriate separatory funnel. Add 60 mL of methylene chloride directly to the blank and LCS/LCSD(s).
- 7.11 Ensure that each screw cap is secured tightly to the separatory funnel to prevent leaks. Shake briefly and vent in hood to release pressure. Extract the sample by shaking the funnel on mechanical shaker for 3 minutes. Allow phases to separate for at least 10 minutes. Drain the methylene chloride layer into the 250 mL amber collection bottle.
- 7.12 If an emulsion forms, mechanical techniques must be employed to achieve maximum separation and solvent recovery. Such means include swirling and centrifugation and draining through a small separatory funnel. In certain instances, transferring the entire sample into a continuous liquid-liquid extractor may be the only alternative. If any such techniques are used, they must be noted in the extractions logbook.
- 7.13 Add a second 60 mL aliquot of methylene chloride to the separatory funnel and extract for the second time (see 7.10 - 7.12). Collect the methylene chloride layer in the same 250 mL amber collection bottle.
- 7.14 Repeat the extraction for a third time as described in 7.13.
- 7.15 Measure the initial volume of the sample by filling the sample bottle with water up to the level marked earlier. Record the volume and any notable characteristics (e.g. color, sediment present, odor) in the extraction logbook.
- 7.16 Proceed to Section 7.53 for extract concentration procedures.

**CONTINUOUS LIQUID-LIQUID SAMPLE EXTRACTION (CLLE)**

- 7.17 Set up the CLLE apparatus. All glassware should be rinsed three times with methylene chloride and the extract flasks properly labeled.

---

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

---

- 7.17 Add approximately 300 - 400 mL of methylene chloride to the CLLE body.
- 7.19 Add 1 L laboratory reagent grade water to a CLLE body. This is the method blank for this extraction batch. Be sure that no water leaks into the round bottom flask. A method blank is required for every extraction batch of twenty or fewer samples.
- 7.20 Prepare an LCS for every daily extraction batch of twenty or fewer samples and each analysis (pesticide and/or PCB). Add 1 L of laboratory reagent grade water to a CLLE body. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.
- 7.21 Mark the sample levels on the sample bottles. Transfer the samples to the CLLE bodies.
- 7.22 Check the pH of the samples. If it is not between pH 5 and 9, adjust the pH with 10N sodium hydroxide or 1:1 sulfuric acid solution. Note the addition of NaOH or H<sub>2</sub>SO<sub>4</sub> in the extraction logbook.
- 7.23 Transfer two 1 L portions of a sample to CLLE bodies for each analysis for preparation of a matrix spike/matrix spike duplicate if required. An MS/MSD is required if requested by the client or per 20 samples or every 14 days, whichever occurs first, and each analysis. (Refer to the logbook page, "date QC expires").
- 7.24 For each sample, rinse the original sample container with approximately 30 mL of methylene chloride. Add this rinse to the CLLE body.
- 7.25 Determine the initial volume of the sample as described in 7.15 and record it and any notable characteristics in the extraction logbook.
- 7.26 Add 1.0 mL of the Pesticide/PCB Surrogate Spike to each sample including the blank, LCS/LCSD and MS/MSD, if performed.
- 7.27 Add 1.0 mL of Pesticide or PCB Matrix Spike to the appropriate LCS/LCSD and MS/MSD pair, if performed, and stir.
- 7.28 Attach cooling water Allihn condensers, after first rinsing each 45/50 joint with methylene chloride. Turn on the heating mantles and allow the samples to extract for 18 ± 2 hours. Turn off the mantles and let samples cool.
- 7.29 Proceed to Section 7. 53 for sample extract concentration procedures.

---

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

---

### EXTRACTION WITH AUTOMATED EXTRACTOR SYSTEM (SPE)

Alternatively, samples may be extracted using the Horizon Automated Extractor System (Figure 2)

#### Purging the Extractor Vessels

- 7.30 Check and fill all four solvent bottles (methanol, acetone, laboratory reagent grade water and hexane) as needed. Check and empty the two waste containers as needed.
- 7.31 Turn on nitrogen tank to 60 psi. Turn the instrument pressure on top of the controller to 50 psi. Turn the solvent bottle pressure to 10 psi.
- 7.32 Turn on the Horizon controller (switch in the back).
- 7.33 Check the lubrication oil on the air pump. Fill as needed. Turn the air pump on.
- 7.34 Attach the 40 ml collection vials beneath the disk holder platform of the extractors. Place the adaptors on the C-18 speedisks and place the speedisks on top of the disk holder platform. There should be roughly 1 cm separating the speedisk from the extractor downtube. Do not use a fresh speedisk. Use a previously discarded one or one reserved for this purpose.
- 7.35 Check to be sure that all extractors have empty sample bottles loaded on top. If not, use a Horizon cap on a one liter empty bottle and firmly place the bottlenose down into the extractor.
- 7.36 Press *select* on the control panel to designate an extractor (1, 2 or "." for both), then press *enter*.
- 7.37 Press 1, enter to select purge method. Once the method is loaded, start the extractors by pressing the *start* buttons on the individual extractors. The red LED will blink when the method is complete. You should collect 5-10 mls of solvent.
- 7.38 Repeat this process 2-3 times before using the Horizon autoextractor.

### ANALYSIS OF SAMPLES WITH AUTOEXTRACTOR

- 7.39 Label a 40 ml vial with each sample to be extracted. Attach the 40 ml collection vials to the extractor. Replace the speedisk with a fresh C-18 speedisk.

---

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

---

- 7.40 Mark the volume level of liquid in each sample on the outside using a grease pencil. Remove the cap and add 1.0 mL of surrogate, recap and shake well. Remove the cap and add 5.0 mL of 1:1 H<sub>2</sub>SO<sub>4</sub>.
- 7.41 Add 1 L laboratory reagent grade water to 1 L amber bottle. This is the method blank for this batch. A method blank is required for every extraction batch of twenty or fewer samples.
- 7.42 Prepare an LCS for every daily extraction batch of twenty or fewer samples and each analysis. Add 1 L of laboratory reagent grade water to a 1 L amber bottle. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required to meet client specific or program specific requirements. This information will be disseminated from the project manager or department manager.
- 7.43 Add 1.0 mL of Pesticide/PCB surrogate spike to each sample including the blank, LCS and MS/MSD, if required. Recap samples and shake well.
- 7.44 Add 1.0 mL of pesticide and/or PCB matrix spike to the appropriate LCS and MS/MSD samples. Recap and shake well.
- 7.45 Remove cap and add 5.0 mL of 1:1 H<sub>2</sub>SO<sub>4</sub> to each sample including the blank, LCS and MS/MSD set immediately prior to extracting the sample.
- 7.46 Remove the cap from each sample bottle and cover with tin foil. Screw a Horizon adapter cap over the tin foil. Invert the bottle and check for leaks.
- 7.47 Load the sample bottle on the holder and twist  $\frac{3}{4}$  of a rotation. Stop twisting when air bubbles rise to the top of the sample bottle. Do not twist completely around. The foil may loosen and jam the valve.
- 7.48 Press *select* on the control panel to designate an extractor (1, 2 or "." for both), then press *enter*.
- 7.49 Type in 8081 for the method and press enter. Once the method is loaded, start the extractors by pressing the *start* buttons on the individual extractors. The red LED will blink when the method is complete. The extract will be collected in the 40 ml vial.
- 7.50 If the 90 mm Empore disks are used due to significant particulate matter in the sample, the 125 or 250 mL Erlenmeyer flask with 19/22 ground glass joint must be used. The analyst must determine this, with the supervisor's assistance, if needed. Refer to the operator's manual for proper assembly of the Empore Disk Holder.

---

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

---

- 7.51 Pour each extract into a 125 ml glass separatory funnel, pre-rinsed with hexane. Rinse each 40 ml vial with hexane. Pour the bottom layer (acetone) from the separatory funnel back into the 40 ml vial and discard. Pour the remaining extract (hexane) into a 125 ml Erlenmeyer flask. Rinse the separatory funnel with hexane and combine with that in the round bottom flask.
- 7.52 Sample is now ready to reduce to 10 mL final volume.

### CONCENTRATION OF WATER SAMPLE EXTRACTS

- 7.53 Rinse the K-D glassware (flask, concentration tube, funnel and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride (or hexane for samples extracted with the Autoextractor) before assembling. Add two boiling chips to the K-D. Insert fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride (hexane for samples extracted with the Autoextractor). Place the assembled K-D's under the funnels.
- 7.54 For methylene chloride extracts, fill funnel with Hexane and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only.
- 7.55 Transfer the methylene chloride or hexane extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract bottle three times with ~ 2 – 3 mLs of methylene chloride (or hexane for samples extracted with the Autoextractor). Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride (or hexane for samples extracted with the Autoextractor) and allow to drain.
- 7.55 Transfer the labels from the collection bottles or round bottom flasks (from the CLLE extraction) to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride (or hexane for samples extracted with the Autoextractor).
- 7.56 Place the K-D in a hot water bath (85-90°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 2 mL, remove the K-D from the water bath. Allow the

---

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

---

K-D to cool for 10 minutes. Rinse the Snyder column lower joint with  $\approx$  1 mL of hexane. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with  $\approx$  1 mL hexane.

- 7.57 Reduce the extracts to  $\approx$  10 mL using Nitrogen blow-down apparatus then proceed to section 7.54. The bath temperature must be no higher than the boiling point of the solvent (45 °C for hexane). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with  $\approx$ 1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N<sub>2</sub> sparging needle closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook. Transfer extract to a 12 or 16 mL vial. Using a reference vial for volume comparison, adjust the final extract volume to 10 mL by rinsing sides of tube with hexane and transferring rinsings to vial.
- 7.59 Transfer the label from the concentrator tube to the vial. Store extract vials at a temperature of  $4 \pm 2$  °C until ready for analysis. Indicate in the extractions logbook the box number and "tray location" of the individual extract vials.
- 7.60 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. All sample extracts for 8081 pesticide analysis do not undergo further cleanup unless requested by the client. All sample extracts for combined 8081/8082 analyses must be split. Prior to splitting, mix contents of vial well. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

---

## 8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency

---

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

---

must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

A method blank must be extracted for each and every item listed below:

- Each day of extraction (24 hours midnight - midnight)
- Each extraction method
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticide and/or PCB) to be performed

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

---

## 9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

---

## 10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Methods 3510C and 3520C, USEPA SW-846, Third Edition, Final Update III, December 1996.

---

### LIST OF TABLES AND FIGURES

Table 1	SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)
Table 2	SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)
Figure 1	Example of Runlog Page
Figure 2	Horizon Autoextractor System Diagram

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-515-03	METHOD 3510, current revision
Apparatus/Materials	1) micro-Snyder not used 2) 12 or 16 mL vials used for final extract 3) 250 mL amber bottle or flask used 4) 1.0 mL syringe 5) short stem funnels	1) solvent exchange in macro K-D 2) 2 mL vials used for final extract 3) 250 mL Erlenmeyer flask 4) 5.0 mL syringe 5) drying column
Reagents		
Sample preservation/handling	1) entire contents of 1 L sample bottle transferred to separatory funnel	1) one liter graduated cylinders used to transfer initial sample volume to separatory funnel
Procedures	1) extract collection in amber bottle or Erlenmeyer flask 2) extract dried using Na <sub>2</sub> SO <sub>4</sub> in short stem funnels 3) hexane added directly to K-D body prior to sample addition	1) extract collection in Erlenmeyer flask 2) extract dried using Na <sub>2</sub> SO <sub>4</sub> in drying columns 3) solvent exchange via large K-D with 50 mL hexane

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

TABLE 2

SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)

TOPIC	KATAHDIN SOP CA-515-03	METHOD 3520, current revision
Apparatus/Materials	1) short-stem funnels 2) micro-Snyder not used 3) 12 or 16 mL vials used for final extract	1) drying columns 2) solvent exchange in macro K-D 3) 2 mL vials used for final extract
Sample preservation/handling	1) entire contents of 1 L sample bottle transferred to CLLE	1) one liter graduated cylinders used to transfer initial sample volume to CLLE
Procedures	1) CLLE for 18 ± 2 hours 2) extract dried using Na <sub>2</sub> SO <sub>4</sub> in short stem funnels 3) hexane added directly to K-D body prior to sample addition	1) CLLE for 18-24 hours 2) extract dried using Na <sub>2</sub> SO <sub>4</sub> in drying columns 3) solvent exchange via macro K-D with 50 mL of hexane

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

FIGURE 1

EXAMPLE OF LOGBOOK PAGE

PCB + TCLP Verst  
SEP

KATAHDIN ANALYTICAL SERVICES, INC.  
ORGANIC EXTRACTIONS LOG - AQUEOUS PESTICIDE/PCB

Extraction Method: SW846 3520 (CLLE) SW846 3510 (SEP)  SW846 3535 (SPE)  
Analytical Method: SW846 8081  SW846 8082 EPA 808 CLP OLM03.1 CLP OLM04.2 CLP OLC2.1  
Date QC Started: 3/3/06 QC Expiration Date: 4-14-06  
Surrogate ID: 6C0315 Spike ID: 6C0303 Spike ID: 6C0293  
Solvent Lot #: MFC1 C03H16 Solvent Lot #:

Date Extracted	Est. Inlt.	Sample ID	Initial Vol. mL	Sur. Vol.	Spike Vol.	Fraction		Final Vol. mL	Date Conc.	Trey Location	Initials	Clean-Up				Comments
						Post	Pre					GPC	Flt	Acid Wash	Other	
3/3/06	GM	W626854-1	1000	1mL	NR	✓		10mL	3/3/06	PF665 F2	KF					R4785
		-2			1mL	✓				F3						
		-3				✓				F4						
		-4	200		NR	✓				F5						1077664
4/4/06	GM	W626746-1	1000	1mL	NR	✓		10mL	4/4/06	PF666 A5	KF					R47319
		-2			1mL	✓				A6						Pos. Sample sp. 6c
		-3				✓				A7						
4/5/06	GM	W626788-1	1000	1mL	NR	✓		10	4-6-06	PF666 C5	EC					R47373
		-2			1mL	✓				C6						
		-3				✓				C7						
4/4/06	TR	W627077-1 W627078-1	1000	1mL	NR	✓		10mL	4-15-06	PF667 E10	GM					REF: R47144 R4724750
		W627077-2			1mL	✓				E11						
		W627078-2				✓				E12						



Date Extracted	Est. Inlt.	Sample ID	Initial Vol. mL	Sur. Vol.	Spike Vol.	Fraction		Final Vol. mL	Date Conc.	Trey Location	Initials	Clean-Up				Comments
						Post	Pre					GPC	Flt	Acid Wash	Other	
4-7-06	GM	W61246-8	200	1mL	NR	✓		10mL	3-31-06	PF665 EC	KF					TCLP
4-4-06	GM	W61396-1	990	1mL	NR	✓		10mL	4/4/06	PF666 A6	KF					
4-5-06	GM	W61522-1	1060	1mL	NR	✓		10mL	4-6-06	PF666 CR	EC					
		-2	1060			✓				C9						
4/4/06	TR	W61627-10	1000	1mL	NR	✓		10mL	4-25-06	PF667 E1	GM					

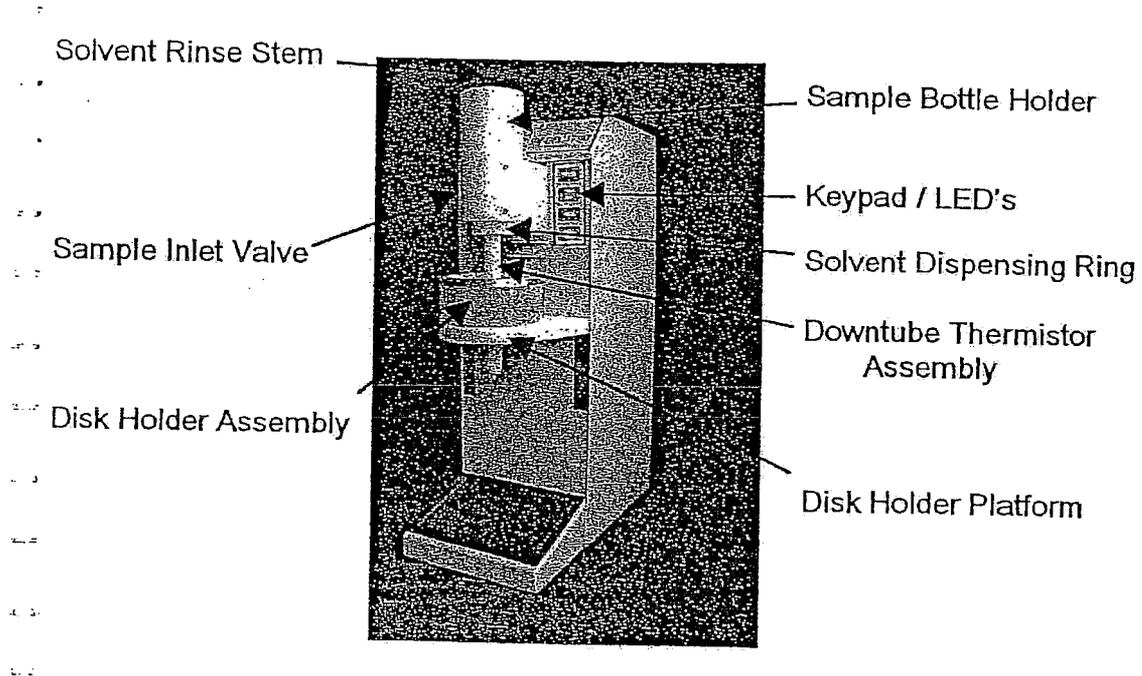
Reviewed By \_\_\_\_\_ Date \_\_\_\_\_

---

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

---

FIGURE 2  
HORIZON AUTOEXTRACTOR SYSTEM DIAGRAM



TITLE: pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.

Prepared By: Wet Chemistry Date: 8/96

Approved By:

Group Supervisor: Keith Tangney Date: 2/13/01

Operations Manager: John C. Burton Date: 2/13/01

QA Officer: Dorothy J. Nadeau Date: 2/13/01

General Manager: Debra F. Keenan Date: 2/12/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03 9045C	Format changes, added pollution prevention, database and operation of Accumet pH meter and calibration.	DN	2/13/01	2/13/01
04 9045C	Addition to scope and Application to include reference for 9040B use when aqueous phase is >20%.	DN	8-27-02	8-27-02
05 9045C	added KIMS minor changes throughout added wording to sect. 6 New fig. 1 and 2	LAD	12/01/04	12/01/04
06 9045C	Added SW-846 reference. Minor formatting changes throughout.	LAD	03/07	03/07

---

**TITLE: pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

---

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

---

I acknowledge receipt of copy \_\_\_\_\_ of document **CA-709-05**, titled **pH Concentration Measurements in Soil Matrices - SW846 Method 9045**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

KATAHDIN ANALYTICAL SERVICES, INC.  
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy \_\_\_\_\_ of document **CA-709-05**, titled **pH Concentration Measurements in Soil Matrices - SW846 Method 9045**.

Recipient: \_\_\_\_\_ Date: \_\_\_\_\_

---

**TITLE: pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

---

## **1.0 SCOPE AND APPLICATION**

The purpose of this SOP is to describe the procedures and techniques followed by Katahdin Analytical Services, Inc. personnel to determine the pH of soils and waste samples in accordance with SW846 method 9045 (current promulgated revision). Method 9045 is an electrometric procedure for measuring pH in soils and waste samples. Wastes may be solids, sludges, or non-aqueous liquids. If water is present, it must constitute less than 20% of the total volume of the sample. If the aqueous phase is greater than 20%, pH determination should be performed in accordance with EPA method 9040 (current promulgated revision). Refer to the current revision of Katahdin SOP CA-708, pH Concentration Measurements in Aqueous Samples.

The procedures in this SOP are applicable to all non-CLP pH measurements performed for all soil matrices analyzed in the laboratory.

### **1.1 Definitions**

pH - A measure of the acidity or alkalinity of a solution, defined as  $-\log [H^+]$ .

### **1.2 Responsibilities**

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of pH in solids by EPA Method 9045. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the determination of pH concentration measurements in solid matrices to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for pH data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

### **1.3 Safety**

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method

**TITLE: pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

---

has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety datasheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Chemical Hygiene Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

**1.4 Pollution Prevention/Waste Disposal**

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Chemical Hygiene Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

---

**2.0 SUMMARY OF METHOD**

A representative aliquot of sample, measured in grams, is mixed with an equivalent volume of laboratory reagent grade water, measured in mL. The solution is allowed to settle, and the pH of the standing water (decanted) is determined electrometrically.

---

**3.0 INTERFERENCES**

3.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode. Strong acid solutions, with a true pH of <1, may give incorrectly high pH measurements.

3.2 Temperature fluctuations will cause measurement errors.

---

**TITLE: pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

---

- 3.3 Errors will occur when the electrodes become coated with an oily material. See section 7.18 for special cleaning instructions.
- 

**4.0 APPARATUS AND MATERIALS**

- 4.1 pH meter, Accumet Model 20 or equivalent with Automatic Temperature Compensation (ATC)
- 4.2 Glass beakers, 25 mL and 400 mL
- 4.3 25 mL dose cups
- 4.4 Teflon coated stir-bars
- 4.5 Stir-bar retriever
- 4.6 Magnetic stirplate
- 4.7 Shaker, 12 place
- 4.8 Analytical balance, capable of weighing to 0.1 g
- 

**5.0 REAGENTS**

- 5.1 Buffer solutions (pH 4.0, 6.0, 7.0, 8.0, 10.0, 12.0)
- 5.2 Laboratory reagent grade water (Lab Water)
- 

**6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING**

Samples are collected in soil jars and stored at 4 ( $\pm 2$ ) °C until analyses. Since there are no published holding times for this method, sample analysis should be performed as soon as possible.

---

**7.0 PROCEDURES**

**SAMPLE PREPARATION**

---

**TITLE: pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

---

- 7.1 Mix samples thoroughly. Discard any foreign objects such as sticks, leaves and rocks. Decant any standing liquid. Using the balance, weigh out 20.0 g of sample into a 400 mL glass beaker. Record weight in pH logbook (Figure 1).
- 7.2 Add 20 mL of Laboratory reagent grade water to the sample. Cover the top of the beaker with parafilm.
- 7.3 Place the sample on the shaker and allow it to shake, at medium speed, for one hour.
- 7.4 After one hour, remove the sample from the shaker and allow it to settle for one hour.
- 7.5 After one hour, decant the standing liquid into a 25 mL beaker. If no standing liquid is present, add sufficient laboratory reagent grade water to result in standing water, cover with parafilm, and repeat steps 7.3 through 7.5.
- 7.6 Record total volume of laboratory reagent grade water added to sample in pH logbook. If volume of laboratory reagent grade water (in mL) added to sample exceeds the initial gram weight of the sample, flag sample data in pH logbook with reason for addition of excess laboratory reagent grade water (eg. minimum volume of water required in order to cover pH probe).

#### CALIBRATION OF PH METER

- 7.7 NORMAL RANGE CALIBRATION (pH range 3.5- 10.5)
  - 7.7.1 Meter should be calibrated daily. As described in the following steps, conduct a two-point calibration with pH buffers 4 and 10. Perform a calibration check using pH 7 buffer. The source/lot number of each solution at the time of analysis must be recorded in the logbook (Figure 1).
  - 7.7.2 Dispose of buffer solutions used the previous day (see Waste Disposal, section 1.4). Put about 20 mL of the appropriate buffer solution into new dose cups. Place a tiny stir bar in each new cup.
  - 7.7.3 Rinse probe (i.e., electrode) with laboratory DI laboratory reagent grade water. Gently blot dry with kimwipe.
  - 7.7.4 Place pH 4 buffer on stir plate. Turn stir plate on so that the stir bar spins without creating a vortex. Push 1 key (meaning add a standard). Then push 4 (calibrate). Record the value in the pH logbook.
  - 7.7.5 Remove pH 4 buffer. Rinse probe. Blot dry.

---

**TITLE: pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

---

- 7.7.6 Repeat step 7.7.4 with the pH 10 buffer. Record the value in the pH logbook. Remove pH 10 buffer. Rinse and dry probe.
- 7.7.7 With the pH 7 buffer, repeat step 7.7.4, but DO NOT press any keys as this reading is a calibration check. Record reading in pH logbook. Results must be within 0.05 pH units of the true value for analysis to proceed.

**NOTE:** If buffer readings are not within 0.05 pH units of expected values (4.00, 7.00 and 10.00) the electrode may need cleaning. Rerun and enter in pH lab notebook that meter was recalibrated with the pH 4 and 10 buffers, with the pH 7 buffer used as a calibration check. Also record any maintenance performed.

## 7.8 LOW RANGE CALIBRATION

- 7.8.1 If pH of a sample as is less than 3.5, the instrument must be recalibrated using buffers that bracket the expected pH of the sample, as described below. The source/lot number and temperature of each solution at the time of analysis must be recorded in the logbook (Figure 1).
- 7.8.2 Rinse probe (i.e., electrode) with laboratory reagent grade water.
- 7.8.3 . Gently blot dry with kimwipe.
- 7.8.4 Place pH 2 buffer on stir plate. Turn stir plate on so that the stir bar spins without creating a vortex. Push 1 key (meaning add a standard). Then push 4 (calibrate). Record the value in the pH logbook.
- 7.8.5 Remove pH 2 buffer. Rinse probe. Blot dry.
- 7.8.6 Repeat step 7.8.3 with the pH 7 buffer. Record the value in the pH logbook. Remove pH 7 buffer. Rinse and dry probe.
- 7.8.7 With the pH 4 buffer, repeat step 7.8.3, but DO NOT press any keys as this reading is a calibration check. Record reading in pH logbook. Results must be within 0.05 pH units of the true value for analysis to proceed.

**NOTE:** If buffer readings are not within 0.05 pH units of expected values (2.00, 4.00 and 7.00) the electrode may need cleaning. Rerun and enter in pH lab notebook that meter was recalibrated with the pH 2 and 7 buffers, with the pH 4 buffer used as a calibration check. Also record any maintenance performed.

## 7.9 HIGH RANGE CALIBRATION

---

**TITLE: pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

---

- 7.9.1 If pH of a sample as determined in Sections 7.10 through 7.17 is greater than 10.5, the instrument must be recalibrated using buffers that bracket the expected pH of the sample, as described below. The source/lot number and temperature of each solution at the time of analysis must be recorded in the logbook (Figure 1).
- 7.9.2 Rinse probe (i.e., electrode) with laboratory reagent grade water. Gently blot dry with kimwipe.
- 7.9.3 Place pH 7 buffer on a stirplate. Turn stir plate on so that the stir bar spins without creating a vortex. Push 1 key (meaning add a standard). Then push 4 (calibrate). Record the value in the pH logbook.
- 7.9.4 Remove pH 7 buffer. Rinse probe. Blot dry.
- 7.9.5 Repeat step 7.9.3 with the pH 12 buffer. Record the value in the pH logbook. Remove pH 12 buffer. Rinse and dry probe.
- 7.9.6 With the pH 10 buffer, repeat step 7.9.3, but DO NOT press any keys as this reading is a calibration check. Record reading in pH logbook. Results must be within 0.05 pH units of the true value for analysis to proceed.

**NOTE:** If buffer readings are not within 0.05 pH units of expected values (7.00, 10.00 and 12.00) the electrode may need cleaning. Rerun and enter in pH lab notebook that meter was recalibrated with the pH 7 and 12 buffers, with the pH 10 buffer used as a calibration check. Also record any maintenance performed.

#### ANALYSIS OF SAMPLES

- 7.10 Sample analysis may proceed once the meter has been calibrated for the day with two buffers that bracket the expected pH of the sample, and after checking the calibration with a mid-range solution between the two buffers used for calibration of the meter (refer to sections 7.7, 7.8, and/or 7.9 as applicable).
- 7.11 For the range being used, rerun the appropriate calibration check point as the laboratory control sample (LCS) for the analytical batch. An LCS is required at the beginning of every batch of twenty or fewer samples.
- 7.12 Record date, time and initials for this analytical session.
- 7.13 The decanted samples should be equilibrated to room temperature prior to analysis (i.e., at the same temperature as the calibration buffers,  $\pm 2$  °C). A more accurate pH

---

**TITLE: pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

---

reading will be achieved when the buffers and the samples are at the same temperature. However, the Accumet® pH meter is equipped with automatic temperature compensation (ATC) for when samples and buffers are not at the same temperature. Refer to the Accumet® Model 20 pH/Conductivity Meter operating Instructions, #300143.3 (Revision C) for information on the ATC probe.

- 7.14 Pour about 25 ml of the supernatant into a clean dose cup. Place a tiny stir bar in cup. Place on stir plate, turn on stir plate and immerse probes.
- 7.15 When meter locks, record value displayed.
- 7.16 Rinse probe with laboratory reagent grade water and blot dry between samples.
- 7.17 Place probe in pH 4 buffer solution to store until next analysis.

#### CLEANING PROCEDURE

- 7.18 Only if an electrode becomes coated with an oily material that will not rinse free, the electrode can either (refer to instrument manual):
  - be cleaned with an ultrasonic bath, or
  - be washed with detergent, rinsed several times with laboratory reagent grade water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with laboratory reagent grade water.

#### REPORTING OF RESULTS

- 7.19 All pH measurements less than 10.0 are to be reported using two significant figures.  
Examples:     2.46 = 2.5  
                  6.32 = 6.3  
                  9.94 = 9.9
- 7.20 All pH measurements which are at or greater than or round up to 10.0 are to be reported to three significant figures.  
Examples:     9.95 = 10.0  
                  12.25 = 12.3  
                  13.76 = 13.8  
                  11.95 = 12.0

---

**TITLE: pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

---

- 7.21 When a sample duplicate is analyzed, both the original result and duplicate result are recorded in the pH logbook; however, the original sample result is to be reported to the client.
- 7.22 After completion of each test, the logbook must be signed and dated by the person performing the test. All unused lines are to be "z-ed" out and initialed and dated.
- 7.23 The sample data results, with any appropriate notations, are entered into KIMS by the analyst. A batch sheet is generated (Figure 2). Raw data and batch sheets are reviewed for completeness and accuracy by the Inorganic Department Manager or other qualified designee.
- 7.24 All batch sheets and copies of the raw logbook data are filed with the Inorganic Department Manager for approximately 3 months, for reference by analysts. Prior data are archived.

---

**8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA**

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations, remaining sample volume and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

- 8.1 One sample duplicate is to be analyzed per batch or every 10 sample analyses.

---

**TITLE: pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

---

- 8.1.1 Acceptance criteria for duplicates is a difference of less than or equal to 20% relative percent difference between sample and duplicate results.
- 8.1.2. If criterion is not met, check calibration and reanalyze sample in duplicate.
- 8.2 One Laboratory Control Sample (LCS) is to be analyzed per batch or every 20 samples.
  - 8.2.1 The LCS must be within 90-110% recovery for analysis to proceed.
  - 8.2.2 If criteria are not met, recalibrate.

---

**9.0 METHOD PERFORMANCE**

Refer to method 9045.

---

**10.0 APPLICABLE DOCUMENTS/REFERENCES**

“Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods”, SW846, third Edition, Final Update III, December 1996, Method 9045C.

---

**LIST OF TABLES AND FIGURES**

Table 1	QC Requirements
Table 2	Summary of Method Modifications
Figure 1	Example of pH - Soils Logbook Page
Figure 2	Example of Batch Sheet for pH

**TITLE: pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.**

TABLE 1  
 QC REQUIREMENTS

Analytical Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW9045	PH (soil)	2-point calibration with pH buffers with a midrange cal. check	Once per day	± 0.05 pH units for every buffer	If calibration is not achieved, check meter, buffer solutions, and probe; replace if necessary; repeat calibration
		LCS	One per batch of twenty or fewer samples	90-110%R	Correct problem, recalibrate
		Sample duplicate	One sample duplicate per every ten field samples	RPD ≤20	(1) Investigate problem and reanalyze sample in duplicate (2) If RPD is still unacceptable, report original result with notation or narration.

TITLE: pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.

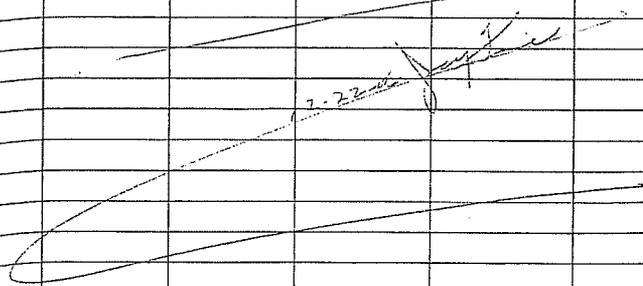
TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-709-06	METHOD SW846 9045, current revision
Apparatus/Materials		
Reagents		
Sample preservation/handling		
Procedures	1) Shake, at medium speed, for one hour. 2) Add more liquid after shaking and settling if there is no standing liquid left. 3) All buffers and samples are analyzed at room temperature. PH meter is equipped with automatic temperature compensation.	1) Continuously stir the suspension for five minutes. 2) No guidance for samples with no standing liquid left. 3) Report both pH and temperature at the time of analysis.
QC – Spikes		
QC – LCS		
QC - Accuracy/Precision		

TITLE: pH CONCENTRATION MEASUREMENTS IN SOIL MATRICES - SW 846 METHOD 9045.

FIGURE 1  
 EXAMPLE OF pH LOGBOOK PAGE

KATAHDIN ANALYTICAL SERVICES, INC.						
CORROSIVITY pH						
pH SOIL						
SW 846 9045A			SOW OLM01.8			
CALIBRATION STDS:		CALIBRATED TO:		LOT NO:		
pH 7.00		7.00		S.L. 2217		
pH 4.00		4.00		S.L. 2014		
pH 5.00						
pH 10.00		10.01		S.L. 2216		
pH 8.00						
pH 12.00		12.00		S.L. 2176		
4635229 R58891						
LAB SAMPLE ID	ANALYSIS TIME	SAMPLE VOL (mL)	SAMPLE WEIGHT(g)	pH	REPORTED pH	
LCS WG 35229-1	1550	20.0	20.000	7.00	7.0	
WW 6750-1B	1550	20.0	20.333	9.27	9.3	
DUP. WG 35229-2	1552	20.0	20.352	9.37	9.4	
L -2A	1554	20.0	20.418	10.76	10.8	
WW 6752-1B	1556	20.0	20.316	10.04	10.0	
L -2B	1558	20.00	20.178	7.40	7.4	
						
ANALYST: <i>[Signature]</i>				DATE: 12-22-06		
CHECKED BY: <i>[Signature]</i>				DATE: 12-28-06		



**APPENDIX B**

**UFP-QAPP WORKSHEETS**

Project-Specific QAPP

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
Site Location: Groton, Connecticut

Revision Number: 0  
Revision Date: October 2007

**QAPP Worksheet #1**  
**Title and Approval Page**

Document Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

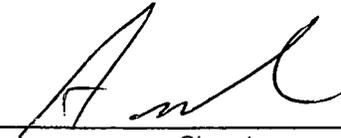
Lead Organization: Naval Facilities Engineering Command Mid-Atlantic (NAVFAC Mid-Atlantic)

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

Preparer's Address, Telephone Number, and E-mail Address: 661 Andersen Drive, Pittsburgh, PA 15220, 412-921-8433, Aaron.Bernhardt@ttnus.com

Preparation Date (Day/Month/Year): 12-Oct-2007

Investigative Organization's Project-Manager/Date: \_\_\_\_\_

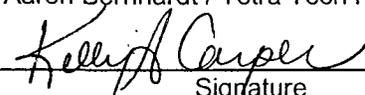


Signature

Printed Name/Organization:

Aaron Bernhardt / Tetra Tech NUS, Inc.

Investigative Organization's Project QA Officer/Date: \_\_\_\_\_



Signature

Printed Name/Organization:

Kelly Carper / Tetra Tech NUS, Inc.

Lead Organization's Project Manager/Date: \_\_\_\_\_

Signature

Printed Name/Organization:

Val Jurka, P.E. / NAVFAC Mid-Atlantic

Approval Signatures:

\_\_\_\_\_

Signature

Printed Name/Title/Date

\_\_\_\_\_

Approval Authority

\_\_\_\_\_

Signature

Printed Name/Title/Date

\_\_\_\_\_

Approval Authority

Other Approval Signatures:

\_\_\_\_\_

Signature

\_\_\_\_\_

Printed Name/Title/Organization/Date

Project-Specific QAPP

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
Site Location: Groton, Connecticut

Revision Number: 0  
Revision Date: October 2007

**QAPP Worksheet #2 -- QAPP Identifying Information**

Site Number/Code: Area A Wetland – Site 2B  
Operable Unit: N/A  
Contractor Name: Tetra Tech NUS, Inc.  
Contractor Number: N62472-03-D-0055  
Contract Title: CLEAN IV  
Work Assignment Number: CTO 439

1. Identify guidance used to prepare QAPP:  
Region 1 QAPP Guidance and UFP QAPP
2. Identify regulatory program: CERCLA
3. Identify approval entity: United States Environmental Protection Agency (USEPA), Connecticut Department of Environmental Protection (CTDEP).
4. Indicate whether the QAPP is a generic or a project-specific QAPP. (circle one)
5. List dates of scoping sessions that were held: 07/02/07; 07/03/07
6. List dates and titles of QAPP documents written for previous site work, if applicable:

Title	Received Date
<u>N/A</u>	<u>N/A</u>
_____	_____
_____	_____

7. List organizational partners (stakeholders) and connection with lead organization:  
USEPA (regulatory oversight), CTDEP (regulatory oversight), NAVFAC Mid-Atlantic (property owner).
8. List data users:  
USEPA (regulatory oversight), CTDEP (regulatory oversight), NAVFAC Mid-Atlantic (property owner).
9. 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 exclusion below: N/A

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

Circle QAPP elements and required information that are not applicable to the project. Provide an explanation in the QAPP.

Required QAPP Element(s) and Corresponding QAPP Section(s)	Optional QAPP Worksheet # in QAPP Workbook	Required Information
<b>Project Management and Objectives</b>		
Title and Approval Page	1	- Title and Approval Page
1.1 Document Format 1.1.1 Document Control Format 1.1.2 Document Control Numbering System 1.1.3 QAPP Identifying Information	2	- Table of Contents - QAPP Identifying Information
1.2 Distribution List and Project Personnel Sign-Off Sheet 1.2.1 Distribution List 1.2.2 Project Personnel Sign-Off Sheet	3 4	- Distribution List - Project Personnel Sign-Off Sheet
1.3 Project Organization Project Organizational Chart 1.3.1 Communication Pathways 1.3.2 Personnel Responsibilities and Qualifications 1.3.3 Special Training Requirements and Certification	5 6 7 8	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table
1.4 Project Planning/Problem Definition 1.4.1 Project Planning (Scoping) 1.4.2 Problem Definition, Site History, and Background	9 10	- Project Planning Session Documentation (including Data Needs tables) - Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site Maps (historical and present)
1.5 Project Quality Objectives and Measurement Performance Criteria 1.5.1 Development of Project Quality Objectives Using the Systematic Planning Process 1.5.2 Measurement Performance Criteria	11 12	- Site-Specific PQOs - Measurement Performance Criteria Table
1.6 Secondary Data Evaluation	13	- Sources of Secondary Data and Information - Secondary Data Criteria and Limitations Table
1.7 Project Overview and Schedule 1.7.1 Project Overview 1.7.2 Project Schedule	14 15 16	- Summary of Project Tasks - Reference Limits and Evaluation Table - Project Schedule/Timeline Table

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

Required QAPP Element(s) and Corresponding QAPP Section(s)	Optional QAPP Worksheet # in QAPP Workbook	Required Information
<b>Measurement/Data Acquisition</b>		
2.1 Sampling Tasks 2.1.1 Sampling Process Design and Rationale 2.1.2 Sampling Procedures and Requirements 2.1.2.1 Sampling Collection Procedures 2.1.2.2 Sample Containers, Volume, and Preservation 2.1.2.3 Equipment/Sample Containers Cleaning and Decontamination Procedures 2.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures 2.1.2.5 Supply Inspection and Acceptance Procedures 2.1.2.6 Field Documentation Procedures	17 18 19 20 21 22	<ul style="list-style-type: none"> <li>- Sampling Design and Rationale</li> <li>- Sample Location Map</li> <li>- Sampling Locations and Methods/ SOP Requirements Table</li> <li>- Analytical Methods/SOP Requirements Table</li> <li>- Field Quality Control Sample Summary Table</li> <li>- Sampling SOPs</li> <li>- Project Sampling SOP References Table</li> <li>- Field Equipment Calibration, Maintenance, Testing, and Inspection Table</li> </ul>
2.2 Analytical Tasks 2.2.1 Analytical SOPs 2.2.2 Analytical Instrument Calibration Procedures 2.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures 2.2.4 Analytical Supply Inspection and Acceptance Procedures	23 24 25	<ul style="list-style-type: none"> <li>- Analytical SOPs</li> <li>- Analytical SOP References Table</li> <li>- Analytical Instrument Calibration Table</li> <li>- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table</li> </ul>
2.3 Sample Collection Documentation, Handling, Tracking, and Custody Procedures 2.3.1 Sample Collection Documentation 2.3.2 Sample Handling and Tracking System 2.3.3 Sample Custody	26 27	<ul style="list-style-type: none"> <li>- Sample Collection Documentation Handling, Tracking, and Custody SOPs</li> <li>- Sample Container Identification</li> <li>- Sample Handling Flow Diagram</li> <li>- Example Chain-of-Custody Form and Seal</li> </ul>
2.4 Quality Control Samples 2.4.1 Sampling Quality Control Samples 2.4.2 Analytical Quality Control Samples	28	<ul style="list-style-type: none"> <li>- QC Samples Table</li> <li>- Screening/Confirmatory Analysis Decision Tree</li> </ul>

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

Required QAPP Element(s) and Corresponding QAPP Section(s)	Optional QAPP Worksheet # in QAPP Workbook	Required Information
2.5 Data Management Tasks 2.5.1 Project Documentation and Records 2.5.2 Data Package Deliverables 2.5.3 Data Reporting Formats 2.5.4 Data Handling and Management 2.5.5 Data Tracking and Control	29 30	- Project Documents and Records Table - Analytical Services Table - Data Management SOPs
2.6 Monitoring Well Inspection Tasks 2.6.1 Monitoring Well Inspection 2.6.2 Database Review 2.6.3 Field Inspection 2.6.4 Minor Monitoring Well Maintenance 2.6.5 Data Collection 2.6.6 Surveying of Monitoring Well Locations 2.7 Evaluation/Decision Process 2.8 Abandonment/Repair 2.9 Equipment Decontamination 2.10 Waste Handling		
<b>Assessment/Oversight</b>		
3.1 Assessments and Response Actions 3.1.1 Planned Assessments 3.1.2 Assessment Findings and Corrective Action Responses	31 32	- Assessments and Response Actions - Planned Project Assessments Table - Audit Checklists - Assessment Findings and Corrective Action Responses Table
3.2 QA Management Reports	33	- QA Management Reports Table
3.3 Outline of Project Report		
<b>Data Review</b>		
4.1 Overview		
4.2 Data Review Steps 4.2.1 Step I: Verification 4.2.2 Step II: Validation 4.2.2.1 Step IIa Validation Activities 4.2.2.2 Step IIb Validation Activities 4.2.3 Step III: Usability Assessment 4.2.3.1 Data Limitations and Actions from Usability Assessment 4.2.3.2 Activities	34 35 36 37	- Verification (Step I) Process Table - Validation (Steps IIa and IIb) Process Table - Validation (Steps IIa and IIb) Summary Table - Usability Assessment
4.3 Streamlining Data Review		Not Applicable

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0  
Revision Date: October 2007**QAPP Worksheet #3**

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

 Worksheet Not Applicable (State Reason)**Distribution List**

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Val Jurka, P.E.	Remedial Project Manager (RPM)	NAVFAC Mid-Atlantic	757-444-6893	757-444-5822	val.jurka@navy.mil	Not applicable
Richard Conant	Environmental Restoration Program Manager	NSB – NLON	860-694-5649	860-694-5320	richard.conant@navy.mil	Not applicable
Kymberlee Keckler	Remedial Project Manager	EPA Region 1	617-918-1385	617-918-0385	Keckler.Kymberlee@epamail.epa.gov	Not applicable
Mark Lewis	Environmental Analyst 3	CTDEP	860-424-3768	860-424-4057	mark.lewis@po.state.ct.us	Not applicable
Kenneth Munney	Contaminants Biologist	USFWS	603-223-2541 , ext.19	603-223-0104	kenneth_munney@fws.gov	Not applicable
Aaron Bernhardt	Project Manager (PM)	TtNUS	412-921-8433	412-921-4040	aaron.bernhardt@ttnus.com	Not applicable
Corey Rich	Base Coordinator	TtNUS	412-921-8984	412-921-4040	corey.rich@ttnus.com	Not applicable
Kelly Carper	Quality Assurance (QA) Officer	TtNUS	412-921-7273	412-921-4040	kelly.carper@ttnus.com	Not applicable
Stan Conti	Field Operations Leader (FOL)/Site Safety Officer(SSO)	TtNUS	412-921-3422	412-921-4040	stanley.conti@ttnus.com	Not applicable
Ed Sedlmyer	Project Chemist	TtNUS	412-921-8704	412-921-4040	ed.sedlmyer@ttnus.com	Not applicable
Andrea Colby	Analytical Services Project Manager	Katahdin Analytical Services, Inc.	207-874-2400	207-775-4029	acolby@katahdinlab.com	Not applicable

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QAPP Worksheet #4**

Have copies of this form signed by key project personnel from each organization to indicate that they have read the applicable QAPP sections 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: Tetra Tech NUS, Inc.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Aaron Bernhardt	PM	412-921-8433		
Corey Rich	Base Coordinator	412-921-8984		
Stan Conti	FOL/SSO	412-921-8422		
Kelly Carper	QA Officer	412-921-7273		
Joe Samchuck	Data Validation Manager (DVM)	412-921-8510		
Ed Sedlmyer	Project Chemist	412-921-8704		

Organization: Katahdin Analytical Services, Inc.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read Email Receipt
Andrea Colby	Analytical Services Project Manager	207-874-2400		

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - New London

Site Location: Groton, Connecticut

Revision Number: 0

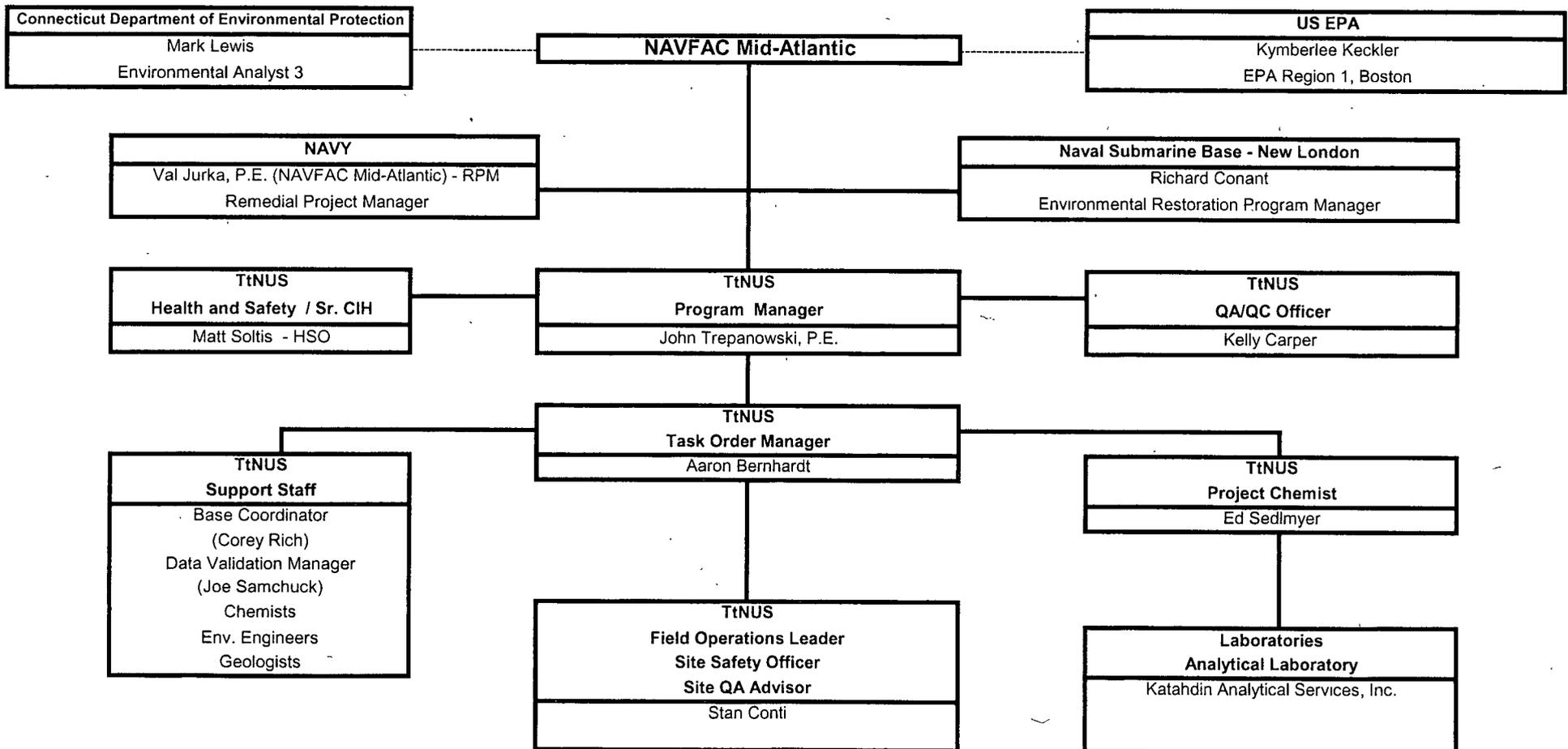
Revision Date: October 2007

**QAPP Worksheet #5**

Identify reporting relationships between all organizations involved in the project, including the lead organization and all contractors 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)

**PROJECT ORGANIZATION CHART**



**QAPP Worksheet #6**

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 modification 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 (FTMRs)	TtNUS FOL	Stan Conti	412-921-8422	Immediately gets approval from TtNUS PM Document via FTMR form
QAPP Amendments	Navy RPM	Val Jurka	757-444-6893	Immediately informs TtNUS PM Document via FTMR form
Changes in Schedule	TtNUS PM	Aaron Bernhardt	412-921-8433	Informs 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 Base Coordinator	Stan Conti Aaron Bernhardt Corey Rich	TBD 412-921-8433 412-921-8984	FOL informs PM; PM informs RPM; RPM issues 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 QA Officer TtNUS Health and Safety Manager (HSM) Navy RPM	Stan Conti Aaron Bernhardt Kelly Carper Matt Soltis  Val Jurka	TBD 412-921-8433 412-921-7273 412-921-8912  757-444-6893	Responsible Party immediately informs subcontractors, the Navy, and Project Team
Analytical data quality issues	Analytical Laboratory TtNUS Project Chemist	Andrea Colby Ed Sedlmyer	207-874-2400 412-921-8704	Immediately notify TtNUS Project Chemist Notify Data Validation Staff and TtNUS PM if necessary

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QAPP Worksheet #7**

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 resumes to this worksheet or note the location of the resumes.

Worksheet Not Applicable (State Reason)

**Personnel Responsibilities and Qualifications Table**

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Aaron Bernhardt	PM	TtNUS	Oversees project, financial, schedule, and technical day to day management of the project.	B.S. Biology, M.S. Chemical Hazard Assessment, 16 years ecological risk assessment experience
Corey Rich	Base Coordinator	TtNUS	Overall coordination of the project and document review	B.A. Physics, B.S. Civil Engineering, 16 years environmental experience
Stan Conti	FOL, SSO	TtNUS	Supervises, coordinates, and performs field sampling activities	B.S. Geology, 36 years environmental experience
Kelly Carper	QA Officer	TtNUS	Prepare QAPP, prepare lab scope, coordinate with lab, and data quality review. Ensure implementation of Quality aspects of the CLEAN program.	B.S. Biology, 15 years environmental experience
Joe Samchuck	Data Validation Manager	TtNUS	Quality assurance of data validation deliverables.	B.S. Chemistry, MBA, M.S. Finance, 23 years environmental experience
Ed Sedlmyer	Project Chemist	TTtNUS	Coordinates analyses with lab chemists, ensures the scope is followed, reviews QA data packages, communicates with TtNUS staff.	B.S. Environmental Science, 19 years environmental experience
Matt Soltis	Health and Safety Manager (HSM)	TtNUS	Oversees CLEAN Program Health and Safety Program	B.S. Industrial Safety Sciences, 24 years of environmental experience

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QAPP Worksheet #8**

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)

**REASON: The only training requirements are Health and Safety requirements that are covered in the Health and Safety Plan.**

**Special Personnel Training Requirements Table**

Project Function	Specialized Training By Title or Description of Course	Training Provider	Training Date	Personnel / Groups Receiving Training	Personnel Titles / Organizational Affiliation	Location of Training Records / Certificates <sup>1</sup>

<sup>1</sup> If training records and/or certificates are on file elsewhere, document their location in this column. If training records and/or certificates do not exist or are not available, then this should be noted.

**Project-Specific QAPP**

Title: QAPP for Phase III Work Plan for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
Site Location: Groton, Connecticut

Revision Number: 0  
Revision Date: October 2007

**QAPP Worksheet #9**

Complete this worksheet for each project scoping session held. Identify project team members who are responsible for planning the project. The following is the generic form used for scoping meetings.

Worksheet Not Applicable (State Reason)

**Participants Sheet**

Project Name: NSB – NLON Projected Date(s) of Sampling: <u>October - November, 2007</u>		Site Name: NSB-NLON  Site Location: GROTON, CONNECTICUT			
Project Manager: Aaron Bernhardt					
<b>Dat (s) of Session: 07/02/2007 and 07/03/2007</b> <b>Scoping Session Purpose: Data Quality Objectives (DQOs) Scoping Meeting</b>					
Name	Title	Affiliation	Phone #	E-mail Address	Project Rol
Aaron Bernhardt	PM	TtNUS	412-921-8433	Aaron.Bernhardt@ttnus.com	Management
Tom Johnston	PM/Corporate QA Mgr.	TtNUS	412-921-8615	Tom.Johnston@ttnus.com	DQO facilitator
Corey Rich	Technical Support	TtNUS	412-921-8984	Corey.Rich@ttnus.com	Technical Review
Catherine Hardison	Environmental Engineer	TtNUS	412-921-8825	Catherine.Hardison@ttnus.com	Technical Support

Note: Corey Rich was only present for the July 3, 2007 meeting.

Comments/Decisions: Develop DQOs for Phase III Work Plan to be inserted in the QAPP.

Action Items: Establish the DQOs in writing.

Consensus Decisions: The writing of the DQOs will be completed by most appropriate personnel and then reviewed by technical personnel and Project Manager.

**Project-Specific QAPP**

Title: QAPP for Phase III Work Plan for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**Participants Sheet**

Project Name: NSB, - NLON Projected Date(s) of Sampling: <u>October - November, 2007</u>	Site Name: NSB-NLON  Site Location: GROTON, CONNECTICUT
Project Manager: Aaron Bernhardt	

**Date of Session: 08/01/2007**  
**Scoping Session Purpose: Sample Collection Discussion for Area A Wetland (Conference Call)**

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Aaron Bernhardt	PM	TtNUS	412-921-8433	Aaron.Bernhardt@ttnus.com	Management
Corey Rich	Technical Support	TtNUS	412-921-8984	Corey.Rich@ttnus.com	Technical Review
Catherine Hardison	Environmental Engineer	TtNUS	412-921-8825	Catherine.Hardison@ttnus.com	Technical Support
Kymberlee Keckler	Remedial Project Manager	EPA Region 1	617-918-1385	Keckler.Kymberlee@epamail.epa.gov	Regulatory Review
Ken Munney	Contaminants Biologist	USFWS	603-223-2541 ext. 19	Kenneth_munney.fws.gov	Regulatory Review
Richard Conant	Environmental Restoration Program Manager	NSB – NLON	860-694-5649	Richard.conant@navy.mil	Management
Bart Hoskins	Ecological Risk Assessor	EPA Region 1	617-918-8375	Hoskins.bart@epa.gov	Regulatory Review

Comments/Decisions: Discuss reasoning behind sampling program and provide regulators with additional information.

Action Items: Provide Sections 3 and 7 from the Phase II RI and surface water data for Area A Landfill Monitoring Program to Ken Munney.

Consensus Decisions: No final consensus decisions were reached, but the following items were presented and discussed.

- 14 surface sediment samples (0-4 inches) focused in the western portion of the wetland near the dike with a few are near the Area A Weapons Center are proposed. This is where the concentrations were greatest in the previous sampling event. Samples will be collected to verify previous concentrations to see whether they have changed over time and to determine whether some areas with the greater concentrations are covered by landfill cap. The depth of 0-4 inches was selected because the top several inches are of concern to sediment invertebrates.
- 4 subsurface sediment samples (2-4 feet) in the western portion of the wetland near the dike are proposed.
- 15 sediment cores throughout the wetland to determine the depth of the organic layer are proposed.
- The regulators proposed analyzing core samples for total organic carbon to help characterize the organic layer.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
Site Location: Groton, Connecticut

Revision Number: 0  
Revision Date: October 2007

**QAPP Worksheet #10**

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

Worksheet Not Applicable (State Reason)

**REASON: DQOs are presented in Section 1.4 of the QAPP**

**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):

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QAPP Worksheet #11**

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 the 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, CTDEP, and EPA

**What will the data be used for?**

1. Determine the extent of contamination in the Area A Wetland
2. Determining the ecological risk in Area A Wetland
3. Determining the depth of the organic layer
4. See Decision Rules in Section 1.4.

**What types of data are needed (matrix, target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)?**

Sediment samples will be analyzed by a fixed-based laboratory for PAHs, pesticides (DDTR), PCBs, and metals. Sediment core samples will be collected to visually determine the thickness of the organic layer above the dredge spoils.

**Matrix:** Sediment

**How "good" do the data need to be in order to support the environmental decision?**

Fulltest level of QC and documentation for performance monitoring assessment and to support the investigation. One hundred percent of laboratory data will undergo full validation in accordance with EPA validation requirements, including EPA Region-specific requirements.

The laboratory must hold a current NELAP accreditation.

See Worksheets #12, 13, and 15

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

**How much data are needed (number of samples for each analytical group, matrix, and concentration)?**

Phase III Investigation - See Worksheet 18

**Where, when, and how should the data be collected/generated?**

Phase III Investigation - See Worksheet 18.

**Who will collect and generate the data?** Samples will be collected by TtNUS. Core samples will be visually inspected in the field to determine the thickness of the organic layer above the dredge spoils. Samples for chemical analysis will be analyzed by Katahdin 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 TtNUS NSB – NLON database.

**How will the data be archived?** Once data are validated, they will be uploaded to the TtNUS master NSB – NLON database and eventually added to the Naval Installation Restoration Information Solution (NIRIS) database. Hardcopy data packages will be stored by a data storage contractor until they are relinquished to the Navy. All records will be offered to the Navy at the completion of the project.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**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)

Matrix	Sediment				
Analytical Group	TAL Metals				
Concentration Level	Low				
Sampling Procedure <sup>1</sup>	Analytical Method/SOP <sup>2</sup>	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)
SA-1.2 and SA-1.3	SW-846 6010B/SOP-12	Data Completeness	95% Overall	Data Completeness Check	S + A
		Comparability	Values 3X QL: Field Duplicates; RPD < 50%	Comparability Check	S + A
		Precision-Overall	Values 3X QL: RPD < 50 %	Field Duplicates	S + A
		Precision-Laboratory	Values 3X QL: RPD < 20 %	Matrix Spike/Matrix Spike Duplicates	A
		Accuracy/Bias	± 25% when sample concentration ≤4X the spike concentration	Matrix Spike/Matrix Spike Duplicates	A
		Accuracy/Bias	Recovery within reference limits supplied by SRM vendor.	Laboratory Control Samples	A
		Accuracy/ Bias-Contamination	No target analytes ≥ QL; with the exception of common field/laboratory contaminants and/or Na, K, and Ca	Equipment Blanks, Trip Blanks, Method Blanks & Instrument Blanks	S + A
		Sensitivity	± 50% of true value at QL	Low Calibration Standard at 2 X QL	A

<sup>1</sup>Reference number from QAPP Worksheet #21 (see Section 3.1.2).

<sup>2</sup>Reference number from QAPP Worksheet #23 (see Section 3.2).

QL – Quantitation Limit  
 RPD – Relative Percent Difference

SRM – Standard Reference Material  
 TAL – Target Analyte List Metals

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON  
**Site Location:** Groton, Connecticut

**Revision Number:** 0  
**Revision Date:** October 2007

Matrix	Aqueous field quality control blanks				
Analytical Group	TAL Metals				
Concentration Level	Low				
Sampling Procedure <sup>1</sup>	Analytical Method/SOP <sup>2</sup>	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)
SA-1.2 and SA-1.3	SW-846 6010B/SOP-12	Data Completeness	95% Overall	Data Completeness Check	S + A
		Comparability	Values 3X QL: Field Duplicates; RPD < 30%	Comparability Check	S + A
		Precision-Overall	Values 3X QL: RPD < 30%	Field Duplicates	S + A
		Precision-Laboratory	Values 3X QL: RPD ≤ 20	Matrix Spike/Matrix Spike Duplicates	A
		Accuracy/Bias	± 25% when sample concentration ≤ 4X the spike concentration	Matrix Spike/Matrix Spike Duplicates	A
		Accuracy/Bias	± 20% of true value	Laboratory Control Samples	A
		Accuracy/ Bias-Contamination	No target analytes ≥ QL; with the exception of common field/laboratory contaminants and/or Na, K, and Ca	Equipment Blanks, Trip Blanks, Method Blanks & Instrument Blanks	S + A
		Sensitivity	± 50% of true value at QL	Low Calibration Standard at 2 X QL	A

<sup>1</sup>Reference number from QAPP Worksheet #21 (see Section 3.1.2).

<sup>2</sup>Reference number from QAPP Worksheet #23 (see Section 3.2).

QL – Quantitation Limit

RPD – Relative Percent Difference

SRM – Standard Reference Material

TAL – Target Analyte List Metals

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

<b>Matrix</b>	Sediment				
<b>Analytical Group</b>	Mercury				
<b>Concentration Level</b>	Low				
<b>Sampling Procedure<sup>1</sup></b>	<b>Analytical Method/SOP<sup>2</sup></b>	<b>Data Quality Indicators (DQIs)</b>	<b>Measurement Performance Criteria</b>	<b>QC Sample and/or Activity Used to Assess Measurement Performance</b>	<b>QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&amp;A)</b>
SA-1.2 and SA-1.3	SW-846 7471A/SOP-13	Data Completeness	95% Overall	Data Completeness Check	S + A
		Comparability	Values $\geq 3X$ QL: Field Duplicates; RPD < 100%	Comparability Check	S + A
		Precision-Overall	Values $\geq 3X$ QL: Field Duplicates RPD < 50%	Field Duplicates	S + A
		Precision-Laboratory	Values $3X$ QL: RPD < 20 %	Matrix Spike/Matrix Spike Duplicate	A
		Accuracy/Bias	$\pm 25\%$ true value when sample concentration $\leq 4X$ the spike concentration	Matrix Spike/Matrix Spike Duplicate	A
		Accuracy/Bias	Recovery within reference limits supplied by SRM vendor.	Laboratory Control Samples	A
		Accuracy/ Bias-Contamination	No target analytes $\geq QL$	Equipment Blanks, Method Blanks & Instrument Blanks	S + A
		Sensitivity	$\pm 50\%$ of true value at QL	Low Calibration Standard at $2 X QL$	A

<sup>1</sup>Reference number from QAPP Worksheet #21 (see Section 3.1.2).

<sup>2</sup>Reference number from QAPP Worksheet #23 (see Section 3.2).

QL – Quantitation Limit

RPD – Relative Percent Difference

Project-Specific QAPP

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

Measurement Performance Criteria Table

Matrix	Aqueous field quality control samples				
Analytical Group	Mercury				
Concentration Level	Low				
Sampling Procedure <sup>1</sup>	Analytical Method/SOP <sup>2</sup>	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)
SA-1.2 and SA-1.3	SW-846 7470A/ SOP-14	Data Completeness	95% Overall	Data Completeness Check	S + A
		Comparability	Values 3X QL: Field Duplicates; RPD < 30%	Comparability Check	S + A
		Precision-Overall	Values ≥3X QL: Field Duplicates RPD < 30%	Field Duplicates	S + A
		Precision-Laboratory	Values 3X QL: RPD < 20 %	Matrix Spike/Matrix Spike Duplicate	A
		Accuracy/Bias	± 25% true value when sample concentration ≤4X the spike concentration	Matrix Spike/Matrix Spike Duplicate	A
		Accuracy/Bias	± 20% of true value	Laboratory Control Samples	A
		Accuracy/ Bias-Contamination	No target analytes ≥QL; with the exception of common field/laboratory contaminants Na, K, and Ca < 5x QL and/or Fe < 4x QL	Equipment Blanks, Method Blanks & Instrument Blanks	S + A
		Sensitivity	± 50% of true value at QL	Low Calibration Standard at 2 X QL	A

<sup>1</sup>Reference number from QAPP Worksheet #21 (see Section 3.1.2).

<sup>2</sup>Reference number from QAPP Worksheet #23 (see Section 3.2).

QL – Quantitation Limit

RPD – Relative Percent Difference

Project-Specific QAPP

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

Matrix	Sediment				
Analytical Group	Select Pesticides				
Concentration Lev I	Low				
Sampling Procedure <sup>1</sup>	Analytical Method/SOP <sup>2</sup>	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)
SA-1.2 and SA-1.3	SW-846 8081A/SOP-2	Data Completeness	95% Overall	Data Completeness Check	S + A
		Comparability	Values > 5X QL: Field Replicates ± 50%	Comparability Check	S + A
		Precision-Overall	Values > 5X QL: Field Duplicates RPD < 50%	Field Duplicates	S + A
		Precision-Laboratory	RPD ≤ 50%	Matrix Spike/Matrix Spike Duplicates	A
		Accuracy/Bias	% Recovery within Statistically derived limits	Matrix Spike/Matrix Spike Duplicates	A
		Accuracy/Bias	% Recovery within Statistically derived limits	Laboratory Control Samples	A
		Accuracy/ Bias-Contamination	No analyte detected ≥ QL	Equipment Blanks, Trip Blanks, Method Blanks	S + A
		Sensitivity	± 100 % of true value at QL	Low Calibration Standard at QL	A

<sup>1</sup>Reference number from QAPP Worksheet #21 (see Section 3.1.2).

<sup>2</sup>Reference number from QAPP Worksheet #23 (see Section 3.2).

QL – Quantitation Limit

RPD – Relative Percent Difference

TCL – Target Compound List

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

Matrix	Aqueous field quality control samples				
Analytical Group	Select Pesticides				
Concentration Level	Low				
Sampling Procedure <sup>1</sup>	Analytical Method/SOP <sup>2</sup>	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)
SA-1.2 and SA-1.3	SW-846 8081A/SOP-2	Data Completeness	95% Overall	Data Completeness Check	S + A
		Comparability	Values > 5X QL: Field Duplicates ± 30%	Comparability Check	S + A
		Precision-Overall	Values > 5X QL: Field Duplicates RPD < 30%	Field Duplicates	S + A
		Precision-Laboratory	RPD ≤ 30% (calculated using concentration)	Matrix Spike/Matrix Spike Duplicates	A
		Accuracy/Bias	% Recovery within Statistically derived limits	Matrix Spike/Matrix Spike Duplicates	A
		Accuracy/Bias	% Recovery within Statistically derived limits	Laboratory Control Samples	A
		Accuracy/ Bias-Contamination	No analyte detected ≥ QL	Equipment Blanks, Trip Blanks, Method Blanks	S + A
		Sensitivity	± 100 % of true value at QL	Low Calibration Standard at QL	A

<sup>1</sup>Reference number from QAPP Worksheet #21 (see Section 3.1.2).

<sup>2</sup>Reference number from QAPP Worksheet #23 (see Section 3.2).

QL – Quantitation Limit

RPD – Relative Percent Difference

TCL – Target Compound List

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

Matrix	Sediment				
Analytical Group	TCL PCBs				
Concentration Level	Low				
Sampling Procedure <sup>1</sup>	Analytical Method/SOP <sup>2</sup>	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)
SA-1.2 and SA-1.3	SW-846 8082 SOP-3	Data Completeness	95% Overall	Data Completeness Check	S + A
		Comparability	Values > 5X QL: Field Duplicates ± 50%	Comparability Check	S + A
		Precision-Overall	Values > 5X QL: Field Replicates ± 50%	Field Duplicates	S + A
		Precision-Laboratory	RPD ± 50%	Matrix Spike/Matrix Spike Duplicates	A
		Accuracy/Bias	% Recovery within Statistically derived limits	Matrix Spike/Matrix Spike Duplicates	A
		Accuracy/Bias	% Recovery within Statistically derived limits	Laboratory Control Samples	A
		Accuracy/ Bias-Contamination	No target analytes ≥QL; with the exception of common field/laboratory contaminants	Equipment Blanks, Trip Blanks, Method Blanks & Instrument Blanks	S + A
		Sensitivity	± 100 % of true value at QL	Low Calibration Standard at QL	A

<sup>1</sup>Reference number from QAPP Worksheet #21 (see Section 3.1.2).

<sup>2</sup>Reference number from QAPP Worksheet #23 (see Section 3.2).

PCB – Polychlorinated Biphenyls

QL – Quantitation Limit

RPD – Relative Percent Difference

TCL – Target Compound List

Project-Specific QAPP

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

<b>Matrix</b>	Aqueous field quality control samples
<b>Analytical Group</b>	TCL PCBs
<b>Concentration Level</b>	Low

Sampling Procedure <sup>1</sup>	Analytical Method/SOP <sup>2</sup>	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)
SA-1.2 and SA-1.3	SW-846 8082/SOP-3	Data Completeness	95% Overall	Data Completeness Check	S + A
		Comparability	Values > 5X QL: Field Duplicates ± 30%	Comparability Check	S + A
		Precision-Overall	Values > 5X QL: Field Duplicates ± 30%	Field Duplicates	S + A
		Precision-Laboratory	RPD ± 30%	Matrix Spike/Matrix Spike Duplicates	A
		Accuracy/Bias	% Recovery within Statistically derived limits	Matrix Spike/Matrix Spike Duplicates	A
		Accuracy/Bias	% Recovery within Statistically derived limits	Laboratory Control Samples	A
		Accuracy/ Bias-Contamination	No target analytes ≥ QL; with the exception of common field/laboratory contaminants	Equipment Blanks, Trip Blanks, Method Blanks & Instrument Blanks	S + A
		Sensitivity	± 100 % of true value at QL	Low Calibration Standard at QL	A

<sup>1</sup>Reference number from QAPP Worksheet #21 (see Section 3.1.2).

<sup>2</sup>Reference number from QAPP Worksheet #23 (see Section 3.2).

PCB – Polychlorinated Biphenyls

QL – Quantitation Limit

RPD – Relative Percent Difference

TCL – Target Compound List

Project-Specific QAPP

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

<b>Matrix</b>	Sediment				
<b>Analytical Group</b>	PAHs				
<b>Concentration Level</b>	Low				
<b>Sampling Procedure<sup>1</sup></b>	<b>Analytical Method/SOP<sup>2</sup></b>	<b>Data Quality Indicators (DQIs)</b>	<b>Measurement Performance Criteria</b>	<b>QC Sample and/or Activity Used to Assess Measurement Performance</b>	<b>QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&amp;A)</b>
SA-1.2 and SA-1.3	SW-846 8270C SIM/SOP-1	Comparability	95% Overall	Data Completeness Check	S + A
		Comparability	All Values > 5X QL, Field Duplicates ± 50%	Comparability Check	S + A
		Precision-Overall	All Values > 5X QL, RPD ≤50%	Field Duplicates	S + A
		Precision-Laboratory	RPD ≤50% when native conc. < 50% analytical spike	Matrix Spike/Matrix Spike Duplicates	A
		Accuracy/Bias	% Recovery within Statistically derived limits	Matrix Spike/Matrix Spike Duplicates	A
		Accuracy/Bias	% Recovery within Statistically derived limits	Laboratory Control Samples	A
		Accuracy/ Bias-Contamination	No target analytes ≥QL; with the exception of common field/laboratory contaminants	Equipment Blanks, Trip Blanks, Method Blanks & Instrument Blanks	S + A
		Sensitivity	± 100 % of true value at QL	Low Calibration Standard at QL	A

<sup>1</sup>Reference number from QAPP Worksheet #21 (see Section 3.1.2).

<sup>2</sup>Reference number from QAPP Worksheet #23 (see Section 3.2).

PAHs - Polycyclic Aromatic Hydrocarbons

QL – Quantitation Limit

RPD – Relative Percent Difference

SIM – Selective Ion Monitoring

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON  
**Site Location:** Groton, Connecticut

**Revision Number:** 0  
**Revision Date:** October 2007

<b>Matrix</b>	Aqueous field quality control samples				
<b>Analytical Group</b>	PAHs				
<b>Concentration Level</b>	Low				
<b>Sampling Procedure<sup>1</sup></b>	<b>Analytical Method/SOP<sup>2</sup></b>	<b>Data Quality Indicators (DQIs)</b>	<b>Measurement Performance Criteria</b>	<b>QC Sample and/or Activity Used to Assess Measurement Performance</b>	<b>QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&amp;A)</b>
SA-1.2 and SA-1.3	SW-846 8270C SIM/SOP-1	Comparability	95% Overall	Data Completeness Check	S + A
		Comparability	All Values > 5X QL, Field Duplicates ± 30%	Comparability Check	S + A
		Precision-Overall	All Values > 5X QL, RPD ≤30%	Field Duplicates	S + A
		Precision-Laboratory	RPD ≤30% when native conc. < 30% analytical spike	Matrix Spike/Matrix Spike Duplicates	A
		Accuracy/Bias	% Recovery within Statistically derived limits	Matrix Spike/Matrix Spike Duplicates	A
		Accuracy/Bias	% Recovery within Statistically derived limits	Laboratory Control Samples	A
		Accuracy/ Bias-Contamination	No target analytes ≥QL; with the exception of common field/laboratory contaminants	Equipment Blanks, Trip Blanks, Method Blanks & Instrument Blanks	S + A
		Sensitivity	± 100 % of true value at QL	Low Calibration Standard at QL	A

<sup>1</sup>Reference number from QAPP Worksheet #21 (see Section 3.1.2).

<sup>2</sup>Reference number from QAPP Worksheet #23 (see Section 3.2).

PAHs - Polycyclic Aromatic Hydrocarbons

QL – Quantitation Limit

RPD – Relative Percent Difference

SIM – Selective Ion Monitoring

Project-Specific QAPP

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

Matrix	Sediment				
Analytical Group	TOC				
Concentration Level	Low				
Sampling Procedure <sup>1</sup>	Analytical Method/SOP <sup>2</sup>	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)
SA-1.2 and SA-1.3	TOC-Lloyd Kahn/SOP-16	Data Completeness	95% Overall	Data Completeness Check	S + A
		Comparability	All Values > 5X QL, Field Duplicates ± 30%	Comparability Check	S + A
		Precision-Overall	Values ≥ 3X QL: Field Duplicates RPD < 50%	Field Duplicates	S + A
		Precision-Laboratory	Values 3X QL: RPD < 20 % calculated using concentration	Matrix Spike/Matrix Spike Duplicate	A
		Accuracy/Bias	± 25% of the true value when sample concentration ≤ 4X the spike concentration	Matrix Spike/Matrix Spike Duplicate	A
		Accuracy/Bias	± 20% of true value	Laboratory Control Samples	A
		Accuracy/ Bias-Contamination	No target analytes ≥ QL;	Equipment Blanks, Method Blanks & Instrument Blanks	S + A
		Sensitivity	± 100 % of true value at QL	Low Calibration Standard at QL	A

<sup>1</sup>Reference number from QAPP Worksheet #21 (see Section 3.1.2).

<sup>2</sup>Reference number from QAPP Worksheet #23 (see Section 3.2).

QL – Quantitation Limit

RPD – Relative Percent Difference

TOC – Total Organic Carbon

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

<b>Matrix</b>	Sediment				
<b>Analytical Group</b>	pH				
<b>Concentration Level</b>	Low				
<b>Sampling Procedure<sup>1</sup></b>	<b>Analytical Method/SOP<sup>2</sup></b>	<b>Data Quality Indicators (DQIs)</b>	<b>Measurement Performance Criteria</b>	<b>QC Sample and/or Activity Used to Assess Measurement Performance</b>	<b>QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&amp;A)</b>
SA-1.2 and SA-1.3	SW8-46 9045C/SOP-15	Data Completeness	95% Overall	Data Completeness Check	S + A
		Precision-Overall	All Values > 5X QL, RPD < 50%	Field Duplicates	S + A
		Accuracy/Bias	%Recovery Statistically derived limits	Laboratory Control Samples	A

<sup>1</sup>Reference number from QAPP Worksheet #21 (see Section 3.1.2).

<sup>2</sup>Reference number from QAPP Worksheet #23 (see Section 3.2).

QL – Quantitation Limit

RPD – Relative Percent Difference



**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QAPP Worksheet #13**

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. Each project specific area must include any limitations on use of the data in the final report. Data from each project specific area is accumulated in the final site report and the limits on data use must be presented.

Worksheet Not Applicable (State Reason)

**Secondary Data Criteria and Limitations Table**

<b>Secondary Data</b>	<b>Data Source</b> (originating organization, report title and date)	<b>Data Generator(s)</b> (originating organization, data types, data generation / collection dates)	<b>How Data Will Be Used</b>	<b>Limitations on Data Use</b>
Phase I and Phase II Remedial Investigation and Focused Feasibility Study	Brown & Root Environmental, March 1997	Analytical data for groundwater, soil, sediment, surface water, and biological tissue, Human Health and Ecological Risk Assessment. Most samples were collected in 1990; 1993, and 1994	Characterization data (soil, sediment, surface water, and biological tissue) were used to reevaluate ecological risk using current methodologies. Data will be combined with Phase III investigation data and used for revised risk assessments.	None
Surface Water Monitoring Data	ECC, June 2006	Monitoring surface water and groundwater data. Samples were collected from 1999 to present.	The surface water monitoring data were used in the updated SERA.	None

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLÓN  
Site Location: Groton, Connecticut

Revision Number: 0  
Revision Date: October 2007

**QAPP Worksheet #14**

Provide a brief overview of the listed project activities. The following table must be completed for each project area.

Worksheet Not Applicable (State Reason)

**Summary of Project Tasks**

**Sampling Tasks:**

Sediment sampling at Area A Wetland – Site 2, including 14 surface sediment samples and 4 subsurface sediment samples. Approximately 15 core samples will be collected for visual observation of the thickness of the organic layer above the dredge spoils.

**Analysis Tasks:**

Sediment Samples:

PAHs, pesticides (DDTR), PCBs, metals, pH, and TOC

Core Samples:

Visual Analysis of organic layer.

Land Surveying:

Sample locations will be surveyed.

IDW Disposal:

IDW will consist of decontamination water from cleaning field equipment. The IDW will be disposed of properly.

Field Measurements:

Organic layer will be measured with a ruler

**Quality Control Tasks:**

QA/QC field samples including duplicates, rinsate blanks, and field blanks. Lab MS/MSD samples. Chain of custody procedures implemented.

**Secondary Data:**

Data collected in the Area A wetland as part of the Phase I and Phase II RIs and the FFS. Surface water collected as part of the Area A Landfill long-term monitoring program.

**Other Data:**

Visual observations of organic layer across the wetland.

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

**Data Management Tasks:**

Incorporate data validation qualifier flags, verify sample numbers and locations, run statistical calculations.

**Documentation and Records:**

Record all field data in logbook and on associated field forms. Data validation reporting.

**Assessment / Audit Tasks**

See Worksheet #31 for information regarding audits of field personnel, field procedures, and 100% data validation for laboratory analytical data.

**Data Review Tasks:**

Data validation, database QA, calculation/data input technical review.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QAPP Worksheet #15**

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 used in place of this worksheet**

**Reference Limits and Evaluation Table**

Analytical Group:

Concentration Level:

Analyte	CAS Number	Project Action Limit (applicable units)	Project (applicable units)	Analytical Method		Achievable Laboratory Limits	
				MDLs	Method QLs	MDLs	QLs

TABLE FOR WORKSHEET #15

REFERENCE LIMITS AND EVALUATION TABLE  
 QAPP FOR THE PHASE III INVESTIGATION  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CT

Analytes	CAS Number	Project Action Limit (mg/kg) <sup>(1)</sup>	Project Quantitation Limit (mg/kg) <sup>(2)</sup>	Katahdin Achievable Laboratory IDLs (mg/kg)	Katahdin Achievable Laboratory Quantitation Limit (mg/kg)
<b>TAL Metals SW-846 6010B</b>					
ALUMINUM	7429-90-5	25500	6375	1.90	30
ANTIMONY	7440-36-0	3	0.75	0.09	0.80
ARSENIC	7440-38-2	9.79	2.45	0.08	0.80
BARIIUM	7440-39-3	48	12	0.05	0.50
BERYLLIUM	7440-41-7	NA	NA	0.01	0.50
CADMIUM	7440-43-9	0.99	0.2	0.01	1.0
CALCIUM	7440-70-2	NA	NA	0.77	5.0
CHROMIUM	7440-47-3	43.4	10.9	0.03	1.5
COBALT	7440-48-4	50	12.5	0.03	3.0
COPPER	7440-50-8	31.6	7.9	0.02	2.5
IRON	7439-89-6	20000	5000	0.52	10
LEAD	7439-92-1	35.8	8.95	0.09	0.5
MAGNESIUM	7439-95-4	NA	NA	0.49	5.0
MANGANESE	7439-96-5	460	115	0.06	0.50
MERCURY	7439-97-6	0.18	0.045	0.0020	0.040
NICKEL	7440-02-0	22.7	5.7	0.04	4.0
POTASSIUM	7440-09-7	NA	NA	8.60	100
SELENIUM	7782-49-2	1	0.25	0.15	1.0
SILVER	7440-22-4	0.5	0.13	0.05	1.5
SODIUM	7440-23-5	NA	NA	1.30	100
THALLIUM	7440-28-0	NA	NA	0.07	1.5
VANADIUM	7440-62-2	57	14.3	0.029	2.5
ZINC	7440-66-6	121	30.3	0.02	2.5

TABLE FOR WORKSHEET #15

REFERENCE LIMITS AND EVALUATION TABLE  
 QAPP FOR THE PHASE III INVESTIGATION  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CT

Analytes	CAS Number	Project Action Limit (ug/L)	Project Quantitation Limit (ug/L)	Katahdin Achievable Laboratory IDLs (ug/L)	Katahdin Achievable Laboratory Quantitation Limit (ug/L)
<b>TAL Metals SW-846 6010B</b>					
ALUMINUM	7429-90-5	NA <sup>(3)</sup>	NA <sup>(3)</sup>	19	300
ANTIMONY	7440-36-0	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.870	8.0
ARSENIC	7440-38-2	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.83	8.0
BARIUM	7440-39-3	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.45	5.0
BERYLLIUM	7440-41-7	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.12	5.0
CADMIUM	7440-43-9	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.099	10
CALCIUM	7440-70-2	NA <sup>(3)</sup>	NA <sup>(3)</sup>	7.7	50
CHROMIUM	7440-47-3	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.28	15
COBALT	7440-48-4	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.260	30
COPPER	7440-50-8	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.22	25
IRON	7439-89-6	NA <sup>(3)</sup>	NA <sup>(3)</sup>	5.20	100
LEAD	7439-92-1	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.91	5.0
MAGNESIUM	7439-95-4	NA <sup>(3)</sup>	NA <sup>(3)</sup>	4.9	50
MANGANESE	7439-96-5	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.57	5.0
MERCURY	7439-97-6	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.012	0.20
NICKEL	7440-02-0	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.41	40
POTASSIUM	7440-09-7	NA <sup>(3)</sup>	NA <sup>(3)</sup>	86.0	1000
SELENIUM	7782-49-2	NA <sup>(3)</sup>	NA <sup>(3)</sup>	1.5	10
SILVER	7440-22-4	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.46	15
SODIUM	7440-23-5	NA <sup>(3)</sup>	NA <sup>(3)</sup>	13.0	1000
THALLIUM	7440-28-0	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.710	15
VANADIUM	7440-62-2	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.29	25
ZINC	7440-66-6	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.23	25

TABLE FOR WORKSHEET #15

REFERENCE LIMITS AND EVALUATION TABLE  
 QAPP FOR THE PHASE III INVESTIGATION  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CT

Analytes	CAS Number	Project Action Limit (ug/kg)	Project Quantitation Limit (ug/kg) <sup>(2)</sup>	Katahdin Achievable Laboratory MDLs (ug/kg)	Katahdin Achievable Laboratory Quantitation Limit (ug/kg)
<b>TCL Pesticides SW-846 8081A</b>					
4,4'-DDD	72-54-8	4.88	1.22	1.12	3.3
4,4'-DDE	72-55-9	3.16	0.79	0.81	3.3
4,4'-DDT	50-29-3	4.16	1.04	0.84	3.3

Analytes	CAS Number	Project Action Limit (ug/L)	Project Quantitation Limit (ug/L)	Katahdin Achievable Laboratory MDLs (ug/L)	Katahdin Achievable Laboratory Quantitation Limit (ug/L)
<b>TCL Pesticides SW-846 8081A</b>					
4,4'-DDD	72-54-8	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.036	0.1
4,4'-DDE	72-55-9	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.038	0.1
4,4'-DDT	50-29-3	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.039	0.1

Analytes	CAS Number	Project Action Limit (ug/kg)	Project Quantitation Limit (ug/kg) <sup>(2)</sup>	Katahdin Achievable Laboratory MDLs (ug/kg)	Katahdin Achievable Laboratory Quantitation Limit (ug/kg)
<b>TAL Metals SW-846 8082</b>					
AROCLOR-1016	12674-11-2	59.8	15	16	17
AROCLOR-1221	11104-28-2	59.8	15	8.99	17
AROCLOR-1232	11141-16-5	59.8	15	5.3	17
AROCLOR-1242	53469-21-9	59.8	15	6.7	17
AROCLOR-1248	12672-29-6	59.8	15	5.7	17
AROCLOR-1254	11097-69-1	59.8	15	13	17
AROCLOR-1260	11096-82-5	59.8	15	14	17

Analytes	CAS Number	Project Action Limit (ug/L)	Project Quantitation Limit (ug/L)	Katahdin Achievable Laboratory MDLs (ug/L)	Katahdin Achievable Laboratory Quantitation Limit (ug/L)
<b>TAL Metals SW-846 8082</b>					
AROCLOR-1016	12674-11-2	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.40	0.5
AROCLOR-1221	11104-28-2	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.17	0.5
AROCLOR-1232	11141-16-5	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.28	0.5
AROCLOR-1242	53469-21-9	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.21	0.5
AROCLOR-1248	12672-29-6	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.19	0.5
AROCLOR-1254	11097-69-1	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.25	0.5
AROCLOR-1260	11096-82-5	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.27	0.5

TABLE FOR WORKSHEET #15

REFERENCE LIMITS AND EVALUATION TABLE  
 QAPP FOR THE PHASE III INVESTIGATION  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CT

Analytes	CAS Number	Project Action Limit (ug/kg) <sup>(1)</sup>	Project Quantitation Limit (ug/kg) <sup>(2)</sup>	Katahdin Achievable Laboratory MDLs (ug/kg)	Katahdin Achievable Laboratory Quantitation Limit (ug/kg)
<b>TCL PAHs SW-846 8270C SIM</b>					
1-METHYLNAPHTHALENE	90-12-0	20.2	5.1	1.7	20
2-METHYLNAPHTHALENE	91-57-6	20.2	5.1	2.8	20
ACENAPHTHENE	83-32-9	290	73	1.5	20
ACENAPHTHYLENE	208-96-8	160	40	1.3	20
ANTHRACENE	120-12-7	57.2	14	2.6	20
BENZO(A)ANTHRACENE	56-55-3	108	27	2.5	20
BENZO(A)PYRENE	50-32-8	150	38	2.1	20
BENZO(B)FLUORANTHENE	205-99-2	1800	450	2.2	20
BENZO(G,H,I)PERYLENE	191-24-2	170	43	3.3	20
BENZO(K)FLUORANTHENE	207-08-9	240	60	1.9	20
CHRYSENE	218-01-9	166	42	2.6	20
DIBENZO(A,H)ANTHRACENE	53-70-3	33	8.3	3.8	20
FLUORANTHENE	206-44-0	423	106	4.0	20
FLUORENE	86-73-7	77.4	19	1.6	20
INDENO(1,2,3-CD)PYRENE	193-39-5	200	50	4.3	20
NAPHTHALENE	91-20-3	176	44	1.8	20
PHENANTHRENE	85-01-8	204	51	4.3	20
PYRENE	129-00-0	195	49	6.9	20

Analytes	CAS Number	Project Action Limit (ug/L)	Project Quantitation Limit (ug/L)	Katahdin Achievable Laboratory MDLs (ug/L)	Katahdin Achievable Laboratory Quantitation Limit (ug/L)
<b>TCL PAHs SW-846 8270C SIM</b>					
1-METHYLNAPHTHALENE	90-12-0	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.027	0.20
2-METHYLNAPHTHALENE	91-57-6	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.035	0.20
ACENAPHTHENE	83-32-9	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.028	0.20
ACENAPHTHYLENE	208-96-8	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.018	0.20
ANTHRACENE	120-12-7	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.035	0.20
BENZO(A)ANTHRACENE	56-55-3	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.043	0.20
BENZO(A)PYRENE	50-32-8	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.080	0.20
BENZO(B)FLUORANTHENE	205-99-2	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.078	0.20
BENZO(G,H,I)PERYLENE	191-24-2	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.072	0.20
BENZO(K)FLUORANTHENE	207-08-9	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.051	0.20
CHRYSENE	218-01-9	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.093	0.20
DIBENZO(A,H)ANTHRACENE	53-70-3	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.11	0.20
FLUORANTHENE	206-44-0	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.071	0.20
FLUORENE	86-73-7	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.033	0.20
INDENO(1,2,3-CD)PYRENE	193-39-5	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.085	0.20
NAPHTHALENE	91-20-3	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.049	0.20
PHENANTHRENE	85-01-8	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.038	0.20
PYRENE	129-00-0	NA <sup>(3)</sup>	NA <sup>(3)</sup>	0.11	0.20

TABLE FOR WORKSHEET #15

REFERENCE LIMITS AND EVALUATION TABLE  
 QAPP FOR THE PHASE III INVESTIGATION  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CT

Analytes	CAS Number	Project Action Limit (mg/kg)	Project Quantitation Limit (mg/kg) <sup>(2)</sup>	Katahdin Achievable Laboratory MDLs (mg/kg)	Katahdin Achievable Laboratory Quantitation Limit (mg/kg)
<b>Micellaneous</b>					
TOC (Lloyd Kahn)	NA	NA <sup>(3)</sup>	NA <sup>(3)</sup>	84.8	400
pH (9045C)	NA	NA <sup>(3)</sup>	NA <sup>(3)</sup>	NA	NA

- (1) – The values and sources of the Project Action Limits are The Ecological Screening Levels and sources of the screening levels listed in Appendix C Table 5-2.
  - (2) – Project quantitation limits were set to be 25% of the project action limit
  - (3) – Action limits are not required for the aqueous samples because they are just quality assurance/quality control samples and project action limits are not needed for TOC or pH.
- NA - Not available

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QAPP Worksheet #16**

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)

**Project Schedule / Timeline Table**

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Rough Draft Phase III Work Plan	TtNUS	06/15/2007	07/16/2007	Rough Draft Phase III Work Plan	07/16/2007
Draft Phase III Work Plan	TtNUS	TBD	08/14/2007	Draft Work Plan	08/14/2007
Final Phase III Work Plan	TtNUS	9/14/2007	10/19/2007	Final Work Plan	10/19/2007
Field Work and Chemical Analysis	TtNUS and Katahdin	October 2007	November 2007	None	None
Field Data Review	TtNUS	October 2007	November 2007	Complete and accurate field notes	December 2007
Laboratory Data Validation	TtNUS	November 2007	December 2007	None	None
Data Analysis and Interpretation /Rough Draft RI/FS	TtNUS	TBD	09/08/2008	Rough Draft RI/FS	09/08/2008
Draft RI/FS	TtNUS	TBD	11/07/2008	Draft RI/FS	11/08/2008
Final RI/FS	TtNUS	TBD	02/26/2009	Final RI/FS	02/26/2009

**QAPP Worksheet #17**

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):**

**Sediment samples:** As discussed in Section 1.4.2.2, the greatest chemical concentrations in the existing sediment samples were generally in the samples located near the area A Landfill and the Area A Weapons Center. The primary reason for collecting additional sediment samples during this phase is to obtain current site data and better characterize nature and extent of contamination. This is being done in part because the landfill cap installation may have covered some of the previous sample locations and because the chemical concentrations in sediment may have changed over time. Figure 2-1 shows the locations of the proposed sediment locations. The locations of the historic samples are provided for a point-of-reference on the figure. Note all of the historic samples were not analyzed for all of the parameters (see Tables 1-1 and 1-2 as well as the figures in Attachment 1 in Appendix B). Samples locations were selected based on professional judgment.

**Subsurface Sediment Samples:** As discussed in Section 1.4.2.2, based on the previous data, the chemical concentrations in the subsurface soil were much less than the concentrations in the surface soil/sediment. However, the subsurface soil samples were not located in the same areas where the greatest sediment concentrations were found. Analytical data for subsurface sediment would be needed to evaluate options in the FS, should one be needed. Figure 2-1 shows the locations of the proposed subsurface sediment locations. Samples locations were selected based on professional judgment.

**Organic Layer Samples:** The approximate depth of the organic layer above the dredge spoils will be investigated using a core sampler and/or hand augers. The approximate thickness and depth of the organic layer will be determined and visually and measured with field instruments (e.g. measuring tapes). Select samples will be analyzed for total organic carbon (TOC) to determine whether there is a difference in TOC between the organic layer and the dredge spoils. The proposed sample locations were spread across the wetland to encompass various habitats and obtain good spatial coverage. Planned sampling density is greatest where previous chemical concentrations were greatest because these are the locations where the presence of an organic layer would be more important to bind and/or cover the contamination. See Figure 2-1 for approximate sample locations. Several of the proposed locations are the same as the sediment samples that will be collected. Actual field locations may be moved (within 100 feet) will be based on site access. Samples locations were selected based on professional judgment.

**Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will 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 Worksheet #18.

Project-Specific QAPP

Title: QAPP for Phase III Investigation for Area A Wetland - Site 2B

Site Name/Project Name NSB - NLON  
 Site Location Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

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

List all site locations that will be sampled and include sample/ID number, if available. (Provide a range of sampling locations of 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.

Sampling Location / ID Number	Sample ID	Matrix	Depth	Analytical Group	Concentration Level	Number of Samples	Sampling SOP Reference	Rationale for Sampling Location
<b>SURFICIAL SEDIMENT SAMPLES</b>								
2W-SD43	2W-SD-SD43-0001	Sediment	0-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 3	Horizontal Extent and Eco Risk
2W-SD44	2W-SD-SD44-0001	Sediment	0-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 4	Horizontal Extent and Eco Risk
2W-SD45	2W-SD-SD45-0001	Sediment	0-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 5	Horizontal Extent and Eco Risk
2W-SD46	2W-SD-SD46-0001	Sediment	0-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 6	Horizontal Extent and Eco Risk
2W-SD47	2W-SD-SD47-0001	Sediment	0-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7.1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 7	Horizontal Extent and Eco Risk
2W-SD48	2W-SD-SD48-0001	Sediment	0-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 8	Horizontal Extent and Eco Risk
2W-SD49	2W-SD-SD49-0001	Sediment	0-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 9	Horizontal Extent and Eco Risk
2W-SD50	2W-SD-SD50-0001	Sediment	0-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1.10	Horizontal Extent and Eco Risk
2W-SD51	2W-SD-SD51-0001	Sediment	0-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7.1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 11	Horizontal Extent and Eco Risk
2W-SD52	2W-SD-SD52-0001	Sediment	0-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 12	Horizontal Extent and Eco Risk
2W-SD53	2W-SD-SD53-0001	Sediment	0-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7.1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 13	Horizontal Extent and Eco Risk
2W-SD54	2W-SD-SD54-0001	Sediment	0-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 14	Horizontal Extent and Eco Risk
2W-SD55	2W-SD-SD55-0001	Sediment	0-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 15	Horizontal Extent and Eco Risk
2W-SD56	2W-SD-SD56-0001	Sediment	0-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 16	Horizontal Extent and Eco Risk
2W-SD57	2W-SD-SD57-0001	Sediment	0-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 16	Horizontal Extent and Eco Risk
2W-SD58	2W-SD-SD58-0001	Sediment	0-4'	Cadmium	Low to Moderate	1	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 16	Horizontal Extent and Eco Risk
<b>Total Number of Surficial Sediment Samples</b>						16		
<b>SUB-SURFACE SEDIMENT SAMPLES</b>								
2W-SD-46	2W-SD-SD46-0204	Sediment	2-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1.17	Vertical Extent
2W-SD-48	2W-SD-SD48-0204	Sediment	2-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7.1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 17	Vertical Extent
2W-SD-50	2W-SD-SD50-0204	Sediment	2-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1.17	Vertical Extent
2W-SD-53	2W-SD-SD53-0204	Sediment	2-4'	PAHs PCBs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Metals, TOC, pH	Low to Moderate	1	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 17	Vertical Extent
<b>Total Number of Subsurface Sediment Samples</b>						4		
<b>SEDIMENT CORE SAMPLES</b>								
2W-SC01	2W-SC-SD01-xxxx	Sediment	0-4'	TOC	Low to Moderate	2 <sup>(1)</sup>	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 3	Thickness of organic layer
2W-SC02	2W-SC-SD02-xxxx	Sediment	0-4'	TOC	Low to Moderate	2	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 4	Thickness of organic layer
2W-SC03	2W-SC-SD03-xxxx	Sediment	0-4'	TOC	Low to Moderate	2	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 5	Thickness of organic layer
2W-SC04	2W-SC-SD04-xxxx	Sediment	0-4'	TOC	Low to Moderate	2	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 6	Thickness of organic layer
2W-SC05	2W-SC-SD05-xxxx	Sediment	0-4'	TOC	Low to Moderate	2	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 7	Thickness of organic layer
2W-SC06	2W-SC-SD06-xxxx	Sediment	0-4'	TOC	Low to Moderate	2	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 8	Thickness of organic layer
2W-SC07	2W-SC-SD07-xxxx	Sediment	0-4'	TOC	Low to Moderate	2	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1.9	Thickness of organic layer

Project-Specific QAPP

Title: QAPP for Phase III Investigation for Area A Wetland - Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

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

List all site locations that will be sampled and include sample/ID number, if available. (Provide a range of sampling locations of 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.

Sampling Location / ID Number	Sample ID	Matrix	Depth	Analytical Group	Concentration Level	Number of Samples	Sampling SOP Reference	Rationale for Sampling Location
2W-SC08	2W-SC-SD08-xxxx	Sediment	0-4'	TOC	Low to Moderate	2	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 10	Thickness of organic layer
2W-SC09	2W-SC-SD09-xxxx	Sediment	0-4'	TOC	Low to Moderate	2	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 11	Thickness of organic layer
2W-SC10	2W-SC-SD10-xxxx	Sediment	0-4'	TOC	Low to Moderate	2	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 12	Thickness or organic layer
2W-SC11	2W-SC-SD11-xxxx	Sediment	0-4'	TOC	Low to Moderate	2	SA-7.1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1.13	Thickness or organic layer
2W-SC12	2W-SC-SD12-xxxx	Sediment	0-4'	TOC	Low to Moderate	2	SA-7 1, CT-04, SA-6.3, SA-6 1, SA-1 2, SA-1 14	Thickness or organic layer
2W-SC13	2W-SC-SD13-xxxx	Sediment	0-4'	TOC	Low to Moderate	2	SA-7 1, CT-04, SA-6 3, SA-6.1, SA-1 2, SA-1 15	Thickness or organic layer
2W-SC14	2W-SC-SD14-xxxx	Sediment	0-4'	TOC	Low to Moderate	2	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 16	Thickness or organic layer
2W-SC15	2W-SC-SD15-xxxx	Sediment	0-4'	TOC	Low to Moderate	2	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 16	Thickness or organic layer
2W-SC16	2W-SC-SD16-xxxx	Sediment	0-4'	TOC	Low to Moderate	2	SA-7 1, CT-04, SA-6 3, SA-6 1, SA-1 2, SA-1 12	Thickness of organic layer
<b>Total Number of Sediment Core Samples</b>						18 <sup>(1)</sup>		
<b>Total Number of Sediment Samples</b>						38		
<b>Total Number of Duplicate Samples</b>						4		

(1) - Sediment samples from the organic layer and the dredge spoils will be analyzed for TOC at approximately half (9) of the sediment core locations. The sample IDs will reflect the depths from which the samples were collected.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**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**

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference <sup>1</sup>	Sample Volume	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)
Sediment	TAL Metals	Low	SW-846 3050, 6010/SOP-11, SOP-12, SOP-13	4 oz.	(1) 4 oz. sediment jar	4°C ± 2°C	6 months to analysis/28 days to analysis(Hg)
Aqueous field quality control samples	TAL Metals	Low	SW-846 3010, 6010B/SOP-10, SOP-12, SOP-14	(1) 500 mL	(1) 500 mL plastic	HNO <sub>3</sub> , pH <2	6 months to analysis/28 days to analysis(Hg)
Sediment	Select Pesticides	Low	SW-846 3540, 3550, 8081A/SOP-4, SOP-8, SOP-2	4 oz.	(1) 4 oz. sediment jar	4°C ± 2°C	14 Days to extraction/40 Days to analysis
Aqueous field quality control samples	Select Pesticides	Low	SW-846 3510, 3520, 8081A/SOP-7, SOP-2	(2) 1 Liter	(2) 1 Liter amber glass	4°C ± 2°C	7 Days to extraction/40 Days to analysis
Sediment	TCL PCB's	Low	SW-846 3540, 3550, 8082/SOP-4, SOP-5, SOP-3	4 oz.	(1) 4 oz. sediment jar	4°C ± 2°C	14 Days to extraction/40 Days to analysis
Aqueous field quality control samples	TCL PCB's	Low	SW8-46 3510, 3520, 8082/SOP-7, SOP-3	(2) 1 Liter	(2) 1 Liter amber glass	4°C ± 2°C	7 Days to extraction/40 Days to analysis

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**Analytical SOP Requirements Table**

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference <sup>1</sup>	Sample Volume	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/analysis)
Sediment	PAHs	Low	SW-846 3540, 3550, 8270C SIM/SOP-6, SOP-9, SOP-1	4 oz.	(1) 4 oz. sediment jar	4°C ± 2°C	14 Days to extraction/40 Days to analysis
Aqueous field quality control samples	PAHs	Low	SW846 3510, 3520, 8270C SIM/SOP-5, SOP-1	(2) 1 Liter	(2) 1 Liter amber glass	4°C ± 2°C	7 Days to extraction/40 Days to analysis
Sediment	TOC	Low	Lloyd Kahn/SOP-16	4 oz.	(1) 4 oz. sediment jar	4°C ± 2°C	14 Days to analysis
Sediment	pH	Low	pH/SOP-17	4 oz.	(1) 4 oz. sediment jar	4°C ± 2°C	14 Days to analysis

<sup>1</sup>Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

HNO<sub>3</sub> – nitric acid

TAL – Target Analyte List

TCL – Target Compound List

PAHs – Polycyclic Aromatic Hydrocarbons

SIM – Selective Ion Monitoring

TOC – Total Organic Carbon

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QAPP Worksheet #20**

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	Conc. Level	Analytical and Preparation SOP Reference <sup>1</sup>	No. of Sampling Locations <sup>2</sup>	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	PAHs	Low	SW-846 3540, 3550, 8270C SIM/SOP-6, SOP-9, SOP-1	18	2	1	0	0	0	23
Sediment	Select Pesticides	Low	SW-846 3540, 3550, 8081A/ SOP-4, SOP-8, SOP-2	18	2	1	0	0	0	23
Sediment	TCL PCBs	Low	SW-846 3540, 3550, 8082/SOP-4, SOP-5, SOP-3	18	2	1	0	0	0	23
Sediment	TAL Metals	Low	SW-846 3050, 6010/SOP-11, SOP-12, SOP-13	18	2	1	0	0	0	23
Sediment	TOC	Low	Lloyd Kahn/SOP-16	38	4	0	0	0	0	42
Sediment	pH	Low	pH/SOP-17	20	2	0	0	0	0	22
Aqueous field quality control samples	PAHs	Low	SW846 3510, 3520, 8270C SIM/SOP-5, SOP-1	0	0	0	1	1	0	2
Aqueous field quality control samples	Select Pesticides	Low	SW-846 3510, 3520, 8081A/SOP-7, SOP-2	0	0	0	1	1	0	2

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

Worksheet Not Applicable (State Reason)

**Field Quality Control Sample Summary Table**

Matrix	Analytical Group	Conc. Level	Analytical and Preparation SOP Reference <sup>1</sup>	No. of Sampling Locations <sup>2</sup>	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
Aqueous field quality control samples	TCL PCBs	Low	SW8-46 3510, 3520, 8082/SOP-7, SOP-3	0	0	0	1	1	0	2
Aqueous field quality control samples	TAL Metals	Low	SW-846 3010, 6010B/SOP-10, SOP-12, SOP-14	0	0	0	1	1	0	2

<sup>1</sup>Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet\_#23 )

<sup>2</sup>Worksheet #18 presents a list of the sample locations.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QAPP Worksheet #21**

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? (Y/N)	Comments
SA-7.1	Decontamination of Field Equipment	TtNUS	Decontamination Equipment (scrub brushes, phosphate free detergent, deionized water)	N	
CT-04	Sample Nomenclature	TtNUS	NA	N	
SA-6.3	Field Documentation	TtNUS	Field Logbook, Field Sample Forms, Boring Logs	N	
SA-6.1	Non-Radiological Sample Handling	TtNUS	Sample Bottleware, Packaging Material, Shipping Materials	N	
SA-1.2	Surface Water and Sediment Sampling	TtNUS	Sampling Procedures, Methods	N	
SA-1.3	Soil Sampling	TtNUS	Sampling Procedures, Methods	N	Although the samples that are being collected are considered sediment, some of the soil sampling procedures such as hand core and bucket auger may be utilized to collect the subsurface sediment samples.



**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QAPP Worksheet #22**

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)

**No field equipment requiring calibration is needed.**

**Field Equipment Calibration, Maintenance, Testing, and Inspection Table**

Field Equipment	Calibration Activity	Maint. Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference <sup>1</sup>

<sup>1</sup>Specify the appropriate reference letter or number from the Project Sampling SOP References table (Worksheet #21).

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON  
**Site Location:** Groton, Connecticut

**Revision Number:** 0  
**Revision Date:** October 2007

**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**

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
SOP-1	"Analysis of Semivolatile Organic Compounds by: SW 846 Method 8270 - Modified for Selected Ion Monitoring (SIM)", SOP No. CA-213, Revision 3, 04/06.	Definitive	PAHs	GC/MS	Katahdin Analytical Services	No
SOP-2	"Analysis of Pesticides By Gas Chromatography/Electron Capture Detector (GC/ECD): SW-846 Method 8081", SOP No. CA-302, Revision 8, 06/07.	Definitive	Select Pesticides	GC/ECD	Katahdin Analytical Services	No
SOP-3	"Analysis Of PCBs As Total Arochlors By Gas Chromatography/Electron Capture Detector (GC/ECD): SW-846 Method 8082", SOP No. CA-329, Revision 6, 04/06.	Definitive	TCL PCBs	GC/ECD	Katahdin Analytical Services	No
SOP-4	"Preparation of Sediment/Soil Samples by Sonication using Method 3550 for subsequent Pesticides/PCBs Analysis", SOP No. CA-500, Revision 3, 04/06.	Definitive	TCL PCBs and Select Pesticides	Ultrasonic Extractions	Katahdin Analytical Services	No
SOP-5	"Preparation Of Aqueous Samples For Extractable Semivolatile Analysis", SOP No. CA-502, Revision 3, 04/06.	Definitive	PAHs	Separatory Funnel/CLLE	Katahdin Analytical Services	No
SOP-6	"Preparation of Sediment/Soil Samples by Sonication using Method 3550 for subsequent Extractable Semi-Volatile Analysis", SOP No. CA-512, Revision 4, 04/06.	Definitive	PAHs	Ultrasonic Extraction	Katahdin Analytical Services	No

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**Analytical SOP References Table**

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?
SOP-7	"Preparation of Aqueous Samples for Pesticides/PCBs Analysis", SOP No. CA-515, Revision 3, 04/06.	Definitive	TCL PCBs and Select Pesticides	Separatory Funnel/CLLE	Katahdin Analytical Services	No
SOP-8	"Preparation of Sediment/Soil Samples by Soxhlet Extraction Using Method 3540 for Pesticide/PCB Analysis", SOP No. CA-524, Revision 3, 04/06.	Definitive	TCL PCBs and Select Pesticides	Soxhlet Extraction	Katahdin Analytical Services	No
SOP-9	"Preparation of Sediment/Soil Samples by Soxhlet Extraction for Subsequent Extractable Semivolatile Analysis", SOP No. CA-526, Revision 3, 04/06.	Definitive	PAHs	Soxhlet Extraction	Katahdin Analytical Services	No
SOP-10	"Acid Digestion of Aqueous Samples by EPA Method 3010 for ICP Analysis of Total or Dissolved Metals", SOP No. CA-604, Revision 3, 04/06.	Definitive	TAL Metals	Acid Digestion	Katahdin Analytical Services	No
SOP-11	"Acid Digestion of Solid Samples by USEPA Method 3050 for Metals by ICP-AES and GFAA", 3050B, SOP No. CA-605, Revision 2, 03/06.	Definitive	TAL Metals	Acid Digestion	Katahdin Analytical Services	No
SOP-12	"Trace Metals Analysis By ICP-AES Using EPA Method 6010", SOP No. CA-608, Revision 4, 05/06.	Definitive	TAL Metals	ICP	Katahdin Analytical Services	No
SOP-13	"Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471", SOP No. CA-611, Revision 3, 03/06.	Definitive	Mercury	CVAA	Katahdin Analytical Services	No
SOP-14	"Digestion And Analysis Of Aqueous Samples For Mercury By USEPA Method 7470", SOP No. CA-615, Revision 1, 04/06.	Definitive	Mercury	CVAA	Katahdin Analytical Services	No
SOP-15	"pH Concentration Measurements In Soil Matrices - SW 846 Method 9045" SOP No. CA-709, Revision 6, 02/07.	Definitive	pH	pH Meter	Katahdin Analytical Services	No

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

**Analytical SOP References Table**

<b>Reference Number</b>	<b>Title, Revision Date, and/or Number</b>	<b>Definitive or Screening Data</b>	<b>Analytical Group</b>	<b>Instrument</b>	<b>Organization Performing Analysis</b>	<b>Modified for Project Work?</b>
SOP-16	"Determination of Total Organic Carbon in Solids using the EPA Region II Lloyd Kahn Method", SOP No. CA-741, Revision 1, 01/07	Definitive	TOC	TOC Analyzer	Katahdin Analytical Services	No
SOP-17	"Sample Receipt and Internal Control", SOP No. SD-902, Revision 7, 02/07	Definitive	Various	Various	Katahdin Analytical Services	No

CLLE – Continuous Liquid-Liquid Extraction

CVAA – Cold Vapor Atomic Absorption

GC/ECD - Gas Chromatography/Electron Capture Detector

GC/MS – Gas Chromatography/Mass Spectroscopy

ICP – Inductively Coupled Plasma

PAHs – Polycyclic Aromatic Hydrocarbons

SIM – Selective Ion Monitoring

TAL – Target Analyte List

TCL – Target Compound List

TOC – Total Organic Carbon

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**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	SOP Reference <sup>1</sup>
TAL Metals - ICP	Initial Calibration	At the beginning of each day or if QC is out of criteria.	One point calibration per manufacturer's guidelines; analytes run at their calibration levels must fall within 95-105% of True Values	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Supervisor	SOP-12
	Continuing Calibration	At the beginning and end of each run sequence and every 10 samples	90-110% of True Values	Check problem, recalibrate and reanalyze any samples not bracketed by passing CCVs.	Analyst, Supervisor	SOP-12
Mercury Analyzer	Initial Calibration	IC-instrument receipt, major instrument change, at the start of each day	Correlation coefficient $\geq 0.995$ .	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Supervisor	SOP-13, SOP-14
	Continuing Calibration	CCV-at beginning and end of each run sequence and every 10 samples	80-120% of True Value	Check problem, recalibrate and reanalyze any samples not bracketed by passing CCVs.	Analyst, Supervisor	SOP-13, SOP-14

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON  
**Site Location:** Groton, Connecticut

**Revision Number:** 0  
**Revision Date:** October 2007

**Analytical Instrument Calibration Table**

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference <sup>1</sup>
Select Pesticides - GC	Initial Calibration	IC-instrument receipt, major instrument change, when CC does not meet criteria	6 pt calibration of all pesticides except for toxaphene and chlordane - coefficient of determination $\geq 0.990$ . Mid-point cal of toxaphene and chlordane.	Repeat Initial calibration and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Supervisor	SOP-2
	Continuing Calibration	After every 10 samples; If calibration curve previously analyzed, analyze daily before samples.	%D $\leq 15$ for both the quantitation and confirmation columns	Evaluate the samples: If the %D $> +15\%$ and sample results are $< PQL$ , narrate. If %D $> \pm 15\%$ only on one channel, narrate. If %D $> \pm 15\%$ for closing CV, and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples back to last acceptable CV.	Analyst, Supervisor	SOP-2

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**Analytical Instrument Calibration Table**

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference <sup>1</sup>
TCL PCBs - GC	Initial Calibration	IC-instrument receipt, major instrument change, when CC does not meet criteria	6 pt calibration of aroclor 1660 – coefficient of determination $\geq$ 0.990 Mid-point cal of other Aroclors	Repeat Initial calibration and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data. If single pt cal Aroclor is identified in analysis of sample, 6-pt calibration run of identified compound with reanalysis of sample.	Analyst, Supervisor	SOP-3
	Continuing Calibration	After every 10 samples; if calibration curve previously analyzed, analyze daily before samples.	%D $\leq$ 15 for both the quantitation and confirmation columns	Evaluate the samples: If the %D $>$ +15% and sample results are $<$ RQL, narrate. If %D $>$ $\pm$ 15% only on one channel, narrate. If %D $>$ $\pm$ 15% for closing CV, and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples back to last acceptable CV.	Analyst, Supervisor	SOP-3

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**Analytical Instrument Calibration Table**

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference <sup>1</sup>
TOC Analyzer	Initial Calibration	Initial Calibration- initially, when the daily CCV does not pass, but, no longer than every 3 months.	Correlation coefficient $\leq 0.995$	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Supervisor	SOP-16
	Continuing Calibration	CCV-every 10 samples and at the end of the run	75-125% of true value for Lloyd Kahn	If the CCV fails high, report samples that are <PQL. Recalibrate and/or reanalyze samples back to last acceptable CCV recovery.	Analyst, Supervisor	SOP-16
PAHs – GC/MS/ SIM	Initial Calibration	IC – Instrument receipt, instrument change (new column, source cleaning, etc.), when CCC is out of criteria or when manual tune performed	IC – minimum RF of $\geq 0.050$ for each SPCC, % RSD of $\leq 30\%$ for each CCC. If RSD for an analyte is $> 15\%$ apply linear or quadratic method for quantitation	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Supervisor	SOP-1
	Calibration Check	CV – at the beginning of each 12 hour shift immediately after DFTPP tune.	CV – minimum RF of each SPCC $\geq 0.050$ , % RSD $\leq 20\%$ for each CCC.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Supervisor	SOP-1

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**Analytical Instrument Calibration Table**

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference <sup>1</sup>
PAHs – GC/MS/ SIM	DFTPP Tune	Every 12 hours	Criteria listed in section 7.4 current rev. of SOP CA-213	Retune and/or clean source	Analyst, Supervisor	SOP-1
pH Meter	Initial Calibration	Once per day	± 0.05 pH units for every buffer	If calibration is not achieved, check meter, buffer solutions, and probe; replace if necessary; repeat calibration	Analyst, Supervisor	SOP-15

<sup>1</sup>Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #230).

%D – Percent Difference

CCC- Continuing Calibration Compound

CCV – Continuing Calibration Verification

CV – Calibration Verification

CVAA – Cold Vapor Atomic Absorption

DFTPP - Decafluorotriphenylphosphine

GC/ECD - Gas Chromatography/Electron Capture Detector

GC/MS – Gas Chromatography/Mass Spectroscopy

Hg – Mercury

IC – Initial Calibration

ICP – Inductively Coupled Plasma

ICV – Initial Calibration Verification

MS – Mass Spectroscopy

PAHs – Polycyclic Aromatic Hydrocarbons

PCBs – Polychlorinated Biphenyls

PQL – Practical Quantitation Limit

QC – Quality Control

R – Correlation Coefficient

RF – Response factor

RSD – Relative Standard Deviation

SIM – Selective Ion Monitoring

SOP – Standard Operating Procedure

SPCC – System performance check compounds

TAL – Target Analyte List

TCL – Target Compound List

TOC – Total Organic Carbon

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**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	SOP Reference <sup>1</sup>
PAHs - GC/MS	Check pressure and gas supply daily. Manual tune if DFTPP not in criteria, change septa as needed, change liner as needed, cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP	QC standards	Ion source, injector liner, column, column flow.	Prior to initial calibration and/or as necessary	Refer to SOP	Refer to SOP	Analyst, Department Manager	SOP-1

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON  
**Site Location:** Groton, Connecticut

**Revision Number:** 0  
**Revision Date:** October 2007

**Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table**

<b>Instrument/ Equipment</b>	<b>Maintenance Activity</b>	<b>Testing Activity</b>	<b>Inspection Activity</b>	<b>Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>	<b>Responsible Person</b>	<b>SOP Reference<sup>1</sup></b>
TAL Metals - ICP	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, replace peristaltic pump tubing as needed. Other maintenance specified in lab Equipment Maintenance SOP.	QC standards	Torch, nebulizer chamber, pump, pump tubing	Prior to initial calibration and as necessary	Refer to SOP	Refer to SOP	Analyst, Department Manager	SOP-12

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table**

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference <sup>1</sup>
Mercury - CVAA	Replace peristaltic pump tubing, replace mercury lamp, replace drying tube, clean optical cell and/or clean liquid/gas separator as needed. Other maintenance specified in lab Equipment Maintenance SOP.	QC standards	Tubing, sample probe, optical cell	Prior to initial calibration and as necessary	Refer to SOP	Refer to SOP	Analyst, Department Manager	SOP-13, SOP-14
TOC Combustion Analyzer	Check level of dilution water, drain vessel water, humidifier water, autosampler rinse water and phosphoric acid vessel and fill as needed. Replace oxygen cylinder.	QC standards	Tubing, sample boat, syringe, humidifier, rinse Reservoir, phosphoric acid vessel, oxygen pressure	Prior to initial calibration and as necessary	Refer to SOP	Refer to SOP	Analyst, Department Manager	SOP-16

Project-Specific QAPP

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table**

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference <sup>1</sup>
Select Pesticides - GC	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	QC standards	Injector liner, septa, column, column flow.	Prior to initial calibration and/or as necessary.	Refer to SOP	Refer to SOP	Analyst, Department Manager	SOP-2
TCL PCBs - GC	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	QC standards	Injector liner, septa, column, column flow.	Prior to initial calibration and/or as necessary.	Refer to SOP	Refer to SOP	Analyst, Department Manager	SOP-1
pH meter	Clean probe	QC standards	probe	As necessary	Refer to SOP	Refer to SOP	Analyst, Department Manager	SOP-15

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

<sup>1</sup>Specify the appropriate reference letter or number from Analytical SOP References table (Worksheet #23).

CVAA – Cold Vapor Atomic Absorption

DFTPP - Decafluorotriphenylphosphine

GC/ECD - Gas Chromatography/Electron Capture Detector

GC/MS – Gas Chromatography/Mass Spectroscopy

ICP – Inductively Coupled Plasma

ICV – Initial calibration verification

PAHs – Polycyclic Aromatic Hydrocarbons

PCBs – Polychlorinated biphenyls

QC – Quality Control

SOP – Standard Operating Procedure

TAL – Target Analyte List

TCL – Target Compound List

TOC – Total Organic Carbon

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Welland – Site 2B

Site Name/Project Name: NSB - NLON  
Site Location: Groton, Connecticut

Revision Number: 0  
Revision Date: October 2007

**QAPP Worksheet #26**

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**

<b>SAMPLE COLLECTION, PACKAGING, AND SHIPMENT</b>
Sample Collection (Personnel/Organization): FOL (TBD)/TtNUS
Sample Packaging (Personnel/Organization): FOL (TBD)/TtNUS
Coordination of Shipment (Personnel/Organization): FOL (TBD)/TtNUS
Type of Shipment/Carrier: Overnight courier service (Federal Express)
<b>SAMPLE RECEIPT AND ANALYSIS</b>
Sample Receipt (Personnel/Organization): Sample custodians/ Katahdin Analytical Services, Inc.
Sample Custody and Storage (Personnel/Organization): Sample custodians/ Katahdin Analytical Services, Inc.
Sample Preparation (Personnel/Organization): Preparation Laboratory Staff/ Katahdin Analytical Services, Inc.
Sample Determinative Analysis (Personnel/Organization): GC/MS, ICP, GC/ECD, Spectrophotometer/ Katahdin Analytical Services, Inc.
<b>SAMPLE ARCHIVING</b>
Field Sample Storage (No. of days from sample collection): Refer to QAPP Worksheet # 19
Sample Extract/Digestate Storage (No. of days from extraction/digestion): Refer to QAPP Worksheet # 19
Biological Sample Storage (No. of days from sample collection): N/A
<b>SAMPLE DISPOSAL</b>
Personnel/Organization: Sample custodians/ Katahdin Analytical Services, Inc.
Number of Days from Analysis: Samples may be disposed of 2 months after data is reported

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON  
**Site Location:** Groton, Connecticut

**Revision Number:** 0  
**Revision Date:** October 2007

**QAPP\_Worksheet\_#27**

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): Samples for the laboratory will be handled, packaged and shipped in accordance with TtNUS SOP SA-6.1.
Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal): Refer to Katahdin Analytical Services SOP SD-902
Sample Identification Procedures: Sample nomenclature will be conducted in general accordance with the procedures outlined in TtNUS SOP CT-04 (Sample Nomenclature). Additional information regarding protocol for sample labeling is contained in TtNUS SOP SA-6.3. Refer to Katahdin Analytical Services SOP SD-902
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. Refer to Katahdin Analytical Services SOP SD-902.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**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)

**QC Samples Table**

Matrix	Aqueous field quality control samples
Analytical Group	TAL Metals
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 6010B/SOP-12
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Independent Calibration Verification (ICV)	Immediately after calibration	± 10 %	Correct problem, recalibrate and reanalyze ICV	Analyst/Supervisor, QA Manager	Accuracy/bias	± 10 %
Initial Calibration Blank (ICB)	Immediately after the ICV	≤PQL	Correct problem, recalibrate and reanalyze ICV and ICB	Analyst/Supervisor, QA Manager	Accuracy/bias, Contamination	≤PQL

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control samples
Analytical Group	TAL Metals
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846-6010B/SOP-12
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
PQL Standard for ICP (PQL)	At the beginning of a sample run, after every 20 samples and at the end of the run	Recovery within 70% - 130 % of true value. For Sb, Pb & Tl recovery within 50% - 150% of true value.	1. Reanalyze immediately for failing elements only. 2. Terminate analysis, correct problem, recalibrate and reanalyze all analytical samples analyzed since last good PQL Std.	Analyst/Supervisor, QA Manager	Sensitivity	Recovery within 70% - 130 % of true value. For Sb, Pb & Tl recovery within 50% - 150% of true value.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control samples
Analytical Group	TAL Metals
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 6010B/SOP-12
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Preparation Blank (PBW)	One per prep batch of twenty or fewer samples of similar matrix	Absolute value < PQL.	1. If blank value > PQL report sample results if < PQL or > 10 x the blank value; otherwise redigest. 2. If blank value is less than negative PQL, report sample results if > 10x the absolute value of the blank result, otherwise redigest.	Analyst/Supervisor, QA Manager	Accuracy/bias-	Absolute value < PQL. Sample results if > 10x the absolute value of the blank result, otherwise redigest.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control samples					
Analytical Group	TAL Metals					
Concentration Level	Low					
Sampling SOP	SA-1.2 and SA-1.3					
Analytical Method/SOP Reference	SW-846 6010B/SOP-12					
Sampler's Name	TBD					
Field Sampling Organization	TtNUS					
Analytical Organization	Katahdin Analytical Services					
No. of Sample Locations	2 blanks					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Serial Dilution (L)	One per prep batch of twenty or fewer samples of similar matrix	If original sample result is at least 50x IDL, 5-fold dilution must agree within $\pm 10\%$ of the original result.	Flag results for affected analytes for all associated samples with "E".	Analyst/Supervisor, QA Manager	Accuracy/Bias, Precision	If original sample result is at least 50x IDL, 5-fold dilution must agree within $\pm 10\%$ of the original result.
Laboratory Control Sample (LCSW)	One per prep batch of twenty or fewer samples of similar matrix	Recovery within $\pm 20\%$ of true value.	Redigest and reanalyze all associated samples for affected analyte.	Analyst/Supervisor, QA Manager	Accuracy/Bias	Recovery within reference limits supplied by SRM vendor.



**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control samples
Analytical Group	TAL Metals
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 6010B/SOP-12
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Sample (D)	One per prep batch of twenty or fewer samples of similar matrix	RPD $\leq$ 20%	Flag results for affected analytes for all associated samples with "*".	Analyst/Supervisor, QA Manager	Accuracy/Bias, Precision	RPD $\pm$ 20%
Matrix Spike Duplicate (P)	One per prep batch of twenty or fewer samples of similar matrix	Recovery $\pm$ 25 % of true value if sample < 4x spike value	Flag results for affected analytes for all associated samples with "N", Perform postdigestion spike for all failing elements, except Ag, at 2x the indigenous level or 2x the PQL, whichever is greater.	Analyst/Supervisor, QA Manager	Accuracy/bias	Recovery $\pm$ 25 % of true value if sample < 4x spike value

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

CA – Corrective Action

EICP – Extracted Ion Current Profile

PQL – Practical Quantitation Limit

QA – Quality Assurance

RPD – Relative Percent Difference

SRM – Standard Reference Material

TAL – Target Analyte List

TBD – To Be Determined

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	TAL Metals
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 6010B/SOP-12
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Independent Calibration Verification (ICV)	Immediately after calibration	± 10% of true value	Correct problem, recalibrate and reanalyze ICV	Analyst/Supervisor, QA Manager	Accuracy/bias	± 10% of true value
Initial Calibration Blank (ICB)	Immediately after the ICV	≤PQL	Correct problem, recalibrate and reanalyze ICV and ICB	Analyst/Supervisor, QA Manager	Accuracy/bias, Contamination	≤PQL
PQL Standard for ICP (PQL)	At the beginning of a sample run, after every 20 samples and at the end of the run	Recovery within 70% - 130 % of true value.	1. Reanalyze immediately for failing elements only. 2. Terminate analysis, correct problem, recalibrate and reanalyze all analytical samples analyzed since last good PQL Std.	Analyst/Supervisor, QA Manager	Sensitivity	Recovery within 70% - 130 % of true value.

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	TAL Metals
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 6010B/SOP-12
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

<b>QC Sample:</b>	<b>Frequency/Number</b>	<b>Method/SOP QC Acceptance Limits</b>	<b>Corrective Action (CA)</b>	<b>Person(s) Responsible for Corrective Action</b>	<b>Data Quality Indicator (DQI)</b>	<b>Measurement Performance Criteria</b>
Preparation Blank (PBS)	One per prep batch of twenty or fewer samples of similar matrix	Absolute value < PQL.	1. If blank value > PQL report sample results if < PQL or > 10 x the blank value; otherwise redigest. 2. If blank value is less than negative PQL, report sample results if > 10x the absolute value of the blank result, otherwise redigest.	Analyst/Supervisor, QA Manager	Accuracy/bias	Absolute value < PQL. Sample results if > 10x the absolute value of the blank result, otherwise redigest.

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	TAL Metals
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 6010B/SOP-12
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Serial Dilution (L)	One per prep batch of twenty or fewer samples of similar matrix	If original sample result is at least 50x IDL, 5-fold dilution must agree within $\pm 10\%$ of the original result.	Flag results for affected analytes for all associated samples with "E".	Analyst/Supervisor, QA Manager	Accuracy/Bias, Precision	If original sample result is at least 50x IDL, 5-fold dilution must agree within $\pm 10\%$ of the original result.
Laboratory Control Sample (LCSS)	One per prep batch of twenty or fewer samples of similar matrix	Recovery within reference limits supplied by SRM vendor.	Redigest and reanalyze all associated samples for affected analyte (except Ag and Sb)	Analyst/Supervisor, QA Manager	Accuracy/Bias	Recovery within reference limits supplied by SRM vendor.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	TAL Metals
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 6010B/SOP-12
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Sample Duplicate (D)	One per prep batch of twenty or fewer samples of similar matrix	RPD $\leq$ 20%, if sample and duplicate $\geq$ 5x PQL; $\pm$ PQL if sample or duplicate $<$ 5x PQL.	Flag results for affected analytes for all associated samples with "*".	Analyst/Supervisor, QA Manager	Accuracy/Bias, Precision	RPD $\leq$ 20%, if sample and duplicate $\geq$ 5x PQL; $\pm$ PQL if sample or duplicate $<$ 5x PQL.
Matrix Spike Sample (S)	One per prep batch of twenty or fewer samples of similar matrix	Recovery $\pm$ 25 % of true value if sample $<$ 4x spike value	Flag results for affected analytes for all associated samples with "N", Perform postdigestion spike for all failing elements, except Ag, at 2x the indigenous level or 2x the PQL, whichever is greater.	Analyst/Supervisor, QA Manager	Accuracy/bias	Recovery $\pm$ 25 % of true value if sample $<$ 4x spike value

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	TAL Metals
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 6010B/SOP-12
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Sample Duplicate (D)	One per prep batch of twenty or fewer samples of similar matrix	RPD $\leq$ 20%	Flag results for affected analytes for all associated samples with "**".	Analyst/Supervisor, QA Manager	Accuracy/Bias, Precision	RPD $\leq$ 20%

CA – Corrective Action

EICP – Extracted ion current profile

PQL – Practical Quantitation Limit

QA – Quality Assurance

RPD – Relative Percent Difference

SRM – Standard Reference Material

TAL – Target Analyte List

TBD – To Be Determined

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment					
Analytical Group	PAHs					
Concentration Level	Low					
Sampling SOP	SA-1.2 and SA-1.3					
Analytical Method/SOP Reference	SW-846 8270C SIM\SOP-1					
Sampler's Name	TBD					
Field Sampling Organization	TtNUS					
Analytical Organization	Katahdin Analytical Services					
No. of Sample Locations	20					
QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per prep batch of twenty or fewer samples of similar matrix	No analytes detected > PQL.	(1) Investigate source of contamination (2) Reprep and analyze method blank and all samples processed with the contaminated blank	Analyst, Supervisor, QA Manager	Accuracy/Bias, Contamination	No analytes detected > PQL.
Matrix Spike (MS)	One per prep batch of twenty or fewer samples of similar matrix	Statistically derived acceptance limits.	CA will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Statistically derived acceptance limits.

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	PAHs
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8270C SIMSOP-1
Sampler's Name	TBD
Field Sampling Organization	TINUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

<b>QC Sample:</b>	<b>Frequency/Number</b>	<b>Method/SOP QC Acceptance Limits</b>	<b>Corrective Action (CA)</b>	<b>Person(s) Responsible for Corrective Action</b>	<b>Data Quality Indicator (DQI)</b>	<b>Measurement Performance Criteria</b>
Matrix Spike Duplicate (MSD)	One per prep batch of twenty or fewer samples of similar matrix	≤ 50% RPD.	CA will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.	Analyst, Supervisor, QA Manager	Accuracy/Bias, Precision	≤ 50% RPD

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	PAHs
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8270C SIM/SOP-1
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Spike (LCS)	One per prep batch of twenty or fewer samples of similar matrix.	Statistically derived acceptance limits.	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS rec. is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Statistically derived acceptance limits.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	PAHs
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8270C SIM\SOP-1
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate spike	Every sample, control, standard, and method blank	Statistically derived acceptance limits.	(1) File Katahdin CAR (2) Check chromatogram for interference; if found, flag data (3) If not found, check instrument performance; if problem is found, correct and reanalyze(4) If still out, reextract and analyze sample (5) If reanalysis is out, flag data	Analyst, Supervisor, QA Manager	Accuracy/Bias	NA

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON  
**Site Location:** Groton, Connecticut

**Revision Number:** 0  
**Revision Date:** October-2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	PAHs
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8270C SIM\SOP-1
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal Standards	Every sample, control, standard, and method blank	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect Mass spectrometer or GC for malfunctions: mandatory reanalysis of samples analyzed while system was malfunctioning. If reanalysis confirms matrix interference, report sample and narrate.	Analyst, Supervisor, QA Manager	Accuracy/Bias	NA

CA – Corrective Action

EICP – Extracted Ion Current Profile

PAHs – Polycyclic Aromatic Hydrocarbons

PQL – Practical Quantitation Limit

QA – Quality Assurance

RPD – Relative Percent Difference

SIM – Selective Ion Monitoring

SRM – Standard Reference Material

TBD – To Be Determined



**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control sample
Analytical Group	PAHs
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8270C SIM\SOP-1
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blank	One per prep batch of twenty or fewer samples of similar matrix	No analytes detected > PQL	(1) Investigate source of contamination (2) Reprep and analyze method blank and all samples processed with the contaminated blank	Analyst, Supervisor, QA Manager	Accuracy/Bias, Contamination	No analytes detected > PQL
Matrix Spike (MS)	One per prep batch of twenty or fewer samples of similar matrix	Statistically derived acceptance limits.	CA will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Statistically derived acceptance limits.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control sample					
Analytical Group	PAHs					
Concentration Level	Low					
Sampling SOP	SA-1.2 and SA-1.3					
Analytical Method/SOP Reference	SW-846 8270C SIMSOP-1					
Sampler's Name	TBD					
Field Sampling Organization	TtNUS					
Analytical Organization	Katahdin Analytical Services					
No. of Sample Locations	2 blanks					
QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicate (MSD)	One per prep batch of twenty or fewer samples of similar matrix	≤30% RPD.	CA will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.	Analyst, Supervisor, QA Manager	Accuracy/Bias, Precision	≤30% RPD



**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control sample
Analytical Group	PAHs
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8270C SIM\SOP-1
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Spike (LCS)	One per prep batch of twenty or fewer samples of similar matrix.	Statistically derived acceptance limits.	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS rec. is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Statistically derived acceptance limits.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control sample
Analytical Group	PAHs
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8270C SIM/SOP-1
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate spike	Every sample, control, standard, and method blank	Statistically derived acceptance limits.	(1) File Katahdin CAR (2) Check chromatogram for interference; if found, flag data (3) If not found, check instrument performance; if problem is found, correct and reanalyze(4) If still out, reextract and analyze sample (5) If reanalysis is out, flag data	Analyst, Supervisor, QA Manager	Accuracy/Bias	NA

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control sample
Analytical Group	PAHs
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8270C SIM\SOP-1
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal Standards	Every sample, control, standard, and method blank	Retention time $\pm$ 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect Mass spectrometer or GC for malfunctions: mandatory reanalysis of samples analyzed while system was malfunctioning. If reanalysis confirms matrix interference, report sample and narrate.	Analyst, Supervisor, QA Manager	Accuracy/Bias	NA

CA – Corrective Action

EICP – Extracted Ion Current Profile

PAHs – Polycyclic Aromatic Hydrocarbons

PQL – Practical Quantitation Limit

QA – Quality Assurance

RPD – Relative Percent Difference

SIM – Selective Ion Monitoring

SRM – Standard Reference Material

TBD – To Be Determined

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	TOC
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	Lloyd Kahn/SOP-16
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per 20 samples	No analyte > PQL	Investigate source of contamination. Report all sample results > 10 x the blank result and flag results with "B". Reprep and analyze method blank and all other samples processed with the contaminated blank.	Analyst, Supervisor, QA Manager	Accuracy/Bias, Contamination	No analyte > PQL
Instrument Blank	After each ICV and CCV,	No analyte >PQL	Samples analyzed before or after an unacceptable blank will be reanalyzed.	Analyst, Supervisor, QA Manager	Accuracy/Bias, Contamination	No analyte > PQL
Laboratory Quadruplicate	One sample quadruplicate per 20 samples.	RSD < 30%	If lab QC in criteria and matrix interference suspected, flag data. Else, reanalyze.	Analyst, Supervisor, QA Manager	Precision	RSD < 30%

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment					
Analytical Group	TOC					
Concentration Level	Low					
Sampling SOP	SA-1.2 and SA-1.3					
Analytical Method/SOP Reference	Lloyd Kahn/SOP-16					
Sampler's Name	TBD					
Field Sampling Organization	TtNUS					
Analytical Organization	Katahdin Analytical Services					
No. of Sample Locations	20					
QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike (MS)	One per 10 samples	75-125 % recovery	If LCS in criteria and matrix interference suspected, flag data. Else, reanalyze.	Analyst, Supervisor, QA Manager	Accuracy/Bias	75-125 % recovery
Laboratory Control Sample(LCS)	One per 20 samples	80-120% of true value	Investigate source of problem. If the LCS fails high, report samples that are < PQL. Reprep the remaining samples.	Analyst, Supervisor, QA Manager	Accuracy/Bias	80-120% of true value

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	TOC
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	Lloyd Kahn/SOP-16
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Low-level Calibration Sample	With each initial calibration	Low-level calibration standard in the initial calibration is spiked at or below the QL. Initial calibration acceptance criteria is a correlation coefficient of > 0.995.	Reanalyze sample	Analyst, Supervisor, QA Manager	Accuracy/Bias	Low-level calibration standard in the initial calibration is spiked at or below the QL. Initial calibration acceptance criteria is a correlation coefficient of > 0.995.

CA – Corrective Action

EICP – Extracted Ion Current Profile

PQL – Practical Quantitation Limit

QA – Quality Assurance

RPD – Relative Percent Difference

RSD – Relative Standard Deviation

SRM – Standard Reference Material

TBD – To Be Determined

TOC – Total Organic Carbon

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment					
Analytical Group	Select Pesticides					
Concentration Level	Low					
Sampling SOP	SA-1.2 and SA-1.3					
Analytical Method/SOP Reference	SW-846 8081A\SOP-2					
Sampler's Name	TBD					
Field Sampling Organization	TiNUS					
Analytical Organization	Katahdin Analytical Services					
No. of Sample Locations	20					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch of twenty or fewer samples of similar matrix	No analyte detected >QL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e., if the blank results are above the QL, report sample results which are <QL or > 10x the blank concentration. Otherwise, reprep a blank and samples >QL and <10x QL.	(1) Analyst/ Supervisor (2) Supervisor (3) Analyst	Accuracy/bias, Contamination	No target > QL

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	Select Pesticides
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8081A/SOP-2
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Reagent Blank	1 per Lot	No analyte detected $\geq$ QL	(1) Investigate source of contamination (2) If required replace Lot	Analyst/ Supervisor	Accuracy/bias, Contamination	No target $\geq$ QL



**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	Select Pesticides
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8081A\SOP-2
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike	One per prep batch of twenty or fewer samples of similar matrix	Statistically derived limits	(1) No CA will be taken for Samples where recoveries are outside limits and surrogate and LCS criteria are met. (2) Where LCS is low outside criteria, a LCS, MS (sample <2.5 ug/L), and MSD may be re-extracted if Project Completeness < Completeness Criteria	Analyst/ Supervisor	Accuracy/bias	Statistically derived limits

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment					
Analytical Group	Select Pesticides					
Concentration Level	Low					
Sampling SOP	SA-1.2 and SA-1.3					
Analytical Method/SOP Reference	SW-846 8081A\SOP-2					
Sampler's Name	TBD					
Field Sampling Organization	TtNUS					
Analytical Organization	Katahdin Analytical Services					
No. of Sample Locations	20					
QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicate	One per prep batch of twenty or fewer samples of similar matrix	≤ 50% RPD	(1) In the event that the MS or MSD surrogates are outside criteria, the MS/MSD will be re-extracted and reanalyzed else no CA will be taken when both MS and MSD surrogates are in criteria	Analyst/Supervisor	Accuracy/bias and Precision	Statistically derived acceptance limits, Precision RPD ≤ 50%



**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	Select Pesticides
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8081A\SOP-2
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	One per prep batch of twenty or fewer samples of similar matrix	Statistically derived acceptance limits.	(1) If an MS/MSD was performed and acceptable, narrate. (2) If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. (3) If the LCS recovery is high but the sample results are <QL, narrate. Otherwise, re-extract blank and affected sample batch.	Analyst/ Supervisor	Accuracy/bias	Statistically derived acceptance limits.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	Select Pesticides
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8081A\SOP-2
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample;	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogates	2 per Sample	Statistically derived acceptance limits.	(1) No CA will be taken when one surrogate is within criteria. (2) If surrogates are outside high and sample is <QL no CA taken. (3) If surrogates are outside low the affected samples are re-extracted and reanalyzed.	Analyst/ Supervisor	Accuracy/bias	Statistically derived acceptance limits.

CA – Corrective Action  
 EICP – Extracted Ion Current Profile  
 PQL – Practical Quantitation Limit  
 QA – Quality Assurance

RPD – Relative Percent Difference  
 SRM – Standard Reference Material  
 TBD – To Be Determined  
 TCL – Target Compound List

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control sample
Analytical Group	Select Pesticides
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8081A\SOP-2
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch of twenty or fewer samples of similar matrix	No analyte detected >QL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the QL, report sample results which are <QL or > 10x the blank concentration. Otherwise, reprep a blank and samples >QL and <10x QL.	(1) Analyst/ Supervisor (2) Supervisor (3) Analyst	Accuracy/bias, Contamination	No target > QL

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control sample					
Analytical Group	Select Pesticides					
Concentration Level	Low					
Sampling SOP	SA-1.2 and SA-1.3					
Analytical Method/SOP Reference	SW-846 8081A\SOP-2					
Sampler's Name	TBD					
Field Sampling Organization	TtNUS					
Analytical Organization	Katahdin Analytical Services					
No. of Sample Locations	2 blanks					
<b>QC Sample:</b>	<b>Frequency/Number</b>	<b>Method/SOP QC Acceptance Limits</b>	<b>Corrective Action (CA)</b>	<b>Person(s) Responsible for Corrective Action</b>	<b>Data Quality Indicator (DQI)</b>	<b>Measurement Performance Criteria</b>
Reagent Blank	1 per Lot	No analyte detected $\geq$ QL	(1) Investigate source of contamination (2) If required replace Lot	Analyst/ Supervisor	Accuracy/bias-Contamination	No target $\geq$ QL



**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control sample
Analytical Group	Select Pesticides
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8081A\SOP-2
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike	One per prep batch of twenty or fewer samples of similar matrix	Statistically derived limits	(3) No CA will be taken for Samples where recoveries are outside limits and surrogate and LCS criteria are met. (4) Where LCS is low outside criteria, a LCS, MS (sample <2.5 ug/L), and MSD may be re-extracted if Project Completeness < Completeness Criteria	Analyst/ Supervisor	Accuracy/bias	Statistically derived limits

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON  
**Site Location:** Groton, Connecticut

**Revision Number:** 0  
**Revision Date:** October 2007

**QC Samples Table**

Matrix	Aqueous field quality control sample					
Analytical Group	Select Pesticides					
Concentration Level	Low					
Sampling SOP	SA-1.2 and SA-1.3					
Analytical Method/SOP Reference	SW-846 8081A\SOP-2					
Sampler's Name	TBD					
Field Sampling Organization	TtNUS					
Analytical Organization	Katahdin Analytical Services					
No. of Sample Locations	2 blanks					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicate	One per prep batch of twenty or fewer samples of similar matrix	≤30% RPD	(1) In the event that the MS or MSD surrogates are outside criteria, the MS/MSD will be re-extracted and reanalyzed else no CA will be taken when both MS and MSD surrogates are in criteria	Analyst/ Supervisor	Accuracy/bias and Precision	Statistically derived acceptance limits, Precision RPD ≤30%



**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control sample
Analytical Group	Select Pesticides
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8081A\SOP-2
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	One per prep batch of twenty or fewer samples of similar matrix	Statistically derived acceptance limits.	(2) If an MS/MSD was performed and acceptable, narrate. (2) If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. (3) If the LCS recovery is high but the sample results are <QL, narrate. Otherwise, re-extract blank and affected sample batch.	Analyst/ Supervisor	Accuracy/bias	Statistically derived acceptance limits.

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

**QC Samples Table**

Matrix	Aqueous field quality control sample
Analytical Group	Select Pesticides
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8081A\SOP-2
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogates	2 per Sample	Statistically derived acceptance limits.	(1) No CA will be taken when one surrogate is within criteria. (2) If surrogates are outside high and sample is <QL no CA taken. (3) If surrogates are outside low the affected samples are re-extracted and reanalyzed.	Analyst/ Supervisor	Accuracy/bias	Statistically derived acceptance limits.

CA – Corrective Action

EICP – Extracted Ion Current Profile

PQL – Practical Quantitation Limit

QA – Quality Assurance

RPD – Relative Percent Difference

SRM – Standard Reference Material

TBD – To Be Determined

TCL – Target Compound List

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland -- Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	TCL PCBs
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8082\SOP-3
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch of twenty or fewer samples of similar matrix	No analyte detected >QL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the QL, report sample results which are <QL or > 10x the blank concentration. Otherwise, reprep a blank and samples >QL and <10x QL.	(1) Analyst/ Supervisor (2) Supervisor (3) Analyst	Accuracy/bias, Contamination	No target > QL

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	TCL PCBs
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8082\SOP-3
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Reagent Blank	1 per Lot	No analyte detected $\geq$ QL	(1) Investigate source of contamination (2) If required replace Lot	Analyst/ Supervisor	Accuracy/bias-Contamination	No target $\geq$ QL

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	TCL PCBs
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8082/SOP-3
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike	One per prep batch of twenty or fewer samples of similar matrix	Statistically derived limits	(1) No CA will be taken for Samples where recoveries are outside limits and surrogate and LCS criteria are met. (2) Where LCS is low outside criteria, a LCS, MS (sample <2.5 ug/L), and MSD may be re-extracted if Project Completeness < Completeness Criteria	Analyst/ Supervisor	Accuracy/bias	Statistically derived limits

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment					
Analytical Group	TCL PCBs					
Concentration Level	Low					
Sampling SOP	SA-1.2 and SA-1.3					
Analytical Method/SOP Reference	SW-846 8082\SOP-3					
Sampler's Name	TBD					
Field Sampling Organization	TtNUS					
Analytical Organization	Katahdin Analytical Services					
No. of Sample Locations	20					
QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicate	One per prep batch of twenty or fewer samples of similar matrix	≤ 50% RPD	(1) In the event that the MS or MSD surrogates are outside criteria, the MS/MSD will be re-extracted and reanalyzed else no CA will be taken when both MS and MSD surrogates are in criteria	Analyst/Supervisor	Accuracy/bias & Precision	Statistically derived acceptance limits, Precision RPD ≤ 50%

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	TCL PCBs
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8082\SOP-3
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	One per prep batch of twenty or fewer samples of similar matrix	Statistically derived acceptance limits.	(3) If an MS/MSD was performed and acceptable, narrate. (2) If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. (3) If the LCS recovery is high but the sample results are <QL, narrate. Otherwise, re-extract blank and affected sample batch.	Analyst/ Supervisor	Accuracy/bias	Statistically derived acceptance limits.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	TCL PCBs
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8082\SOP-3
Sampler's Name	TBD
Field Sampling Organization	TINUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogates	2 per Sample	Statistically derived acceptance limits.	(1) No CA will be taken when one surrogate is within criteria. (2) If surrogates are outside high and sample is <QL no CA taken. (3) If surrogates are outside low the affected samples are re-extracted and reanalyzed.	Analyst/ Supervisor	Accuracy/bias	Statistically derived acceptance limits.

CA – Corrective Action

EICP – Extracted Ion Current Profile

PCBs – Polychlorinated Biphenyls

PQL – Practical Quantitation Limit

QA – Quality Assurance

RPD – Relative Percent Difference

SRM – Standard Reference Material

TBD – To Be Determined

TCL – Target Compound List

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control samples
Analytical Group	TCL PCBs
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8082\SOP-3
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per prep batch of twenty or fewer samples of similar matrix	No analyte detected >QL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e., if the blank results are above the QL, report sample results which are <QL or > 10x the blank concentration. Otherwise, reprep a blank and samples >QL and <10x QL.	(1) Analyst/Supervisor (2) Supervisor (3) Analyst	Accuracy/bias, Contamination	No target > QL

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control samples
Analytical Group	TCL PCBs
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8082\SOP-3
Sampler's Name	TBD
Field Sampling Organization	TiNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Reagent Blank	1 per Lot	No analyte detected $\geq$ QL	(1) Investigate source of contamination (2) If required replace Lot	Analyst/ Supervisor	Accuracy/bias, Contamination	No target $\geq$ QL

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control samples					
Analytical Group	TCL PCBs					
Concentration Level	Low					
Sampling SOP	SA-1.2 and SA-1.3					
Analytical Method/SOP Reference	SW-846 8082\SOP-3					
Sampler's Name	TBD					
Field Sampling Organization	TtNUS					
Analytical Organization	Katahdin Analytical Services					
No. of Sample Locations	2 blanks					
QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike	One per prep batch of twenty or fewer samples of similar matrix	Statistically derived limits	(1) No CA will be taken for Samples where recoveries are outside limits and surrogate and LCS criteria are met. (2) Where LCS is low outside criteria, a LCS, MS (sample <2.5 ug/L), and MSD may be re-extracted if Project Completeness < Completeness Criteria	Analyst/ Supervisor	Accuracy/bias	Statistically derived limits

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control samples
Analytical Group	TCL PCBs
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8082\SOP-3
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Duplicate	One per prep batch of twenty or fewer samples of similar matrix	≤50% RPD	(1) In the event that the MS or MSD surrogates are outside criteria, the MS/MSD will be re-extracted and reanalyzed else no CA will be taken when both MS and MSD surrogates are in criteria	Analyst/ Supervisor	Accuracy/bias and Precision	Statistically derived acceptance limits, Precision RPD ≤50%



**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control samples
Analytical Group	TCL PCBs
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8082\SOP-3
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS	One per prep batch of twenty or fewer samples of similar matrix	Statistically derived acceptance limits.	(4) If an MS/MSD was performed and acceptable, narrate. (2) If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. (3) If the LCS recovery is high but the sample results are <QL, narrate. Otherwise, re-extract blank and affected sample batch.	Analyst/ Supervisor	Accuracy/bias	Statistically derived acceptance limits.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control samples
Analytical Group	TCL PCBs
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 8082\SOP-3
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogates	2 per Sample	Statistically derived acceptance limits.	(1) No CA will be taken when one surrogate is within criteria. (2) If surrogates are outside high and sample is <QL no CA taken. (3) If surrogates are outside low the affected samples are re-extracted and reanalyzed.	Analyst/ Supervisor	Accuracy/bias	Statistically derived acceptance limits.

CA – Corrective Action  
 EICP – Extracted Ion Current Profile  
 PCBs – Polychlorinated Biphenyls

PQL – Practical Quantitation Limit  
 QA – Quality Assurance  
 RPD – Relative Percent Difference

SRM – Standard Reference Material  
 TBD – To Be Determined  
 TCL – Target Compound List

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control samples
Analytical Group	Mercury
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 7470A/SOP-14
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations.	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Independent Calibration Verification (ICV)	Immediately after calibration	± 10% of true value	Correct problem, recalibrate and reanalyze ICV	Analyst/Supervisor, QA Manager	Accuracy/bias	± 10% of true value
Initial Calibration Blank (ICB)	Immediately after the ICV	≤PQL	Correct problem, recalibrate and reanalyze ICV and ICB	Analyst/Supervisor, QA Manager	Accuracy/bias, Contamination	≤PQL
PQL Standard for ICP (PQL)	At the beginning of a sample run, after every 20 samples and at the end of the run	Recovery within 50% - 150 % of true value.	1. Reanalyze immediately for failing elements only. 2. Terminate analysis, correct problem, recalibrate and reanalyze all analytical samples analyzed since last good PQL Std.	Analyst/Supervisor, QA Manager	Sensitivity	Recovery within 50% - 150% of true value.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control samples
Analytical Group	Mercury
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 7470A/SOP-14
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Preparation Blank (PBW)	One per prep batch of twenty or fewer samples of similar matrix	Absolute value < PQL.	1. If blank value > PQL report sample results if < PQL or > 10 x the blank value; otherwise redigest. 2. If blank value is less than negative PQL, report sample results if > 10x the absolute value of the blank result, otherwise redigest.	Analyst/Supervisor, QA Manager	Accuracy/bias	Absolute value < PQL. Sample results if > 10x the absolute value of the blank result, otherwise redigest.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Aqueous field quality control samples
Analytical Group	Mercury
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 7470A/SOP-14
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Serial Dilution (L)	One per prep batch of twenty or fewer samples of similar matrix	If original sample result is at least 50x IDL, 5-fold dilution must agree within $\pm 10\%$ of the original result.	Flag results for affected analytes for all associated samples with "E".	Analyst/Supervisor, QA Manager	Accuracy/Bias, Precision	If original sample result is at least 50x IDL, 5-fold dilution must agree within $\pm 10\%$ of the original result.
Laboratory Control Sample (LCSW)	One per prep batch of twenty or fewer samples of similar matrix	Recovery within $\pm 20\%$ of true value.	Redigest and reanalyze all associated samples for affected analyte (except Ag and Sb)	Analyst/Supervisor, QA Manager	Accuracy/Bias	$\pm 20\%$

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

**QC Samples Table**

Matrix	Aqueous field quality control samples
Analytical Group	Mercury
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 7470A/SOP-14
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	2 blanks

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike (S)	One per prep batch of twenty or fewer samples of similar matrix	Recovery $\pm$ 25 % of true value if sample < 4x spike value	Flag results for affected analytes for all associated samples with "N",	Analyst/Supervisor, QA Manager	Accuracy/bias	Recovery $\pm$ 25 % of true value if sample < 4x spike value
Matrix Spike Duplicate (MSD)	One per prep batch of twenty or fewer samples of similar matrix	RPD $\pm$ 20%	Flag results for affected analytes for all associated samples with "**".	Analyst/Supervisor, QA Manager	Accuracy/Bias, Precision	RPD $\pm$ 20%

CA – Corrective Action  
 EICP – Extracted Ion Current Profile  
 PQL – Practical Quantitation Limit

QA – Quality Assurance  
 RPD – Relative Percent Difference

SRM – Standard Reference Material  
 TBD – To Be Determined

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	Mercury
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 7471A/SOP-13
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Independent Calibration Verification (ICV)	Immediately after calibration	± 10% of true value	Correct problem, recalibrate and reanalyze ICV	Analyst/Supervisor, QA Manager	Accuracy/bias	± 10% of true value
Initial Calibration Blank (ICB)	Immediately after the ICV	≤PQL	Correct problem, recalibrate and reanalyze ICV and ICB	Analyst/Supervisor, QA Manager	Accuracy/bias, Contamination	≤PQL
PQL Standard for ICP (PQL)	At the beginning of a sample run, after every 20 samples and at the end of the run	Recovery within 50% - 150% of true value.	1. Reanalyze immediately for failing elements only. 2. Terminate analysis, correct problem, recalibrate and reanalyze all analytical samples analyzed since last good PQL Std.	Analyst/Supervisor, QA Manager	Sensitivity	Recovery within 50% - 150% of true value.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	Mercury
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 7471A/SOP-13
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performanc Criteria
Preparation Blank (PBS)	One per prep batch of twenty or fewer samples of similar matrix	Absolute value < PQL.	1. If blank value > PQL report sample results if < PQL or > 10 x the blank value; otherwise redigest. 2. If blank value is less than negative PQL, report sample results if > 10x the absolute value of the blank result, otherwise redigest.	Analyst/Supervisor, QA Manager	Accuracy/bias	Absolute value < PQL. Sample results if > 10x the absolute value of the blank result, otherwise redigest.



**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	Mercury
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 7471A/SOP-13
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Serial Dilution (L)	One per prep batch of twenty or fewer samples of similar matrix	If original sample result is at least 50x IDL, 5-fold dilution must agree within $\pm 10\%$ of the original result.	Flag results for affected analytes for all associated samples with "E".	Analyst/Supervisor, QA Manager	Accuracy/Bias, Precision	If original sample result is at least 50x IDL, 5-fold dilution must agree within $\pm 10\%$ of the original result.
Laboratory Control Sample (LCS)	One per prep batch of twenty or fewer samples of similar matrix	Recovery within reference limits supplied by SRM vendor.	Redigest and reanalyze all associated samples for affected analyte (except Ag and Sb)	Analyst/Supervisor, QA Manager	Accuracy/Bias	Recovery within reference limits supplied by SRM vendor.

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	Mercury
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	SW-846 7471A/SOP-13
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike Sample (MS)	One per prep batch of twenty or fewer samples of similar matrix	Recovery $\pm$ 25 % of true value if sample < 4x spike value	Flag results for affected analytes for all associated samples with "N",	Analyst/Supervisor, QA Manager	Accuracy/bias	Recovery $\pm$ 25 % of true value if sample < 4x spike value
Matrix Spike Sample Duplicate (MSD)	One per prep batch of twenty or fewer samples of similar matrix	RPD $\leq$ 20%	Flag results for affected analytes for all associated samples with "**".	Analyst/Supervisor, QA Manager	Accuracy/Bias, Precision	RPD $\leq$ 20%

CA – Corrective Action  
 EICP – Extracted Ion Current Profile  
 PQL – Practical Quantitation Limit  
 QA – Quality Assurance

RPD – Relative Percent Difference  
 SRM – Standard Reference Material  
 TBD – To Be Determined

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QC Samples Table**

Matrix	Sediment
Analytical Group	pH
Concentration Level	Low
Sampling SOP	SA-1.2 and SA-1.3
Analytical Method/SOP Reference	pH/SOP-15
Sampler's Name	TBD
Field Sampling Organization	TtNUS
Analytical Organization	Katahdin Analytical Services
No. of Sample Locations	20

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample(LCS)	One per 20 samples	90-110% recovery	Correct problem, recalibrate	Analyst, Supervisor, QA Manager	Accuracy/Bias	90 - 110% recovery
Sample duplicate	One sample duplicate per every 10 field samples.	RPD < 20%	1) Investigate problem and reanalyze sample in duplicate (2) If RPD is still unacceptable, report original result with notation or narration.	Analyst, Supervisor, QA Manager	Precision	RPD < 20%

CA – Corrective Action  
 QA – Quality Assurance  
 RPD – Relative Percent Difference  
 TBD – To Be Determined

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QAPP\_Worksheet\_#29**

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 -Sampling Notes and Drilling Logs -Photographs -Field Task Modification Forms -This QAPP -Health and Safety Plan	-Field Sample Data -Field Logbook -Field Sample Forms -Sampling Instrument Calibration Logs -Field Sample Data -Chain of Custody record/Form -Field Task Modification Form	-Sample receipt, custody, and tracking record -Standards traceability 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 -Sample storage and disposal records -Telephone logs -Extraction/clean-up records -Raw data (stored electronically)	-Field Sampling Audit Checklist -Analytical Audit Checklist -Data Review Reports -Laboratory QA Plan -Tabulated Data Summary Forms -Data Validation Memoranda -Performance Monitoring Report	

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QAPP\_Worksheet\_#30**

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	Concentration Level	Sample Location/ID Numbers	Analytical SOP	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)
Sediment	PAHs	Low	See worksheet 18	SW-846 3540, 3550, 8270C SIM/SOP-6, SOP-9, SOP-1	Requested: 21 day Actual:	Andrea Colby Katahdin Analytical Services 600 Technology Way Scarborough, ME 04074 Telephone: (207) 874-2400 Fax: (207) 775-4029	Leslie Diamond
Sediment	Select Pesticides	Low	See worksheet 18	SW-846 3540, 3550, 8081A/ SOP-4, SOP-8, SOP-2	Requested: 21 day Actual:	Andrea Colby Katahdin Analytical Services 600 Technology Way Scarborough, ME 04074 Telephone: (207) 874-2400 Fax: (207) 775-4029	Leslie Diamond
Sediment	TCL PCBs	Low	See worksheet 18	SW-846 3540, 3550, 8082/SOP-4, SOP-5, SOP-3	Requested: 21 day Actual:	Andrea Colby Katahdin Analytical Services 600 Technology Way Scarborough, ME 04074 Telephone: (207) 874-2400 Fax: (207) 775-4029	Leslie Diamond
Sediment	TAL Metals	Low	See worksheet 18	SW-846 3050, 6010/SOP-11, SOP-12, SOP-13	Requested: 21 day Actual:	Andrea Colby Katahdin Analytical Services 600 Technology Way Scarborough, ME 04074 Telephone: (207) 874-2400 Fax: (207) 775-4029	Leslie Diamond
Sediment	TOC	Low	See worksheet 18	Lloyd Kahn/SOP-16	Requested: 21 day Actual:	Andrea Colby Katahdin Analytical Services 600 Technology Way Scarborough, ME 04074 Telephone: (207) 874-2400 Fax: (207) 775-4029	Leslie Diamond

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**Analytical Services Table**

Matrix	Analytical Group	Concentration Level	Sample Location/ID Numbers	Analytical SOP	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)
Sediment	pH	Low	See worksheet 18	pH/SOP-17	Requested: 21 day Actual:	Andrea Colby Katahdin Analytical Services 600 Technology Way Scarborough, ME 04074 Telephone: (207) 874-2400 Fax: (207) 775-4029	Leslie Diamond
Aqueous field quality control samples	PAHs	Low	See worksheet 18	SW846 3510, 3520, 8270C SIM/SOP-5, SOP-1	Requested: 21 day Actual:	Andrea Colby Katahdin Analytical Services 600 Technology Way Scarborough, ME 04074 Telephone: (207) 874-2400 Fax: (207) 775-4029	Leslie Diamond
Aqueous field quality control samples	Select Pesticides	Low	See worksheet 18	SW-846 3510, 3520, 8081A/SOP-7, SOP-2	Requested: 21 day Actual:	Andrea Colby Katahdin Analytical Services 600 Technology Way Scarborough, ME 04074 Telephone: (207) 874-2400 Fax: (207) 775-4029	Leslie Diamond
Aqueous field quality control samples	TCL PCBs	Low	See worksheet 18	SW8-46 3510, 3520, 8082/SOP-7, SOP-3	Requested: 21 day Actual:	Andrea Colby Katahdin Analytical Services 600 Technology Way Scarborough, ME 04074 Telephone: (207) 874-2400 Fax: (207) 775-4029	Leslie Diamond
Aqueous field quality control samples	TAL Metals	Low	See worksheet 18	SW-846 3010, 6010B/SOP-10, SOP-12, SOP-14	Requested: 21 day Actual:	Andrea Colby Katahdin Analytical Services 600 Technology Way Scarborough, ME 04074 Telephone: (207) 874-2400 Fax: (207) 775-4029	Leslie Diamond

NA – Not Applicable  
 SIM – Selective Ion Monitoring  
 TAL – Target Analyte List  
 TCL – Target Compound List  
 TOC – Total Organic Carbon

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON

Site Location: Groton, Connecticut

Revision Number: 0

Revision Date: October 2007

**QAPP Worksheet #31**

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)
Health and Safety	1 per contract year	Int.	TtNUS	TBD	PM	Auditor and Health and Safety Manager	Health and Safety Manager Matt Soltis
Laboratory Systems Audit	Every 18 months	Ext.	NFESC	TBD	Laboratory QA Manager	Laboratory QA Manager	Laboratory QA Manager
Field Sampling Systems Audit	1 per contract year	Int.	TtNUS	TBD	PM	Auditor and QA Manager (Kelly Carper TtNUS)	QA Manager (Kelly Carper TtNUS)

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**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
Health and Safety Audit	Audit checklist and written audit finding summary	Project Manager TtNUS, Field Operations Leader TtNUS, and Program Manager TtNUS	Dependant on findings, if major a stop work maybe issued immediately, however if minor within 1 week of audit	Written memo	Health and Safety Manager TtNUS, Auditor TtNUS, Program Manager TtNUS	Within 48 hours of notification
Field sampling system audit	Audit checklist and written audit finding summary	Project Manager TtNUS, Field Operations Leader TtNUS, and Program Manager TtNUS	Dependant on findings, if major a stop work maybe issued immediately, however if minor within 1 week of audit	Written memo	Quality Assurance Manager TtNUS, Auditor TtNUS, Program Manager TtNUS	Within 48 hours of notification
Laboratory systems Audit	Written audit report	Laboratory QA Manager	Not specified by NFESC	Letter	NFESC	Specified by NFESC

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QAPP Worksheet #33**

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	Data Validation Manager or designee	Data Validation Manager or designee	PM (TtNUS), project file
Major analysis problem identification (Internal Memorandum)	When persistent analysis problems are detected	Immediately	QA Manager (TtNUS)	PM (TtNUS), QAM (TtNUS), Program Manager (TtNUS), project file
Project monthly progress report	Monthly for duration of the project	Monthly	PM (TtNUS)	Navy, project file
Field progress reports	Daily, oral, during the course of sampling	Every day that field sampling occurs	FOL (TtNUS)	PM (TtNUS)
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately	Subcontracted laboratories	TtNUS, project file

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
Site Location: Groton, Connecticut

Revision Number: 0  
Revision Date: October 2007

**QAPP\_Worksheet\_#34**

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	Int.	FOL or designee TtNUS
Chain of custody	Chain of custody records will be reviewed internally by the Project Manager or designee and compared against sample tables listing the proposed samples to verify that all planned samples have been collected.	Int.	PM 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)	Int.	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.	Int.	Data validator TtNUS
Sample log sheets	Log sheets completed as samples are collected in the field are verified for completeness and are maintained at the project office.	Int.	PM or designee TtNUS

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
Site Location: Groton, Connecticut

Revision Number: 0  
Revision Date: October 2007

**QAPP\_Worksheet\_#35**

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 data base	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

**Project-Specific QAPP**

Title: QAPP for Phase III Investigation for Area A Wetland – Site 2B

Site Name/Project Name: NSB - NLON  
 Site Location: Groton, Connecticut

Revision Number: 0  
 Revision Date: October 2007

**QAPP\_Worksheet\_#36**

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	Sediment/ Aqueous field quality control samples	PAHs	Low	SW-846, Region I Part 2 Volatile/semivolatile Data Validation Functional Guidelines (12/96), DOD QSM.	TtNUS Staff Chemist
IIa and IIb	Sediment/ Aqueous field quality control samples	Pesticides	Low	SW-846, Region I Part 2 Volatile/semivolatile Data Validation Functional Guidelines (12/96), DOD QSM.	TtNUS Staff Chemist
IIa and IIb	Sediment/ Aqueous field quality control samples	PCBs	Low	SW-846, Region I Part 2 Volatile/semivolatile Data Validation Functional Guidelines (12/96), DOD QSM.	TtNUS Staff Chemist
IIa and IIb	Sediment/ Aqueous field quality control samples	Metals	Low	SW-846, Region I Inorganic Data Validation Functional Guidelines (2/89), DOD QSM.	TtNUS Staff Chemist
IIa and IIb	Sediment	TOC and pH	Low	Method-specific criteria, Region I Inorganic Data Validation Functional Guidelines (2/89) to extent practicable, DOD QSM.	TtNUS Staff Chemist

### **QAPP Worksheet #37**

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 on the use of the data will be handled.

#### **Usability Assessment**

**Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:**

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 contaminant concentration detected at that sample location.

If the data sets support statistical interpretations, 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.

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 sediment 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 PM. 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.

As part of the data usability evaluations, the precision, accuracy, representativeness, completeness, comparability, and sensitivity of the data will be evaluated. This will generally require tabulations or summaries of the quantitative parameters (precision, accuracy, completeness, and sensitivity), and qualitative discussions of the others. The tabulations or summaries will be quantitative in nature showing statistics such as minimum or maximum values, average values, standard deviations, etc. as deemed by the TtNUS project manager as useful in understanding the data quality and usability for supporting project objectives. If important data values are rejected during data validation or other data assessment processes, additional data collection may be required.

The data quality will be reconciled with MPCs to determine whether sufficient data of acceptable quality are available for decision making. A series of inspections and statistical analyses will be performed to estimate several of the data set characteristics. The statistical evaluations will include simple summary statistics for target analytes, such as the maximum concentration, minimum concentration, number of samples

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

exhibiting no detectable analyte, the number of samples exhibiting detectable analytes, and the proportion of samples with detectable and undetectable analytes.

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

Field data will be reviewed while in the field for reasonableness based on an assessment of the quantities being measured and the conditions under which the measurements are made. This process will rely heavily on the judgment of the Field Operations Leader with support from the PM as necessary. Laboratory data accuracy will be evaluated by reviewing accuracy indicators such as matrix spike recoveries, laboratory control sample recoveries, calibration performance, and blank contamination. Precision indicators will be used to assess the uncertainty of the data. This will, at a minimum, involve a comparison of laboratory and field precision to determine whether there could be any unusual level of uncertainty in the data. Sensitivity will be evaluated by reviewing the minimum and maximum non-detect values for each parameter for each environmental matrix analyzed. Completeness will be evaluated as described in Section 1.5.2.5. Comparability and representativeness will be evaluated by reviewing planned sample collection and analysis specifications against what actually occurred. Significant deviations from the QAPP will be noted. 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.

**Identify the personnel responsible for performing the usability assessment:**

One hundred percent of the Phase III laboratory chemical analysis data will be validated. Validation of analytical data will be conducted by TtNUS. Final review and approval of validation deliverables will be completed by the DVM. PAHs, pesticides, PCBs, and metals will be validated according to the requirements of the EPA Region I National Functional Guidelines for Organic and Inorganic Data Review (USEPA, 1996 and 1989).

**Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:**

The data will be presented in a tabular format. The usability assessment is 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 using statistics, as applicable.

**Project-Specific QAPP**

**Title:** QAPP for Phase III Investigation for Area A Wetland – Site 2B

**Site Name/Project Name:** NSB - NLON

**Site Location:** Groton, Connecticut

**Revision Number:** 0

**Revision Date:** October 2007

- Identify elevated detection limits and explain their impacts on the attainment of project objectives.
- Identify unusable data (i.e., data qualified as “UR” and “R”).
- Evaluate project assumptions against actual conditions, when possible.
- Characterize data set distributions (e.g., Shapiro-Wilk W test) if enough data are available and if these characterizations will enhance an understanding of the data.
- 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.

**APPENDIX C**  
**ECOLOGICAL RISK ASSESSMENT**

## ECOLOGICAL RISK ASSESSMENT

### 1.0 INTRODUCTION

This Screening Level Ecological Risk Assessment (SERA) evaluates whether adverse ecological impacts are present as a result of exposure to chemicals released to the environment through historical activities at the Area A Wetland at Naval Submarine Base, New London (NSB-NLON), Groton, Connecticut. A SERA was previously conducted for the Area A Wetland as part of the Phase II Remedial Investigation Report for NSB-NLON (Brown and Root Environmental, 1997). The ERA concluded that chemicals in the surface water, sediment, and surface soil of the Area A Wetland represent a potential risk to both aquatic and terrestrial receptors. Before proceeding further in the ERA process, the Navy determined that the SERA should first be updated using current methodologies and toxicity data because the risk assessment is more than ten years old. It is anticipated that this process will be phased with Phase III consisting of collecting additional media data (i.e., soil, sediment) for chemical analysis. Phase IV, if necessary, could consist of collecting additional abiotic media data for chemical analysis as well as toxicity/bioaccumulation testing.

The results of this updated SERA were used to determine additional data needs in the Phase III Remedial Investigation (RI) Quality Assurance Project Plan (QAPP) for the Area A Wetland. The results of the Phase III data, in combination with the existing data, will be used to plan the Phase IV investigation, if necessary.

The ERA methodology follows guidance presented in the following documents:

- Final Guidelines for Ecological Risk Assessment [United States Environmental Protection Agency (USEPA), 1998].
- Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Ecological Risk Assessments (USEPA, 1997).
- Navy Policy for Conducting Ecological Risk Assessments [Department of Navy (DON), 1999].

These documents are more current than the ones used as guidance for the SERA in the Phase II RI. Also, toxicity data, screening levels, bioaccumulation factors, and general methodologies have changed since the SERA in the Phase II RI was finalized.

The ERA process consists of the following eight steps that are required by the above documents for any ecological risk assessment:

- Step 1 – Screening-Level Problem Formulation and Ecological Effects Evaluation
- Step 2 – Screening-Level Exposure Estimate and Risk calculation
- Step 3 – Baseline Risk Assessment Problem Formulation
- Step 4 – Study Design and Data Quality Objective Process
- Step 5 – Field Verification of Sampling design
- Step 6 – Site Investigation and Analysis Phase
- Step 7 – Risk Characterization
- Step 8 – Risk Management

This updated SERA for the Area A Wetland consists of Steps 1 and 2, along with the first part of Step 3, which is termed Step 3a. Step 3a considers factors other than comparisons of chemical concentrations to screening levels to further refine the list of COPCs (see Section 6.0 for more details). The remaining steps (the rest of Step 3 through Step 7) are conducted only if additional evaluations or investigations are necessary; these steps were not conducted as part of this report. A decision to proceed to a Baseline ERA (BERA) is usually only made after the results of the SERA are evaluated.

In summary, there are two primary objectives of this updated ERA:

- Use the refined list of COPCs from Step 3a to determine the analytical parameter list for the Phase III RI investigation.
- Present the methodologies that will be used to update the ERA after the Phase III data are collected.

## 2.0 PROBLEM FORMULATION

Problem formulation is the first part of the SERA and discusses the goals, breadth, and focus of the assessment. It includes general descriptions of the Area A Wetland with emphasis on the habitats and ecological receptors present. This phase also involves characterization of site-related chemicals, chemical sources, migration routes, and an evaluation of routes of chemical exposure.

### 2.1 Environmental Setting

The Area A Wetland is adjacent to the northeast edge of the Area A Landfill and is approximately 25.76 acres in size. The location of the Area A Wetland within NSB-NLON is shown on Figure 1-1. Figure 1-2 shows the surface features of the Area A Wetland. The Area A Wetland is a relatively flat lying, swampy, vegetated area. In general, the surface elevation of the wetland is between 70 and 80 feet. This portion of NSB-NLON was undeveloped, wooded land, and possibly wetland, until the late 1950s.

The Area A Wetland is characterized as a palustrine emergent, nonpersistent, narrow-leaved and broad-leaved deciduous scrub/shrub wetland with a non-tidal artificial water regime. Areas of open water (generally shallow) are scattered across this wetland unit. The soft organic sediments that characterize these wetlands support a monoculture of the reed *Phragmites communis*, which dominates all other vegetative forms. While providing good cover, no species of wildlife is known to utilize this emergent as a source of food. There are scattered patches of open water between the stands of reeds; scattered duckweed (*Lemna spp.*) and filamentous algae found in these areas. As the substrates become firmer, the vegetation becomes more typical of vegetation associated with old fields and upland areas. Vegetation species found in the zone of transition include viburnum (*Viburnum recognitum*), spicebush (*Lindera benzoin*), and black alder (*Ilex verticillata*) (Atlantic, 1994c). The areas near the Area A Wetland provide good habitat for ecological receptors that may use the wetlands as a source of drinking water. In addition, the open water areas are known to be utilized by aquatic birds, amphibians, and aquatic insects; amphibians and aquatic insects represent potential prey for wildlife that could forage in this area.

There is a small pond located at the southern portion of the wetland and between one and three feet of standing water is present during all seasons. *Phragmites* is the predominant type of vegetation. Water from the wetland discharges through an earthen dike at the western edge of the wetland via four 24-inch metal culverts to the Area A Downstream Watercourses. These

watercourses subsequently discharge into the Thames River. Several shallow intermittent drainage channels cross the wetland.

The surface elevation increases to nearly 100 feet in the northeast corner of the wetland. This area was historically a stream valley. After the earthen dike was constructed and the ground surface of the wetland was raised by filling with dredge spoil, groundwater levels rose to the point such that the dredge spoil placed in the northeast corner became saturated.

The most prominent topographic feature is a bedrock knob, located between the Area A Weapons Center and the Area A Landfill. Bedrock was within 1 foot of the ground surface at this location. Additionally, the local bedrock knob is confirmed by the historical surficial geology map, which predates the construction of the earthen dike and filling activities.

The SCS Soils Map (SCS, 1983) classifies the soil at the Area A Wetland as Udorthents-Urban land. This soil type is defined as excessively drained to moderately drained soils that have been disturbed by cutting and filling. This is consistent with historical information regarding the placement of dredge spoils in the area. The surface of the Area A Wetland is covered with a 2-foot layer of roots and plant debris derived from *Phragmites*, the predominant vegetation.

The Area A Wetland is underlain by dredge spoils that consist of silt and clay with traces of fine sand and shell fragments. The makeup of the dredge spoils reflects their original depositional environment, i.e., river bottom sediments. The dredge spoils extend across the present site southeast to 2WMW3 (near the tennis courts) and southwest beneath the Area A Landfill. Dredge spoils are between 25 and 35 feet thick on the south side of the wetland adjacent to the landfill, and 10 to 15 feet thick on the northeast side of the wetland. Where dredge spoil does not lie directly on bedrock, it is underlain by a thin remnant of topsoil, which consists of dark, organic-rich silt, clay, and traces of roots. The topsoil is in turn underlain by alluvial deposits.

Groundwater is present within the overburden and bedrock underlying the Area A Wetland. As is typical for wetland environments, the water table is nearly at the ground surface throughout most of the Area A Wetland. Groundwater flows from higher elevations toward the bedrock valley and ultimately travels to the Area A Downstream Watercourses through a combination of discharge to local streams within the wetland and aquifer underflow. The hydraulic gradient is relatively flat across the Area A Wetland.

## 2.2 Potential Sources of Contamination

In the late 1950s, dredge spoils from the Thames River were pumped to the A Wetland area and contained within an earthen dike that extends from the Area A Landfill to the south side of the Area A Weapons Center. Based on the boring logs, the total volume of dredged material in the wetlands is approximately 1.2 million cubic yards.

It was also reported that pesticide "bricks" were placed on the wetland ice during winter and allowed to dissolve as a mosquito control measure. These "bricks" consisted of formulated (water-soluble) DDT and were used in the 1960s, prior to the 1972 ban on DDT.

Other sources of contamination to the Area A Wetland include runoff from the Area A Landfill prior to it being capped and stormwater discharges from the Area A Weapons Center.

## 2.3 Previous Investigations

Section 1.4.2.1 in the main portion of the QAPP presents a detailed discussion of the previous investigations conducted at the sites, as well as investigations conducted at adjacent sites (Area A Landfill and Area A Weapons Center). Sample locations from these previous investigations are depicted on Figures 1-2 and 1-3. Attachment 1 presents figures for selected chemical (total PAHs, total DDTs, Aroclor-1260, cadmium, copper, lead, mercury, nickel, and zinc). For the chemical parameters, the sample locations are shaded green if the concentrations in sediment or soil are less than the sediment screening values used in the ERA, they are shaded red if the concentrations are greater than the Probable Effects Concentrations (PECs) from MacDonald et al., (2000), and are shaded yellow if the concentration is between those values. If a chemical was not detected in the sample, one-half of the detection limit was used except for total PAHs and total DDTs which used positive detections only. The figures also indicate which sample results are based on non-detections. Note that the concentrations in the soil samples were compared to the sediment screening levels in this figure because the soil may be covered with water during some periods and/or the soil may erode into the sediment. The soil samples were evaluated separately from the sediment in this ERA, however. One figure also depicts total organic carbon (TOC) concentrations in the sediment samples which are used to evaluate the potential bioavailability of the chemicals. The following bullets describe how the data from previous investigations was used in this ERA:

### **Phase I Remedial Investigation (RI)**

- Surface soil samples were used to quantify risks to terrestrial plants, invertebrates, and wildlife.
- Sediment samples were used to quantify risks to sediment invertebrates and wildlife.
- Surface water samples were used to quantify risks to aquatic receptors and wildlife.
- Frog tissue samples were discussed qualitatively in the risk characterization section.
- Bird tissue samples were not evaluated in the ERA.

### **Phase II RI**

- Sediment samples were used to quantify risks to sediment invertebrates and wildlife.
- Surface water samples were used to quantify risks to aquatic receptors and wildlife.

### **Focused Feasibility Study**

- Sediment samples were used to quantify risks to sediment invertebrates and wildlife.

### **Additional Soil Samples**

- The additional surface soil samples that were collected during the installation of monitoring wells at the Area A landfill were used to quantify risks to terrestrial plants, invertebrates, and wildlife.

### **Area A Landfill Groundwater Monitoring Program**

- Appendix D presents a summary of the surface water portion of the Area A Landfill groundwater monitoring program. The surface water analytical data from Rounds 14 through 17 are evaluated for potential risks to aquatic receptors. These rounds were selected because they are the four most recent rounds for which data are readily available (Rounds 18 and 19 have not yet been loaded into the New London database), so the data represent relatively current conditions. Appendix D also presents a printout of the analytical data for the surface water samples collected as part of the monitoring program for the first 17 rounds. Only chemicals that were positively detected in at least one sample are included in the appendix.

### **Area A Weapons Center Investigations**

- The sediment analytical were used to determine potential sources of chemicals from the Area A Weapons Center to the Area A Wetland and to help define the extent of contamination in the Area A Wetland.

## **2.4 Potential Exposure Pathways**

In general, chemicals released from materials dumped in terrestrial areas can initially contaminate surface soils. Natural precipitation can then cause the chemical contaminants to leach downward into subsurface soils and groundwater. Discharge of the groundwater can result in the contamination of surface water and sediment inhabited by aquatic receptors. The following subsections discuss the potential exposure pathways in more detail.

### **2.4.1 Surface Soil**

Terrestrial ecological receptors such as plants, soil invertebrates, mammals, birds, and reptiles can be exposed to contaminated surface soil through direct contact as they search for food and burrow into the soil. Mammals, birds, and reptiles can also ingest contaminated surface soil and food items in which contaminants have accumulated. Some terrestrial receptors such as burrowing mammals or deep-rooted trees could be exposed to shallow layers of contaminated subsurface soils or to shallow groundwater. Most terrestrial receptors are not substantially exposed to subsurface soils or to groundwater that has not discharged to surface water, so these pathways are not evaluated in this ERA.

### **2.4.2 Sediment**

Aquatic ecological receptors, such as fish, sediment invertebrates, reptiles, and amphibians, can be exposed to sediment contamination through direct contact and incidental sediment ingestion. Terrestrial wildlife may also be exposed to the sediment, although to a lesser degree through direct contact and incidental sediment ingestion. Terrestrial vertebrates, such as piscivorous wildlife, may be exposed to contaminated sediment through ingestion of aquatic prey (i.e., fish).

### **2.4.3 Surface Water**

Aquatic ecological receptors, such as fish, sediment invertebrates, reptiles, and amphibians, can be exposed to surface water contamination through direct contact and surface water ingestion. Terrestrial wildlife may also be exposed to the surface water through direct contact and surface water ingestion.

## 2.5 Endpoints

### 2.5.1 Assessment Endpoints

Assessment endpoints are explicit expressions of the environmental value that is to be protected (USEPA, 1997a). The selection of these endpoints is based on the habitats present, the migration pathways of probable contaminants, and the routes that contaminants may take to enter receptors.

The habitats present at the Area A Wetland consist primarily of a wetland with some forested pockets within the wetland and along the boundaries. There is also a small pond located within the wetlands. Therefore, the assessment endpoints include the protection of the following groups of receptors from adverse effects of contaminants on their growth, survival, and reproduction:

- Carnivorous birds and mammals
  - Soil Invertivorous birds and mammals
  - Sediment Invertivorous birds and mammals
- Herbivorous birds and mammals
- Soil invertebrates
- Sediment invertebrates
- Fish and other aquatic organisms
- Terrestrial vegetation

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

Carnivorous Birds and Mammals - Carnivorous birds and mammals consume invertebrates, fish, mammals and birds. At the Area A Wetland, the focus will be on invertivorous birds and mammals, which are considered first-level carnivores. Piscivorous birds and mammals also are present in the wetland, and well as carnivorous birds and mammals that feed on other birds and mammals, but they are less densely distributed than first-level carnivores because they require a larger area to hunt for their food. Carnivores may be exposed to and accumulate contaminants that are present in the food items they consume.

Herbivorous Birds and Mammals – Herbivorous birds and mammals (animals that consume only plant tissue) forage at the site. Their role in the community is essential because, without them,

higher trophic-level animals could not exist. They may be exposed to and accumulate contaminants that are present in the plants they consume.

Soil invertebrates – Soil invertebrates include earthworms, the juvenile stages of many insects, and other small organisms that directly inhabit the surface soil. These organisms are expected to be present in the soil in terrestrial habitats at the site. Soil invertebrates promote plant growth by aiding in the formation of soil and through redistribution and decomposition of organic matter. Soil invertebrates also serve as a food source for many mammals and birds. Contaminants can bioaccumulate from the soil into the tissues of soil invertebrates used as a food source by mammals and birds.

Sediment Invertebrates - Sediment invertebrates serve as a food source for higher trophic level organisms (i.e., fish, amphibians, birds, mammals). They also can accumulate some contaminants, which can then be transferred to higher trophic level organisms that consume invertebrates. Sediment macroinvertebrates are present in the Area A Wetland.

Fish and Other Aquatic Organisms – Small fish may be present in the Area A Wetland, but based on the relatively small size of the standing water bodies, a large and diverse population is not expected. Fish feed primarily on invertebrates, plants, and/or other fish. Fish are exposed to and can accumulate contaminants from the food items they consume or from the surface water/sediment in which they live.

Terrestrial Vegetation – Terrestrial vegetation at the site consists of herbs (grasses, rushes, ferns, and other non-woody plants), shrubs, woody vines, and trees. These plants serve as a source of food and shelter for many organisms and help prevent soil erosion and excessive surface runoff. Plants can also bioaccumulate some chemical contaminants from the soil that can then be transferred to organisms that feed on the plant tissue.

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

Large carnivorous birds and mammals were not selected as assessment endpoints because their home range is much larger than the site and most of their food would come from other locations.

Therefore, risks would be greater to small mammals and birds that obtain all of the food from the site. Although amphibians and reptiles are likely to be present in and along the streams and pond near the site, they were not selected as assessment endpoints because of the general lack of toxicity information and the lack of methods to evaluate their exposure to contaminants. Finally, aquatic and semi-aquatic plants were not selected as assessment endpoints because there is limited toxicity data to evaluate potential risks to these receptors.

### **2.5.2 Measurement Endpoints**

Measurement endpoints are estimates of biological impacts (e.g., mortality, growth, reproduction) that are used to evaluate the assessment endpoints. The following measures of effects were used to evaluate the assessment endpoints, where appropriate:

- Decreases in survival, reproduction and growth of plants, soil/sediment invertebrates, and aquatic organisms were evaluated by comparing chemical concentrations in surface soil, sediment, and surface water to screening values designed to be protective of ecological receptors.
- Decreases in survival and reproduction of birds and mammals were evaluated by comparing estimated ingested doses of contaminants in surface soil, sediment, surface water, plants, and invertebrates to no observed adverse effects levels (NOAELs) and lowest observed adverse effects levels (LOAELs).

### **2.5.3 Selection of Receptor Species**

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

- Herbivorous mammal: Meadow vole
- Herbivorous bird: Bobwhite quail
- Soil Invertivorous mammal: Short-tailed shrew

- Soil Invertivorous bird: American robin
- Sediment Invertivorous mammal: Raccoon
- Sediment Invertivorous bird: Mallard

Receptor profiles for the above species are presented in Attachment 2.

#### **2.5.4 Conceptual Site Model**

A conceptual site model (CSM) in ERA problem formulation is a written description of predicted relationships between ecological entities and the stressors to which they may be exposed (USEPA, 1998). The CSM consists of two primary components: predicted relationships among stressor, exposure, and assessment endpoint response, and a diagram that illustrates the relationships (USEPA, 1998). At the Area A Wetland, the chemical sources consist of dredge spoils in the subsurface soil, surface runoff from the Area A Landfill and the Area A Weapons Center, and groundwater discharge (via surface water) from the Area A Landfill. Groundwater from the Area A Wetland also discharges to the surface of the Area A Wetland. The exposure media for ecological receptors are the surface soil, sediment, and surface water. Plants, invertebrates, and vertebrates are exposed to the media by direct contact and ingestion of soil, sediment, surface water, and food items. Terrestrial vertebrates may be exposed to chemicals found in the air via inhalation. Although this pathway is possible, it is not a significant pathway and is not evaluated in this ERA.

### **3.0 ECOLOGICAL EFFECTS EVALUATION**

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

#### **3.1 Terrestrial Plants/Invertebrates and Sediment Invertebrates**

Potential risks to terrestrial plants/invertebrates and sediment invertebrates resulting from exposure to chemicals were evaluated by comparing chemical concentrations to ecological screening levels. These toxicity values are expressed in units of concentration because the media of concern are in direct contact with the terrestrial plants/invertebrates and sediment invertebrates.

#### **3.2 Terrestrial Wildlife**

Risk to terrestrial receptors for exposure to chemicals of potential concern (COPCs) in surface soil, sediment, and surface water were determined using food chain models to estimate the Chronic Daily Intake (CDI) and compare the CDI to toxicity reference values (TRVs) representing acceptable daily doses in mg/kg-day. The TRVs were developed from NOAELs and LOAELs obtained from wildlife studies. The majority of the TRVs were obtained from the ORNL Toxicological Benchmarks for Wildlife: 1996 Revision (Sample et al., 1996) and the EPA Eco SSL documents, but were supplemented with other toxicity information when necessary.

Attachment 3 presents the TRVs for the mammals and birds that were used in the ERA and the source of the TRVs. If a subchronic study was used to develop the TRV, the final value was multiplied by a factor of 0.1 to account for uncertainty between subchronic and chronic effects. Also, the LOAEL was multiplied by a factor of 0.1 to estimate a NOAEL TRV if only a LOAEL study was available. The Eco SSL document provided NOAELs and LOAELs for various studies but overall TRVs values were calculated only for NOAELs in the chemical-specific Eco SSL documents, because the Eco SSLs are conservative screening levels. Therefore, the data from the chemical-specific Eco SSL documents were used to calculate overall LOAEL values for several metals as the geometric mean of growth and reproduction data.

#### 4.0 CHARACTERIZATION OF EXPOSURE

This portion of the ERA includes identification of contaminant concentration data used as the exposure point concentrations (EPCs) to represent ecological exposure in various media. Terrestrial plants and invertebrates are exposed to chemicals in the surface soil, and aquatic receptors are exposed to chemicals in the surface water and sediment through ingestion and/or direct contact. Maximum concentrations were used as the EPCs for comparison to the screening values in order to select COPCs.

Total exposure of terrestrial wildlife to COPCs in soil, surface water, and sediment (and associated food items such as plants and invertebrates) were determined for the surrogate wildlife species using the following equation:

$$CDI = \frac{[(C_f * I_f) + (C_s * I_s) + (C_w * I_w)] * H}{BW}$$

Where:

CDI	=	Chronic daily intake (mg/kg-day)
Cf	=	Contaminant concentration in food – (see discussion below)
If	=	Food ingestion rate (kg/day)
Cs	=	Contaminant concentration in surface soil or sediment (mg/kg)
Is	=	Incidental surface soil or sediment ingestion rate (kg/day)
Cw	=	Contaminant concentration in surface water (µg/L)
Iw	=	Incidental surface water ingestion rate (L/day)
H	=	Portion of food intake from the contaminated area (unitless)
BW	=	Body weight (kg)

Chemical concentrations in food items for soil invertivorous and herbivorous receptors were calculated using soil-to-invertebrate or soil-to-plant bioaccumulation factors (BAFs) and regression equations from the EPA Eco SSL Guidance Document (EPA, 2005) or BAFs from published sources. The following equation was used to calculate the chemical concentration in plants or invertebrates when BAFs were used:

$$C_f = C_s * BAF$$

Where:

C <sub>f</sub>	=	Contaminant concentration in food (mg/kg)
C <sub>s</sub>	=	Contaminant concentration in surface soil (mg/kg)
BAF	=	Biota-soil bioaccumulation factor (unitless)

Chemical concentrations in food items for sediment invertivorous receptors were calculated using sediment-to-invertebrate BSAFs from the Biota Sediment Accumulation Factors for Invertebrates: Review and recommendations for the Oak Ridge Reservation (ORNL, 1998) or sediment-to-fish BSAFs from the Incidence and Severity of Sediment Contamination in Surface Waters of the United States, Volume 1: National Sediment Quality Survey: Second Edition (USEPA, November 2004). The fish BSAF were used for the organic chemicals (except PCBs) because sediment invertebrate BSAF were not available for most organic chemicals. Contaminant concentrations in food items for the sediment invertivorous mammals and birds were calculated as follows:

$$C_f \text{ (for metals)} = C_{sd} * BSAF$$

Where:

C <sub>f</sub>	=	Contaminant concentration in food (mg/kg)
C <sub>sd</sub>	=	Contaminant concentration in sediment (mg/kg)
BSAF	=	Biota-sediment bioaccumulation factor (unitless)

$$C_f \text{ (for organics)} = C_{sd} * \left( BSAF * \frac{\%L}{\%TOC} \right)$$

Where:

C <sub>f</sub>	=	Contaminant concentration in food (mg/kg)
C <sub>sd</sub>	=	Contaminant concentration in sediment (mg/kg)
BSAF	=	Biota-sediment bioaccumulation factor (for organics)(unitless)
%L	=	Percent lipids [9.44% (dry weight) for invertebrates (see Attachment 2)]
%TOC	=	Percent total organic carbon (TOC) [4.2% (average TOC for the site)]

Although frog and bird tissue data were collected from the site, the tissue data was not used in the food chain model because the locations of these samples is not known; some of the samples were collected at other sites (i.e., Area A Downstream). However, the tissue data is discussed in the uncertainty analysis section of this SERA. Attachment 4 presents the analytical data for the tissue samples. The exposure assumptions (i.e., ingestion rate, body weight) were obtained primarily from the Wildlife Exposure Factors Handbook (USEPA, 1993) with other sources used

as necessary. Table 5-5 summarizes the exposure factors that were used for the food chain model and Attachment 2 presents the derivation of those parameters. The percent lipids for invertebrates were calculated by averaging the dry-weight percent lipid values for freshwater crustacea, freshwater molluscs, and freshwater worms in from the US Army Engineer research and Development Center, Waterways Experiment Station, Environmental Laboratory (see Attachment 2). The food ingestion rates are on a dry weight basis. The sources of the BAFs and BSAFs are presented in Attachment 2. Contaminants that do not have BAFs or BSAFs were assigned a default value of 1.0.

The following input parameters were used in the dose equation under the conservative screening scenario:

- Maximum surface soil, sediment, and surface water concentrations
- Maximum BAFs and BSAFs
- Conservative receptor body weights and ingestion rate

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

- Average surface soil, sediment, and surface water concentrations
- Average BAFs or BSAFs
- Average receptor body weights and ingestion rates

## 5.0 RISK CHARACTERIZATION

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

The EEQs for surface soil receptors were calculated as follows:

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

where:

- EEQ = Ecological Effects Quotient (unitless)
- $C_{ss}$  = Contaminant concentration in surface soil (ug/kg or mg/kg)
- SSSL = Surface soil screening level (ug/kg or mg/kg)

The EEQs for aquatic receptors were calculated as follows:

$$EEQ = \frac{C_{sw}}{SwSL} \text{ or } \frac{C_{sd}}{SdSL}$$

where:

- EEQ = Ecological Effects Quotient (unitless)
- $C_{sw}$  = Contaminant concentration in surface water (ug/L)
- $C_{sd}$  = Contaminant concentration in sediment (ug/kg or mg/kg)
- SwSL = Surface water screening level (ug/L)
- SdSL = Sediment screening level (ug/kg or mg/kg)

The EEQ for the terrestrial wildlife model was calculated as follows:

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

where:

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

### 5.1 Selection of Contaminants of Potential Concern

The final part of the screening evaluation is selection of COPCs. Chemicals that are not selected as COPCs are assumed to present negligible risk to ecological receptors and are not considered for further evaluation in the ERA. Chemicals that are retained as COPCs are evaluated further in Step 3a to determine if they are carried through as chemicals of concern (COCs). Ecological COPCs were selected using the following procedures:

- Chemicals with EEQs greater than 1.0 (using screening values) were initially selected as COPCs for further evaluation because they have a potential to cause risk to ecological receptors.
- Chemicals with EEQs greater than 1.0 based on the food chain model using NOAELs were initially selected as COPCs because they have the potential to cause risk to higher trophic level mammals and birds.
- Chemicals without screening values were initially selected as COPCs but are only evaluated qualitatively.
- Calcium, magnesium, potassium, and sodium were not retained as COPCs because they are essential nutrients that can be tolerated by living systems even at high concentrations. No evidence indicates that these chemicals are related to site operations, and they are not considered hazardous chemicals.

### 5.1.1 COPCs for Terrestrial Invertebrates and Plants

The data used to select COPCs for terrestrial invertebrates and plants include the surface soil (0-2 feet) samples collected as part of the Phase I RI. Attachment 4 presents the analytical results for chemicals that were detected in at least one surface soil sample. Table 5-1 presents the summary statistics for the surface soil data, along with the selection of the COPCs. In summary, as a result of the screening process:

- Four VOCs were initially selected as COPCs because screening levels are not available for those chemicals.
- Two SVOCs [plus total polyaromatic hydrocarbons (PAHs)] were initially selected as COPCs because their maximum detected concentrations were greater than their respective screening levels. Two SVOCs were initially selected as COPCs because screening levels are not available for those chemicals.
- Two pesticides were initially selected as COPCs because screening levels are not available for those chemicals.
- Nine metals were initially selected as COPCs because their maximum detected concentrations were greater than their respective screening levels. Also, aluminum and iron were initially selected as COPCs because the Eco-SSL documents for these metals indicates that the toxicity of these metals are related to the pH of the soil. However, soil pH data were not available, so these metals were initially selected as COPCs.

### 5.1.2 COPCs for Sediment Invertebrates

The data used to select COPCs for sediment invertebrates include the sediment samples collected as part of the Phase I and Phase II RIs, and the FFS. The sediment data from the Area A Weapons Center was not used for the ERA but the data are included on the Figures in Attachment 1. Attachment 4 presents the analytical results for chemicals that were detected in at least one sediment sample. Table 5-2 presents the summary statistics for the sediment data, along with the selection of the COPCs. In summary, as a result of the screening process:

- Three VOCs were initially selected as COPCs because their maximum detected concentrations were greater than their respective screening levels.

- Twenty SVOCs (plus total PAHs) were initially selected as COPCs because their maximum detected concentrations were greater than their respective screening levels. One SVOC was initially selected as a COPC because a screening level was not available for this chemical.
- Fifteen pesticides (plus total DDT) and one PCB were initially selected as COPCs because their maximum detected concentrations were greater than their respective screening levels.
- Fourteen metals were initially selected as COPCs because their maximum detected concentrations were greater than their respective screening levels. Three metals were initially selected as COPCs because screening levels are not available for those metals.

### 5.1.3 COPCs for Aquatic Receptors

The data used to select COPCs for aquatic receptors include the surface water samples collected as part of the Phase I and Phase II RIs, and the surface water samples collected as part of the Area A Landfill monitoring program with the exception of the seep sample at location 3MSP01 because that sample is not located in the Area A Wetland. Attachment 4 presents the analytical results for chemicals that were detected in at least one surface water sample during the Phase I and Phase II RIs. Appendix D presents the analytical results for chemicals that were detected in at least one surface water sample collected as part of the Area A Landfill monitoring program. Tables 5-3 and 5-4 present the summary statistics for the surface water data collected as part of the Phase I and Phase II RIs, and Area A Landfill monitoring program, respectively, along with the selection of the COPCs. In summary, as a result of the screening process:

#### Phase I and Phase II RI Data

- Nine metals (total) were initially selected as COPCs because their maximum detected concentrations were greater than their respective screening levels. One metal (total) was initially selected as a COPC because a screening level was not available for this metal.
- Five metals (dissolved) were initially selected as COPCs because their maximum detected concentrations were greater than their respective screening levels. One metal (dissolved) was initially selected as a COPC because a screening level was not available for this metal.

#### Area A Landfill Data

- Three PAHs and one non-PAH SVOC were initially selected as COPCs because their maximum detected concentration were greater than their respective screening levels.
- Fourteen metals (total) were initially selected as COPCs because their maximum detected concentrations were greater than their respective screening levels.
- Seven metals (dissolved) were initially selected as COPCs because their maximum detected concentrations were greater than their respective screening levels.

#### **5.1.4**      COPCs for Wildlife

Tables 5-6 and 5-7 list the chemicals that retained for food chain modeling because they were bioaccumulative chemicals and/or were detected at concentrations that were greater than screening levels based on risks to wildlife. Most of the screening levels used to select the COPCs in sections 5.1.1 through 5.1.3 are not based on risks to wildlife, but are based on direct risks to plants or invertebrates. Therefore, chemicals that are considered to be important bioaccumulative chemicals (USEPA, 2000) were retained for food chain modeling even if their detected concentrations were less than their respective screening level. A few exceptions are noted on Tables 5-1 and 5-2. The surface water data from the Phase I and Phase II RIs were included in the dose equation in the food chain model, for the chemicals that were retained as COPCs in soil and sediment.

Of the detected chemicals in surface soil, fourteen SVOCs, five pesticides, one PCB, and nine metals were retained for food chain modeling. Of the detected chemicals in sediment, 19 SVOCs, 19 pesticides, one PCB, and ten metals were retained for food chain modeling. Tables 5-6 and 5-7 summarize the results of the conservative food chain modeling for the terrestrial and aquatic receptors, respectively. Attachment 5 presents the calculation worksheets. Food chain modeling using maximum chemical concentrations and conservative input parameters resulted in the following:

#### Soil Herbivorous Receptors

- Mercury and selenium were retained as COPCs because the NOAEL EEQs were greater than 1.0 in the conservative scenario food chain model.

#### Soil Invertivorous Receptors

- Aroclor-1260 and seven metals were retained as COPCs because the NOAEL EEQs were greater than 1.0 in the conservative scenario food chain model.

#### Sediment Invertivorous Receptors

- Eight PAHs, three pesticides, Aroclor-1260 and eight were retained as COPCs because the NOAEL EEQs were greater than 1.0 in the conservative scenario food chain model.

## 6.0 STEP 3A REFINEMENT

Step 3a consists of a refinement of the conservative exposure assumptions/concentrations to evaluate the potential risks to ecological receptors (i.e., plants, invertebrates, and wildlife receptors). The objective of the Step 3a evaluation is to further reduce the number of chemicals that are retained as COPCs in order to focus additional efforts on chemicals that are of major ecological concern. Because the primary objective of this updated ERA is to assist in determining the data needs for the Phase III investigation, the Step 3a refinement will primarily focus on general classes of chemicals to focus future sampling efforts on the risk drivers.

### 6.1 Terrestrial Plants and Invertebrates

Table 5-1 lists the chemicals detected in surface soil that were initially selected as COPCs for plants and invertebrates. Attachment 4 in Appendix B presents the positive detection data for surface soil.

Four VOCs in the surface soil samples were retained as COPCs because screening levels were not available for those chemicals. Generally, VOCs are not considered toxic to plants and invertebrates at low concentrations as indicated by the relatively high screening levels for the VOCs that did have screening levels tetrachloroethene (3,800 ug/kg) and trichloroethene (3,000 ug/kg). Also, the four VOCs, acetone, 2-butanone, carbon disulfide, and methylene chloride, are common or potential laboratory contaminants. For these reasons, VOCs are not chemicals of ecological concern for plants or invertebrates that warrant further evaluation at this site.

Two PAHs (plus total PAHs) were retained as COPCs because they were detected at concentrations that exceeded the Canadian SQG for benzo(a)pyrene of 0.7 mg/kg which was based on decreased growth efficiency in woodlice (EC, 1999a). This value is based on a food concentration that was converted to a soil concentration assuming a 0.3 percent fraction of organic carbon. This approach was used because the soil concentration of benzo(a)pyrene required to produce an effect in the organisms tested were too high to go into solution for application (EC, 1999a). In Appendix III of the Canadian SQG document (EC, 1999a), a No Observed Effects Concentration (NOEC) of 26,000 mg/kg (based on mortality) was reported for earthworms after 14 days. The lowest reported NOEC value for plants was 4,400 mg/kg and was based on seedling emergence after 3 days of exposure. Because the maximum detected concentrations of both of these PAHs were only slightly greater than the SQG, impacts to plants and invertebrates are not expected. The only sample in which the SQG was exceeded was at 2WMW39DS, which is located immediately next to several sediment samples (see total PAH

figure in Attachment 1) so this location may be more representative of sediment. In fact, the boring log from the installation of the monitoring well at this location indicated that the ground was saturated). Risks to sediment invertebrates in this area are discussed in Section 6.2. For these reasons, PAHs are not chemicals of ecological concern for plants or invertebrates that warrant further evaluation at this site.

Two other SVOCs, benzoic acid and bis(2-ethylhexyl)phthalate were initially selected as COPCs because they did not have soil screening levels. Bis(2-ethylhexyl)phthalate is a common laboratory contaminant and is not expected to be site-related. Benzoic acid does not appear to be site-related because it was detected at relatively low concentrations across the site. Therefore, SVOCs are not chemicals of ecological concern for plants or invertebrates that warrant further evaluation at this site.

Two pesticides (alpha- and gamma-chlordane) were initially selected as COPCs because they did not have soil screening levels. They were detected in only 1 of 9 samples each, at relatively low concentrations (3 and 2.2 ug/kg). Therefore, they are not widespread across the site and are unlikely to impact plants or invertebrates. Therefore, pesticides are not chemicals of ecological concern for plants or invertebrates that warrant further evaluation at this site.

Eight metals were initially selected as COPCs because their maximum detections exceeded screening levels. Also, aluminum and iron were initially selected as COPCs because the Eco-SSL documents for these metals indicates that they should be retained as COPCs only when the soil pH is less than 5.5 (for aluminum) or less than 5 or greater than 8 (for iron). Although soil pH data were not available for the Area A Wetland, aluminum and iron are typically not considered to be toxic to terrestrial plants or invertebrates because their bioavailability is low. Therefore, aluminum and iron are not likely to impact plants or invertebrates at the site and do not warrant further evaluation at this site for those receptors.

Cadmium, chromium, copper, lead, silver, and vanadium were initially selected as COPCs because they were detected at concentrations that exceeded the lower of their respective plant, invertebrate, or wildlife Eco-SSL values. For all of these metals, the wildlife Eco-SSL value was the lowest, or there were not Eco-SSL values for plants or invertebrates. Table 6-1 presents the maximum and average concentrations of each of these metals, along with the lower of the plant or invertebrate Eco-SSL values (USEPA, 2005b, 2005c, 2006, and 2007), if available. Of these metals, the maximum detected concentrations of cadmium, copper, and silver were less than these Eco-SSL values, and the maximum concentration of lead was just slightly greater than the Eco-SSL value, so risks to plants or invertebrates are not expected from these metals. Eco-SSL

for plants or invertebrates are not available for chromium or vanadium so Table 6-1 presents the Canadian Soil Quality Guidelines (SQGs) for these metals (EC, 1999b and 1999c). The Canadian SQGs were calculated to be protective of plants and invertebrates from direct contact. The maximum chromium concentration slightly exceeds the Canadian SQG, but the average concentration is less than the SQG. Therefore, slight exceedence of the SQG does not warrant retaining chromium as a COPC for further evaluation of risks to plants or invertebrates. The maximum vanadium concentration is less than the Canadian SQG so risks to plants or invertebrates are not expected.

Finally, mercury, selenium, and zinc were initially selected as COPCs because they were detected at concentrations that exceeded conservative benchmarks for plants or invertebrate (Efroymsen et al., 1997a, 1997b). Therefore, the concentrations of these three metals were compared to The Canadian SQGs, which are based on more current data than the screening benchmarks (see Table 6-1). The maximum concentrations of mercury and zinc were less than their respective Canadian SQGs so risks to plants or invertebrates are not expected. Concentrations of selenium were slightly greater than its Canadian SQG in a few samples so slight risks to plants and/or invertebrates are possible at these locations. However, based on the relatively low concentrations of selenium in the soil (<2.5 mg/kg), impacts are not expected to be significant enough to warrant retaining selenium as a COPC for further evaluation of risks to plants or invertebrates.

## **6.2 Sediment Invertebrates**

Table 5-2 lists the chemicals detected in sediment that were initially selected as COPCs for sediment invertebrates. Attachment 4 presents the positive detection data for sediment.

Of the nine VOCs that were detected in the sediment, three were retained as COPCs because their maximum detections exceeded the screening level (acetone, 2-butanone, and carbon disulfide). All three of these chemicals are common or possible laboratory contaminants. Therefore, these VOCs might not be related to site activities. Also, the screening values are secondary chronic values (SCV) which were calculated using equilibrium partitioning (Jones et al., 1997). A footnote in Jones et al., (1997) indicates that acetone and 2-butanone are polar nonionic organic compounds, for which the equilibrium partitioning model is likely to provide a conservative estimate of exposure. Therefore, those screening levels are very conservative. Finally, these VOCs were detected relatively infrequently in the sediment samples (5/28 to 7/28). For these reasons, risks to sediment invertebrates from VOCs are not expected to be significant enough to warrant retaining them as COPC for further evaluation of risks to these receptors.

Of the 21 SVOCs (plus total PAHs) that were initially selected as COPCs in the sediment samples, 16 were PAHs, and the rest were 2,4-dimethylphenol, benzoic acid, bis(2-ethylhexyl)phthalate, carbazole, and pentachlorophenol. Attachment 1 presents a figure that shows the total PAH results compared to the screening level (1,610 ug/kg), which is the Threshold Effects Concentration (TEC), as well as the Probable Effects Concentration (PEC) of 22,800 ug/kg from MacDonald et al. (2000). Total PAHs were evaluated instead of the individually detected PAHs in the sediment as part of the Step 3a refinement. This was done because the toxicity of PAHs is additive and several studies have reported toxicity data for total PAHs (Di Toro, et al., 2000). As can be seen from the figure, total PAHs were detected in several samples at concentrations that exceeded the TEC, and were detected in four samples at concentrations that exceeded the PEC. The samples with the greatest concentrations were located adjacent to the Area A Landfill and Area A Weapons Center. The sediment samples in the eastern portion of the Area A Wetland had PAH concentrations that were less than the screening level. Therefore, risks to sediment invertebrates from PAHs are possible so they are retained as COPCs for further evaluation of risks to these receptors.

The remaining five SVOCs are not retained as COPCs for further evaluation of risks to sediment invertebrates for several reasons. 2,4-dimethylphenol; bis(2-ethylhexyl)phthalate, and pentachlorophenol were detected infrequently in 1 of 28 samples and benzoic acid was detected in 2 of 28 samples. Carbazole was detected in 6 of 19 samples. Toxicity data are not available for this chemical but the maximum detected concentrations is relatively low (130 ug/kg) compared to the concentrations for the PAHs. Therefore, it is assumed that because PAHs are being retained as COPCs for further evaluation, any potential risks from carbazole would be accounted for by evaluating risks to sediment invertebrates from PAHs.

Several pesticides were initially selected as COPCs in the sediment samples because they were detected at concentrations that exceeded their respective screening levels. Of these pesticides, 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT are considered to be the risk drivers. They were detected most frequently (in approximately 36% of the samples) in the sediment samples, and had the greatest EEQs compared to their respective screening levels (see Table 5-2). The other pesticides were detected less frequently, and/or at much lower concentrations compared to their respective screening levels. Alpha and gamma chlordane were detected in approximately 30 percent of the sediment samples, but at much lower concentrations compared to the screening level for chlordane. Attachment 1 presents a figure that shown the total DDT (sum of 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT) results compared to the screening level (5.28 ug/kg), which is the TEC, as well as the PEC of 572 ug/kg from MacDonald et al., (2000). As can be seen from the figure,

total DDT was detected in several samples at concentrations that exceeded the TEC and PEC. The samples with the greatest concentrations were located adjacent to the Area A Landfill. The sediment samples in the northern and eastern portion of the Area A Wetland had total DDT concentrations that were less than the screening level. Therefore, risks to sediment invertebrates from pesticides are possible so they are retained as COPCs for further evaluation of risks to these receptors. However, the further evaluation will focus on 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT because these are the primary pesticide risk drivers. It is assumed that any potential risks to sediment invertebrates from the other pesticides would be accounted for by evaluating risks from 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT.

Ecological-Based Remedial Action Objectives (RAO) were developed as part of the Feasibility Study for Soil and Sediment Area A Downstream/OBDA Site 3 (Brown and Root Environmental, 1997). For sediment, an RAO of 2 mg/kg was developed for total DDT (DDTR) using a combination of sediment chemistry data, toxicity test data, and macroinvertebrate data. Three of the detected concentrations DDTR in the Area A Wetland were greater than 2 mg/kg at locations 2WSD24, 2WSD25, and TB3). As can be seen from the total DDT figure in Attachment 1, these samples are located in the far western portion of the wetland.

Aroclor-1260 was the only PCB initially selected as a COPC in the sediment samples. Attachment 1 presents a figure that shows the Aroclor-1260 results compared to the screening level (59.8 ug/kg), which is the TEC, as well as the PEC of 676 ug/kg from MacDonald et al., (2000). Although from the figure, it appears that Aroclor-1260 was detected in most samples at concentrations that exceeded the TEC, as discussed in Section 2.3, the figure shows non-detects at the detection limit. As seen on Table 5-2 and the positive detection table in Attachment 4, Aroclor-1260 was detected in 6 of 33 samples. All of the detections were in the samples located adjacent to the Area A Landfill (T1A, T1B, T6A, T6B, T7A, and T7B). Therefore, risks to sediment invertebrates from Aroclor-1260 are possible in this area so PCBs are retained as COPCs for further evaluation of risks to these receptors.

Several metals were retained as COPCs because they were detected at concentrations that exceeded their respective screening levels. Attachment 1 presents figures that show the concentrations of selected metals (cadmium, copper, lead, mercury, nickel, and zinc) compared to their respective TECs and PECs. As can be seen from the figures, several samples had concentrations of these metals that exceeded the TEC, but fewer samples had concentrations that exceeded the PEC. There does not appear to be a specific pattern to the elevated detections of the metals, with the exception that the concentrations were greatest at 2WTB2, adjacent to the Area A Landfill, and in a few samples near the Area A Weapons Center. Other

metals that were not plotted also were detected at concentrations that exceeded their respective screening levels. Therefore, risks to sediment invertebrates from metals are possible so metals are retained as COPCs for further evaluation of risks to these receptors.

### **6.3 Aquatic Organisms**

Tables 5-3 and 5-4 list the chemicals detected in surface water that were initially selected as COPCs for aquatic organisms. Attachment 4 in Appendix B presents the positive detection data for surface water. Two surface water samples were collected during the Phase I RI and nine samples were collected during the Phase II RI. The two locations that were sampled as part of the Phase I were resampled during Phase II. Therefore, Table 5-3 only presents the Phase II data.

Gross alpha and beta were the only parameters that were analyzed for during the Phase I RI that were not analyzed for during the Phase II RI. Radionuclides are not known to be a concern in the Area A Wetland or related to site activities so they are not retained as COPCs for further evaluation of risks to aquatic organisms.

Bis(2-ethylhexyl)phthalate was the only non-PAH SVOC that was initially selected as a COPC. Although it was detected at a concentration that exceeded its screening level, it was detected in only 2 of 24 samples. Also, bis(2-ethylhexyl)phthalate is a common laboratory contaminant. For these reasons, it is not retained as a COPC for further evaluation of risks to aquatic organisms.

Three PAHs were initially selected as COPCs because they were detected at concentrations that exceeded their respective screening levels. They were detected relatively infrequently (in 2 of 24 or 5 of 24 samples) at concentrations that just slightly exceeded their screening levels. Therefore, they are not likely to significantly impact aquatic organisms at the site and are not retained as COPCs for further evaluation of risks to aquatic organisms.

Several metals were initially selected as COPCs in the total portion because their maximum detections exceeded screening levels. Eight metals were initially selected as COPCs in the dissolved portion because their maximum detections exceeded screening levels and boron was initially selected as a COPC because it does not have a screening level (see Tables 5-3 and 5-4). In accordance with USEPA (1993), dissolved metal more closely approximates the bioavailable fraction of metal in the water column than total recoverable metal. Six of the nine surface water samples collected during the Phase II RI were analyzed for both total and dissolved metals; three samples were only analyzed for total metals. The three samples that were only analyzed for total

metals, 2WSW8, 2WSW9, and 2WSW10, were located south of the Area A wetland, in areas with little aquatic habitat (see Figure 1-3). Therefore, this evaluation will only focus on the six samples that were analyzed for dissolved metals.

Based on the Phase II RI data, several metals were detected in the dissolved fraction at concentrations that exceeded screening levels. The metals with the greatest magnitude of exceedences compared to the screening levels are barium, iron and manganese (see Table 5-3 and the positive detection table in Attachment 4). The greatest concentrations of these metals were in the samples located closest to the landfill (2WSW11 and 2WSW12) and the sample just above the dike (2WSW2). Similar results were found for the chemicals detected in the samples collected as part of the Area A Landfill monitoring program, with concentrations of barium, iron, and manganese having the greatest magnitude of criteria exceedences (see Table 5-4 and the positive detection table in Appendix D). Concentrations of other metals such as copper, lead, and zinc exceeded their respective criteria in only a few samples.

In summary, metals were detected in several samples at concentrations that exceeded screening levels so impacts to aquatic organisms from the metals are possible. The amount of water in the wetland varies throughout the year based on the amount of rainfall. Because of this, the concentrations of metals in the surface water samples vary throughout the rounds. For example, at location SG-19, concentrations of iron in the filtered samples were 50 ug/L, 160 ug/L, 11,500 ug/L, and 1,900 ug/L (50 U in the duplicate sample) in samples collected during Rounds 16, 17, 18, and 19, respectively. Two of the concentrations are less than the screening level of 1,000 ug/L and two concentrations are greater than the screening level. Therefore, the potential for impacts to aquatic organisms will vary over time.

#### **6.4 Terrestrial Receptors**

As presented in Section 5.1.4, the EEQs from the terrestrial food chain modeling were greater than 1.0 for several chemicals using maximum chemical concentrations and conservative exposure assumptions. Therefore, as part of the Step 3a refinement, risks were recalculated using average chemical concentrations and average exposure assumptions.

Table 6-2 presents the NOAEL and LOAEL EEQs from the terrestrial food chain models for soil herbivorous and invertivorous receptors using average inputs and Table 6-3 presents the NOAEL and LOAEL EEQs from the terrestrial food chain models for sediment invertivorous receptors using average inputs. The following bullets summarize the results for the various receptors:

- Soil herbivorous receptors: No chemicals had NOAEL EEQs greater than 1.0 using the average scenario.
- Soil invertivorous receptors: NOAEL EEQs greater than 1.0 for cadmium, copper, lead, mercury, vanadium, and zinc in the robin food chain model; NOAEL EEQs greater than 1.0 for Aroclor-1260, cadmium, and mercury in the shrew food chain model. Mercury in the robin model was the only chemical that had a LOAEL EEQ slightly greater than 1.0 with an EEQ of 1.6.
- Sediment invertivorous receptors: NOAEL EEQs were greater than 1.0 for Aroclor-1260, copper, mercury, and nickel in the mallard food chain model; the NOAEL EEQ was greater than 1.0 for Aroclor-1260 in the raccoon food chain model.

With the exception of mercury in the robin model, the EEQs based on NOAELs in the robin and shrew food chain models were low (<4.0) and the EEQs based on LOAELs were less than 1.0. Therefore, it is not likely that small mammals or birds will be significantly impacted by these chemicals so they are not retained as COPCs for further evaluation of risks to these receptors.

In the robin model, mercury had an EEQ of 16 based on the NOAEL, and an EEQ of 1.6 based on the LOAEL. Mercury was detected in 4 of 9 soil samples at maximum and average concentrations of 0.69 mg/kg and 0.25 mg/kg, respectively. The average mercury concentration is slightly greater than the site-specific background concentrations of mercury (0.055 mg/kg). The elevated EEQs is primarily due to the conservativeness of the model and the very low TRVs for birds. However, based on the relatively low concentrations of mercury in the soil samples across the site (see Table 1 in Attachment 4), it does not appear that mercury is related to site activities. Finally, although the exact locations of the bird samples is not known, mercury was detected in only 1 of 17 bird samples at a low concentration of 0.083 mg/kg (see Tables 4 and 6 in Attachment 4). This is likely because the high levels of organic carbon in the Area A Wetland (see Figure in Attachment 1) are reducing the bioavailability of mercury. Therefore, mercury is not retained as a COPC for further evaluation of risks to birds.

For metals in the mallard and raccoon models, the EEQs based on NOAELs were low (<4.0) and the EEQs based on LOAELs were less than 1.0. Therefore, it is not likely that mammals or birds will be significantly impacted by metals so they are not retained as COPCs for further evaluation of risks to these receptors.

For Aroclor-1260, the EEQs based on the NOAEL were greater than 1.0 for the mallard and the raccoon models, but the EEQs were less than 1.0 based on the LOAELs. Aroclor-1260 was detected in only 6 of 33 samples at a maximum concentration of 1.5 mg/kg. As indicated in Section 6.2, all of the detections were in the samples located adjacent to the Area A Landfill. The TOC in these samples was relatively high 2 to 6 percent (see TOC Figure in Attachment 1 and the analytical results in Table 2 in Attachment 4) so the bioavailability of the PCBs is expected to be low. Finally, the home range for the mallard (275 to 1500 acres) and the raccoon (96 to 6300 acres) are much larger than the size of the Area A Wetland (23 acres). Therefore, because the EEQs based on the LOAEL were less than 1.0, the bioavailability of the PCBs are expected to be low, and invertivorous birds and mammals will not likely obtain all of their food from the site, risks to these receptors are expected to be low so PCBs are not retained as COPCs for further evaluation of risks to invertivorous birds and mammals.

## 7.0 ECOLOGICAL RISK UNCERTAINTY ANALYSIS

This section presents some of the general uncertainties associated with ecological risk assessment.

### 7.1 Uncertainty in Measurement and Assessment Endpoints

Measurement endpoints were used to evaluate the assessment endpoints that were selected for this ERA, but the measures of effects were not the same as the assessment endpoints. Therefore, the measures were used to predict effects to the assessment endpoints by selecting surrogate species that were evaluated. For example, mortality of a raccoon was used to assess mortality of the small mammal population. However, predicting mortality to a raccoon may either under or overprotect the small mammal population, resulting from differences in ingestion rates, toxicity, food preferences, etc., between the different species.

Several endpoints were not quantitatively evaluated in the ERA. For example, risks to reptiles and amphibians were not quantitatively evaluated because exposure factors are not established for most species, and toxicity data are very limited.

### 7.2 Uncertainty in Exposure Characterization

The contaminant dose to terrestrial wildlife is calculated using an equation that incorporates ingestion rates, body weights, bioaccumulation factors, and other exposure factors. These exposure factors are obtained from literature studies or predicted using various equations. Ingestion rates and body weights vary between species, especially between species inhabiting different areas.

Bioaccumulation of contaminants into various biological media (i.e., plants, invertebrates, small mammals) depends on characteristics of the media such as pH, organic carbon, etc. The bioaccumulation factors that were used for the ERA came from a variety of sources, as indicated in Appendix G.4. All the values were from the literature because no site-specific values are available. There are uncertainties associated with accumulation factors from the literature because they may either underpredict or overpredict tissue concentrations, depending upon how representative the factors are for site conditions. For the organic chemicals, adjustments are made for differences in percent lipid values for invertebrates which helps decrease some of the uncertainties.

Frog tissue samples were collected during the Phase I RI. The frog data were not used in the food chain model because it was assumed that the raccoons would consume 100 percent sediment invertebrates, which is more conservative than a diet of invertebrates and frogs. This can be seen by comparing the maximum concentrations of metals in the tissue samples (Table 5 in Attachment 4) to the metals concentrations estimated in sediment invertebrates in the food chain model (Attachment 5). Therefore, potential risks to sediment invertivorous mammals would have been lower than what was predicted in the food chain model had the frog data been used.

### **7.3 Uncertainty in Ecological Effects Data**

Uncertainty exists in the ecological effects data, including the screening levels and wildlife TRVs. Several of the screening levels are very conservative, and typically are based on studies where the bioavailability of the chemical is much greater than it is in the environment.

The NOAELs/LOAELs used for the wildlife endpoints species are based on species other than the endpoint species (i.e., rats, mice). Uncertainty exists in the application of toxicity data across species because the contaminant may be more or less toxic to the endpoint species than it was to the test study species.

### **7.4 Uncertainty in Risk Characterization**

The potential for adverse risks may be anticipated if an EEQ is greater than or equal to 1.0 regardless of the magnitude of the EEQ. Although the relationship between the magnitude of an EEQ and toxicity is not necessarily linear, the magnitude of an EEQ can be used as rough approximation of the extent of potential risks, especially if there is sufficient confidence in the screening level used. Uncertainty exists in how the predicted risks to a species at the sites translate into risk to the population in the area as a whole.

## 8.0 SUMMARY/CONCLUSIONS

This section presents a summary of the conclusions of the ecological risk assessment that was conducted for the site.

### 8.1 Risks to Terrestrial Plants and Invertebrates

Although several chemicals were detected at concentrations that exceeded their respective screening levels, during the Step 3a evaluation, it was determined that risks were not great enough for any chemicals to warrant further evaluation at this site.

### 8.2 Sediment Invertebrates

Several chemicals were initially selected as COPCs because they were detected at concentrations that exceeded their respective screening levels. However, during the Step 3a evaluation, it was determined that the primary risk drivers were PAHs, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, PCBs, and metals. Therefore, risks to sediment invertebrates need to be further evaluated for these chemicals.

### 8.3 Aquatic Organisms

Several chemicals were initially selected as COPCs because they were detected at concentrations that exceeded their respective screening levels. Therefore, there may be potential impacts to aquatic organisms that are exposed to these chemicals. The concentrations of most of the chemicals only exceeded their respective screening level in a few samples. However, concentrations of barium, iron, and manganese had the greatest numbers and magnitude of criteria exceedences. The concentrations of metals in the surface water samples vary throughout the rounds so the potential for impacts to aquatic organisms will vary over time.

### 8.4 Terrestrial Receptors

Several chemicals had EEQs greater than 1.0 based on the conservative food chain models. Fewer chemicals had EEQs greater than 1.0 after the food chain model was refined in Step 3a. During the Step 3a evaluation, it was determined that risks to wildlife were not great enough for any chemicals to warrant further evaluation at this site.

## References

Brown and Root Environmental, 1997a. Phase II Remedial Investigation Report for Naval Submarine Base - New London, Groton, Connecticut. Wayne, Pennsylvania. March.

EC (Environment Canada), 1999a. Canadian Soil Quality Guidelines for Benzo(a)pyrene. Scientific Supporting Document. National Guidelines and Standards Office, Environmental Quality Branch, Environment Canada. Ottawa.

EC. 1999b. Canadian Soil Quality Guidelines for Chromium. Scientific Supporting Document. National Guidelines and Standards Office, Environmental Quality Branch, Environment Canada. Ottawa.

EC. 1999c. Canadian Soil Quality Guidelines for Vanadium. Scientific Supporting Document. National Guidelines and Standards Office, Environmental Quality Branch, Environment Canada. Ottawa.

Efroymsen, R.A., M.E. Will, and G.W. Suter II. 1997a. Toxicological Benchmarks for Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision. Oak Ridge National Laboratory. November. ES/ER/TM-126/R2.

Efroymsen, R.A., M.E. Will, G.W. Suter II, and A.C. Wooten. 1997b. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision. Oak Ridge National Laboratory. November. ES/ER/TM-85/R3.

Jones, D.S., R.N. Hull, and G.W. Suter II, 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Sediment-Associated Biota: 1997 Revision. Risk Assessment Program, Health Sciences Division, Oak Ridge, Tennessee. ES/ER/TM-95/R4. November.

MacDonald, D.D., C.G. Ingersoll, and T.A. Berger, 2000. "Development and Evaluation of Consensus-Based Sediment Quality Guidelines for Freshwater Ecosystems." Archives of Environmental Contamination and Toxicology, Vol. 39, pp. 20-31.

Navy (Department of the Navy). 1999. Navy Policy For Conducting Ecological Risk Assessments. Memo from Chief of Naval Operations to Commander, Naval Facilities Engineering Command, 05 April 1999. Department of the Navy, Washington, DC.

ORNL (Oak Ridge National Laboratory). 1998. Biota Sediment Accumulation Factors for Invertebrates: Review and recommendations for the Oak Ridge Reservation. BJC/OR-112. August.

Sample, B.E., D.M. Opresko, and G.W. Suter II. 1996. Toxicological Benchmarks for Wildlife: 1996 Revision. Oak Ridge National Laboratory. June. ES/ER/TM-86/R3.

USEPA (U.S. Environmental Protection Agency), 1993. Wildlife Exposure Factors Handbook. Office of Research and Development. Washington, D.C. EPA/600/R-93/187a. December.

USEPA, 1997. Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Ecological Risk Assessments. Interim Final. Environmental Response Team. June 5.

USEPA, 1998. Final Guidelines for Ecological Risk Assessment. Risk Assessment Forum, Washington, DC, EPA/630/R095/002F. April.

USEPA, 2004. The Incidence and Severity of Sediment Contamination in Surface Waters of the United States, Volume 1: National Sediment Quality Survey: Second Edition. Office of Science and Technology. Washington, D.C. EPA 823-R-04-007. November.

USEPA, 2005. Guidance for Developing Ecological Soil Screening Level. Office of Solid Waste and Emergency and Response. OSWER Directive 92857-55. February.

USEPA, 2005. Ecological Soil Screening Level for Antimony, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-61. February.

USEPA, 2005. Ecological Soil Screening Level for Arsenic, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-62. March.

USEPA, 2005. Ecological Soil Screening Level for Barium, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-63. February.

USEPA, 2005. Ecological Soil Screening Level for Beryllium, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-64. February.

USEPA, 2005. Ecological Soil Screening Level for Cadmium, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-65. March.

USEPA, 2005. Ecological Soil Screening Level for Chromium, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-66. March.

USEPA, 2005. Ecological Soil Screening Level for Cobalt, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-67. March.

USEPA, 2005. Ecological Soil Screening Level for Lead, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-70. March.

USEPA, 2005. Ecological Soil Screening Level for Dieldrin, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-57. March.

USEPA, 2005. Ecological Soil Screening Level for Pentachlorophenol, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-58. March.

USEPA, 2005. Ecological Soil Screening Level for Vanadium, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-75. April.

USEPA, 2006. Ecological Soil Screening Level for Copper, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-68. July.

TABLE 5-1

OCCURRENCE, DISTRIBUTION, AND SELECTION OF ECOLOGICAL CHEMICALS OF POTENTIAL CONCERN - SURFACE SOIL (PHASE I RI)  
SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT  
PAGE 1 OF 2

Chemical	Minimum Concentration <sup>(1)</sup>	Maximum Concentration <sup>(1)</sup>	Sample of Maximum Concentration	Average of All Samples <sup>(2)</sup>	Average of Positive Detections <sup>(3)</sup>	Frequency of Detection	Ecological Risk Screening					Retained for Food Chain Modeling? <sup>(6)</sup>
							Screening Level	Source of Screening Level	Ecological Effects Quotient <sup>(4)</sup>	Retained as COPC (yes/no)	Rationale	
Volatile Organics (ug/kg)												
2-BUTANONE	5 J	310	083090-2WTB8(1-3)	50	134	3/9	NA	NA	NA	Yes	NTX	No
ACETONE	25	850	083090-2WTB8(1-3)	181	320	5/9	NA	NA	NA	Yes	NTX	No
CARBON DISULFIDE	6 J	8 J	083090-2WTB8(1-3)	6	7	2/9	NA	NA	NA	Yes	NTX	No
METHYLENE CHLORIDE	3	6	2W-SU-45DS-00-99	4	4	3/9	NA	NA	NA	Yes	NTX	No
TETRACHLOROETHENE	7 J	7 J	083090-2WTB8(1-3)	5	7	1/9	3800	CCME	0.002	No	BSL	No
TRICHLOROETHENE	4 J	4 J	090690-2WTB4(0-2)	5	4	1/9	3000	CCME	0.001	No	BSL	No
Semivolatile Organics (ug/kg)												
ACENAPHTHENE	270	270	2W-SU-39DS-00-99	311	270	1/9	20000	ORNL - Plant	0.01	No	BSL	Yes
ACENAPHTHYLENE	59 J	120 J	083090-2WTB8(1-3)	220	85	3/9	700	CCME	0.17	No	BSL	Yes
ANTHRACENE	52 J	79 J	083090-2WTB8(1-3)	226	66	2/9	700	CCME	0.11	No	BSL	Yes
BENZO(A)ANTHRACENE	30	400 J	090690-2WTB2(0-2)	222	214	8/9	700	CCME	0.57	No	BSL	Yes
BENZO(A)PYRENE	52	390 J	083090-2WTB8(1-3)	291	209	6/9	700	CCME	0.56	No	BSL	Yes
BENZO(B)FLUORANTHENE	74 J	550 J	083090-2WTB8(1-3)	322	259	7/9	700	CCME	0.79	No	BSL	Yes
BENZO(G,H,I)PERYLENE	49	260	2W-SU-39DS-00-99	312	155	2/9	700	CCME	0.37	No	BSL	Yes
BENZO(K)FLUORANTHENE	71 J	390 J	083090-2WTB8(1-3)	271	225	6/9	700	CCME	0.56	No	BSL	Yes
BENZOIC ACID	130 J	220 J	090590-2WTB7(0-2)-D	1191	170	3/7	NA	NA	NA	Yes	NTX	No
BIS(2-ETHYLHEXYL)PHTHALATE	51.4 J	1300	083090-2WTB8(1-3)	445	482	6/9	NA	NA	NA	Yes	NTX	No
CHRYSENE	110 J	600 J	083090-2WTB8(1-3)	327	308	6/9	700	CCME	0.86	No	BSL	Yes
DIBENZO(A,H)ANTHRACENE	32	32	2W-SU-39DS-00-99	282	32	1/9	700	CCME	0.05	No	BSL	Yes
FLUORANTHENE	53 J	890	2W-SU-39DS-00-99	342	342	9/9	700	CCME	1.3	Yes	ASL	Yes
INDENO(1,2,3-CD)PYRENE	140	270 J	083090-2WTB8(1-3)	286	205	2/9	700	CCME	0.4	No	BSL	Yes
PHENANTHRENE	120 J	340 J	083090-2WTB8(1-3)	269	223	6/9	700	CCME	0.5	No	BSL	Yes
PYRENE	98 J	810	2W-SU-39DS-00-99	354	354	9/9	700	CCME	1.16	Yes	ASL	Yes
TOTAL PAHS <sup>(5)</sup>	53	4279	083090-2WTB8(1-3)	1809	1809	9/9	700	CCME	6.11	Yes	ASL	No <sup>(11)</sup>
Pesticides/PCBs (ug/kg)												
4,4'-DDD	40	69 J	090690-2WTB6(0-2)	26	50	3/9	12500	CCME	0.006	No	BSL	Yes
4,4'-DDE	5.2	10	2W-SU-45DS-00-99	13	7.6	2/9	12500	CCME	0.0008	No	BSL	Yes
4,4'-DDT	28	28	2W-SU-39DS-00-99	14	28	1/8	12500	CCME	0.002	No	BSL	Yes
ALPHA-CHLORDANE	3	3	2W-SU-39DS-00-99	54	3	1/9	NA	NA	NA	Yes	NTX	Yes
AROCLOR-1260	110	370 J	090690-2WTB2(0-2)	136	184	2/9	40000	ORNL - Plant	0.01	No	BSL	Yes
TOTAL AROCLOR	110	370	090690-2WTB2(0-2)	33	148	2/9	40000	ORNL - Plant	0.01	No	BSL	No
GAMMA-CHLORDANE	2.2	2.2	2W-SU-39DS-00-99	54	2.2	1/9	NA	NA	NA	Yes	NTX	Yes
TOTAL DDT <sup>(5)</sup>	50	73.2	2W-SU-39DS-00-99	21	64	3/9	12500	CCME	0.005856	No	BSL	No <sup>(11)</sup>
Inorganics (mg/kg)												
ALUMINUM	4860	17900 J	083090-2WTB8(1-3)	13979	13979	9/9	pH<5.5	EPA ECO-SSL <sup>(9,10a)</sup>	NA	Yes	NTX <sup>(10b)</sup>	No
ARSENIC	1.2 B	15.1	2W-SU-45DS-00-99	7.2	7.2	9/9	18	EPA ECO-SSL <sup>(9)</sup>	0.84	No	BSL	No <sup>(7)</sup>
BARIUM	28.3 B	93.8	083090-2WTB8(1-3)	54.1	54.1	9/9	330	EPA ECO-SSL <sup>(9)</sup>	0.28	No	BSL	No <sup>(7)</sup>
BERYLLIUM	0.28 B	1.8 J	090590-2WTB7(0-2)-D, 090690-2WTB2(0-2), 083090-2WTB8(1-3)	1.0	1.1	8/9	21	EPA ECO-SSL <sup>(9)</sup>	0.09	No	BSL	No
CADMIUM	0.16 B	7.2	090690-2WTB2(0-2)-D	3.8	4.2	8/9	0.36	EPA ECO-SSL <sup>(9)</sup>	20	Yes	ASL	Yes
CALCIUM	1170 J	3690 J	090690-2WTB2(0-2)-D	1799	1799	9/9	NA	NA	NA	NA	NUT	No
CHROMIUM	12.2	102	083090-2WTB8(1-3)	58.5	58.5	9/9	26	EPA ECO-SSL <sup>(9)</sup>	3.9	Yes	ASL	Yes
COBALT	4.8 B	12.7	090590-2WTB7(0-2)-D	9.2	9.2	9/9	13	EPA ECO-SSL <sup>(9)</sup>	0.98	No	BSL	No <sup>(7)</sup>
COPPER	16.9	64.1 J	090690-2WTB6(0-2)	36.0	36.0	9/9	28	EPA ECO-SSL <sup>(9)</sup>	2.29	Yes	ASL	Yes
IRON	8100	30800	090690-2WTB4(0-2)	23317	23317	9/9	5>pH>8	EPA ECO-SSL <sup>(9,10a)</sup>	NA	Yes	NTX <sup>(10b)</sup>	No

TABLE 5-1

OCCURRENCE, DISTRIBUTION, AND SELECTION OF ECOLOGICAL CHEMICALS OF POTENTIAL CONCERN - SURFACE SOIL (PHASE I RI)  
SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT  
PAGE 2 OF 2

Chemical	Minimum Concentration <sup>(1)</sup>	Maximum Concentration <sup>(1)</sup>	Sample of Maximum Concentration	Average of All Samples <sup>(2)</sup>	Average of Positive Detections <sup>(3)</sup>	Frequency of Detection	Ecological Risk Screening					Retained for Food Chain Modeling <sup>(6)</sup>
							Screening Level	Source of Screening Level	Ecological Effects Quotient <sup>(4)</sup>	Retained as COPC (yes/no)	Rationale	
LEAD	11.2 J	128 J	090690-2WTB4(0-2)	42.4	42.4	9/9	120	EPA ECO-SSL <sup>(9)</sup>	1.07	Yes	ASL	Yes
MAGNESIUM	2170	7840	083090-2WTB8(1-3)	5898	5898	9/9	NA	NA	NA	NA	NUT	No
MANGANESE	99.2	376	090690-2WTB2(0-2)-D	222	222	9/9	500	ORNL - Plant	0.75	No	BSL	No
MERCURY	0.21	0.69 J	090690-2WTB4(0-2)	0.25	0.47	4/9	0.1	ORNL - Invertebrate	6.9	Yes	ASL	Yes
NICKEL	14.7	26.9	083090-2WTB8(1-3)	21.3	21.3	9/9	38	EPA ECO-SSL <sup>(9)</sup>	0.71	No	BSL	No <sup>(7)</sup>
POTASSIUM	1000 B	4320	090690-2WTB4(0-2)	3211	3211	9/9	NA	NA	NA	NA	NUT	No
SELENIUM	0.7	2.4	2W-SU-45DS-00-99	0.92	1.35	5/9	1	ORNL - Plant	2.4	Yes	ASL	Yes
SILVER	3.5	4.5	083090-2WTB8(1-3)	1.5	3.5	2/9	4.2	EPA ECO-SSL <sup>(9)</sup>	1.07	Yes	BSL	Yes
SODIUM	118 J	3150	083090-2WTB8(1-3)	1175	1175	9/9	NA	NA	NA	NA	NUT	No
VANADIUM	33.2	75 J	083090-2WTB8(1-3)	50.8	50.8	9/9	7.8	EPA ECO-SSL <sup>(9)</sup>	9.6	Yes	ASL	Yes <sup>(8)</sup>
ZINC	49.7	125 J	082390-2WMMW2(0-2)	70.1	70.1	9/9	50	ORNL - Plant	2.5	Yes	ASL	Yes

**Footnotes**

- 1 - Sample and duplicate are considered as two separate samples when determining the minimum and maximum concentrations detected and as one sample when determining the frequency of detection.
- 2 - Average of all analytical results are calculated using half of the detection limit for nondetects.
- 3 - Average of positive analytical results only
- 4 - The ecological effects quotient is the maximum detected concentration divided by the screening level
- 5 - Values are based on positive detections only.
- 6 - Important bioaccumulative chemicals from USEPA (2000) were retained for food chain modeling even if the maximum detected concentration was less than the screening level, except as noted in Footnote 7 and 8
- 7 - Not retained as a COPC because the maximum detected concentration was less than the Eco SSL based on mammal and/or birds.
- 8 - Although vanadium is not considered an important bioaccumulative chemical from USEPA (2000), it was retained for food chain modeling because the maximum detected concentration was greater than an Eco SSL based on wildlife.
- 9 - Source of screening value is the lower of the plant, invertebrate, or wildlife ecological soil screening level (Eco-SSL) (U.S. EPA, 2003 and 2005)
- 10 - a) Eco SSL is based on the pH of the soil; b) Soil pH data are not available.
- 11 - Although these parameters are considered bioaccumulative, they are not included in the food chain model because the food chain model is conducted on the individual chemicals that make up the totals

COPC = Chemical of Potential Concern

CCME = Canadian Council of Ministers of the Environment

NA = Not Available or Not Applicable.

ORNL - Oak Ridge National Laboratory

**Rationale Codes:**

For Selection as a COPC or for Further Evaluation

ASL = Above COPC Screening Level

BSL = Below COPC Screening Level

NTX = No Toxicity Data Available/Screening Level not Available

NUT = Essential Nutrient

**Sources:**

- CCME, 1997 *Recommended Canadian Soil Quality Guidelines*, Canadian Council of Ministers of the Environment, Ottawa, Ontario March, and subsequent updates
- ORNL-Plant (Efroymsen, R.A., M.E. Will, G.W. Suter II, and A.C. Wooten, 1997a. *Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision*. Oak Ridge National Laboratory, November. ES/ER/TM-85/R3)
- ORNL Invertebrate (Efroymsen, R.A., M.E. Will, and G.W. Suter II, 1997b. *Toxicological Benchmarks for Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision*. Oak Ridge National Laboratory November. ES/ER/TM-126/R2)
- EPA Eco SSL - USEPA Ecological Soil Screening Guidance Document for each chemical available at: <http://www.epa.gov/ecotox/ecossl/>.

TABLE 5-2

OCCURENCE, DISTRIBUTION, AND SELECTION OF ECOLOGICAL CHEMICALS OF POTENTIAL CONCERN - SEDIMENT (PHASE I AND II RIS AND FFS)  
SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT  
PAGE 1 OF 3

Chemical	Minimum Concentration <sup>(1)</sup>	Maximum Concentration <sup>(1)</sup>	Sample of Maximum Concentration	Average of All Samples <sup>(2)</sup>	Average of Positive Detections <sup>(3)</sup>	Frequency of Detection	Ecological Risk Screening					Retained for Food Chain Modeling?
							Screening Level	Source of Screening Level	Ecological Effects Quotient <sup>(4)</sup>	Retained as COPC (yes/no)	Rationale	
Volatile Organics (ug/kg)												
2-BUTANONE	42 J	1400	T8-B	88	424	5/28	270	SCV	5.2	Yes	ASL	No
ACETONE	130	720	112690-2WSD7(0-0.5)	78	294	6/28	8.7	SCV	83	Yes	ASL	No
CARBON DISULFIDE	3 J	18 J	112690-2WSD3(0-0.5)	9.5	12.4	7/28	0.85	SCV	21	Yes	ASL	No
CHLOROBENZENE	2 J	14 J	T7-B	9.1	6.3	3/29	820	USEPA	0.017	No	BSL	No
METHYLENE CHLORIDE	2 J	10 J	112690-2WSD4(0-0.5), 112690-2WSD6(0-0.5)	13	6.0	5/28	370	SCV	0.03	No	BSL	No
TETRACHLOROETHENE	4 J	16 J	112690-2WSD4(0-0.5)	9.5	10	4/28	530	USEPA	0.03	No	BSL	No
TOLUENE	2 J	6 J	T8-B	8	4	6/28	670	USEPA	0.01	No	BSL	No
TOTAL XYLENES	3 J	3 J	T6-B	9	3	1/28	4	NOAA	0.8	No	BSL	No
TRICHLOROETHENE	3 J	11 J	112690-2WSD4(0-0.5)	9.6	6.7	3/28	1600	USEPA	0.007	No	BSL	No
Semivolatile Organics (ug/kg)												
1,4-DICHLOROBENZENE	42 J	42 J	T6-B	746	42	1/28	350	USEPA	0.12	No	BSL	Yes
2,4-DIMETHYLPHENOL	210 J	210 J	T8-B	753	210	1/28	18	NOAA	12	Yes	ASL	No
2-METHYLNAPHTHALENE	40 J	-55 J	T3-A	728	49	4/28	20.2	NOAA	2.7	Yes	ASL	Yes
4-METHYLPHENOL	43 J	43 J	T9-A	750	43	1/28	100	NOAA	0.43	No	BSL	No
ACENAPHTHENE	30 J	380 J	T7-B	693	122	9/29	290	NOAA	1.3	Yes	ASL	Yes
ACENAPHTHYLENE	34 J	390	T3-A	743	137	4/28	160	NOAA	2.4	Yes	ASL	Yes
ANTHRACENE	34 J	2400 J	112690-2WSD9(0-0.5)	635	242	15/28	57.2	TEC	42	Yes	ASL	Yes
BENZO(A)ANTHRACENE	25 J	27000	112690-2WSD9(0-0.5)	1581	1584	22/29	108	TEC	250	Yes	ASL	Yes
BENZO(A)PYRENE	30 J	35000	112690-2WSD9(0-0.5)	1895	2008	21/29	150	TEC	233	Yes	ASL	Yes
BENZO(B)FLUORANTHENE	44 J	55000	112690-2WSD9(0-0.5)	2412	2863	21/29	1800	NOAA	30.6	Yes	ASL	Yes
BENZO(G,H,I)PERYLENE	27 J	23000	112690-2WSD9(0-0.5)	1427	1411	20/29	170	OMOE	135	Yes	ASL	Yes
BENZO(K)FLUORANTHENE	62 J	45000	112690-2WSD9(0-0.5)	2198	2463	22/29	240	OMOE	188	Yes	ASL	Yes
BENZOIC ACID	780 J	32000 J	112690-2WSD9(0-0.5)	3995	16390	2/28	65	NOAA	492	Yes	ASL	No
BIS(2-ETHYLHEXYL)PHTHALATE	3500 J	3500 J	T10-B	871	3500	1/28	750	NOAA	4.67	Yes	ASL	No
BUTYL BENZYL PHTHALATE	21 J	390 J	T7-B	718	112	5/29	11000	TEC	0.04	No	BSL	No
CARBAZOLE	25 J	130 J	T5-A	212	53	6/19	NA	NA	NA	Yes	NTX	No
CHRYSENE	38 J	42000	112690-2WSD9(0-0.5)	2128	2276	23/29	166	TEC	253	Yes	ASL	Yes
DIBENZO(A,H)ANTHRACENE	72 J	310 J	T2-A-D	726	125	6/28	33	TEC	9.39	Yes	ASL	Yes
DIBENZOFURAN	35 J	1000 J	112690-2WSD9(0-0.5)	599	169	10/29	2000	USEPA	0.5	No	BSL	No
DI-N-BUTYL PHTHALATE	23 J	63 J	T3-B	771	36	8/28	11000	USEPA	0.006	No	ASL	No
FLUORANTHENE	56 J	80000	112690-2WSD9(0-0.5)	3407	3647	26/29	423	TEC	189	Yes	ASL	Yes
FLUORENE	21 J	1000 J	112690-2WSD9(0-0.5)	596	169	12/29	77.4	TEC	13	Yes	ASL	Yes
INDENO(1,2,3-CD)PYRENE	22 J	23000	112690-2WSD9(0-0.5)	1396	1318	21/29	200	OMOE	115	Yes	ASL	Yes
NAPHTHALENE	56 J	77 J	T9-A	725	68	5/28	176	TEC	0.44	No	BSL	Yes
PENTACHLOROPHENOL	240 J	240 J	T1-B	3212	240	1/28	17	NOAA	14	Yes	ASL	Yes
PHENANTHRENE	22 J	36000	112690-2WSD9(0-0.5)	1841	1920	23/29	204	TEC	176	Yes	ASL	Yes
PYRENE	46 J	42000 J	112690-2WSD9(0-0.5)	2226	2295	27/29	195	TEC	215	Yes	ASL	Yes
TOTAL PAHS <sup>(5)</sup>	310	411400	112690-2WSD9(0-0.5)	17410	18700	27/29	1610	TEC	256	Yes	ASL	No <sup>(6)</sup>
Pesticides/PCBs (ug/kg)												
4,4'-DDD	4.5 J	4800 J	2WSD25 (0.0-1.0)	239	799	17/59	4.88	TEC	984	Yes	ASL	Yes
4,4'-DDE	6.2 J	720 J	2WSD25 (0.0-1.0)	42	117	18/62	3.16	TEC	228	Yes	ASL	Yes
4,4'-DDT	4.4 J	2900	2WSD25 (0.0-1.0), T3-B	146	586	13/56	4.16	TEC	697	Yes	ASL	Yes
ALDRIN	3.2 J	3.2 J	T5-A	4.4	3.2	1/38	2	OMOE	1.60	Yes	ASL	Yes
ALPHA-CHLORDANE	2.9 J	29	T6-A, T6-A-D	24	11	12/39	3.24	TEC	8.95	Yes	ASL	Yes
AROCLOR-1260	82 J	1500	T6-A-D	181	490	6/33	59.8	TEC	25.1	Yes	ASL	Yes

TABLE 5-2

OCURRENCE, DISTRIBUTION, AND SELECTION OF ECOLOGICAL CHEMICALS OF POTENTIAL CONCERN - SEDIMENT (PHASE I AND II RIs AND FFS)  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT  
 PAGE 2 OF 3

Chemical	Minimum Concentration <sup>(1)</sup>	Maximum Concentration <sup>(1)</sup>	Sample of Maximum Concentration	Average of All Samples <sup>(2)</sup>	Average of Positive Detections <sup>(3)</sup>	Frequency of Detection	Ecological Risk Screening					Retained for Food Chain Modeling?
							Screening Level	Source of Screening Level	Ecological Effects Quotient <sup>(4)</sup>	Retained as COPC (yes/no)	Rationale	
BETA-BHC	2.7 J	2.7 J	T5-A	4.4	2.7	1/38	2.37	TEC <sup>(6)</sup>	NA	Yes	ASL	Yes
DELTA-BHC	4.2 J	4.2 J	T5-A	4.4	4.2	1/38	2.37	TEC <sup>(6)</sup>	NA	Yes	ASL	Yes
DIELDRIN	8.4 J	26	T6-A	11.7	16.5	2/61	120	SQB	0.22	No	BSL	Yes
ENDOSULFAN I	2.1 J	11 J	T3-A	4.6	5.1	4/38	2.9	USEPA	3.79	Yes	ASL	Yes
ENDOSULFAN II	6.9 J	31 J	T2-A	9.6	12	2/36	14	USEPA	2.21	Yes	ASL	Yes
ENDOSULFAN SULFATE	6.9 J	14 J	T3-A	8.7	10	2/37	5.4	USEPA	2.59	Yes	ASL	Yes
ENDRIN	7.8 J	16 J	T5-A	9.0	12	2/38	54	SQB	0.30	No	BSL	Yes
ENDRIN ALDEHYDE	5.6 J	16 J	T3-B, T2-A	6.2	10	4/28	54	SQB <sup>(7)</sup>	0.30	No	BSL	Yes
ENDRIN KETONE	20 J	20 J	T5-A	9.2	20	1/37	54	SQB <sup>(7)</sup>	0.37	No	BSL	Yes
GAMMA-BHC (LINDANE)	3.5 J	3.5 J	T5-A	4.4	3.5	1/38	2.37	TEC	NA	Yes	ASL	Yes
GAMMA-CHLORDANE	3.7 J	23 J	T6-A-D	24.1	8.8	11/38	3.24	TEC	7.1	Yes	ASL	Yes
HEPTACHLOR	2.8 J	4.5 J	T5-A	4.5	3.5	3/38	2.47	TEC <sup>(6)</sup>	1.82	Yes	ASL	Yes
HEPTACHLOR EPOXIDE	2.2 J	4.5	T1-B	4.6	3.5	4/38	2.47	TEC	1.82	Yes	ASL	Yes
METHOXYCHLOR	38 J	38 J	T5-A	44.6	38.0	1/37	19	USEPA	2.00	Yes	ASL	Yes
TOTAL DDT <sup>(6)</sup>	4.4	8420	2WSD25 (0.0-1.0)	341	928	25/68	5.28	TEC	1595	Yes	ASL	No <sup>(9)</sup>
Inorganics (mg/kg)												
ALUMINUM	2690	27100 J	T7-B	12223	12223	29/29	25500	NOAA	1.06	Yes	ASL	No
ANTIMONY	0.48 J	1.2 J	T7-A	0.43	0.82	4/19	3	NOAA	0.4	No	BSL	No
ARSENIC	1 J	14.1	T8-B	6.55	6.55	29/29	9.79	TEC	1.44	Yes	ASL	Yes
BARIUM	11.9	318 J	T7-B	67.5	67.5	29/29	48	NOAA	6.6	Yes	ASL	No
BERYLLIUM	0.14 J	4.1 J	T7-B	0.70	0.70	29/29	NA	NA	NA	Yes	NTX	No
BORON	14.7	39.6	T9-B	8.35	30.4	5/19	NA	NA	NA	Yes	NTX	No
CADMIUM	0.12 J	6.1	112690-2WSD9(0.0-0.5)	1.7	1.8	26/29	0.99	TEC	6.16	Yes	ASL	Yes
CALCIUM	868	6800	112690-2WSD8(0.0-0.5)	2525	2525	29/29	NA	NA	NA	No	NUT	No
CHROMIUM	7	96.8	T8-B	43.9	43.9	29/29	43.4	TEC	2.23	Yes	ASL	Yes
COBALT	3	13.6 J	T7-B	7.29	7.29	29/29	50	OMOE	0.27	No	BSL	No
COPPER	14.7	173 J	T7-B	53.6	53.6	29/29	31.6	TEC	5.47	Yes	ASL	Yes
CYANIDE	0.9 J	6.1 J	T7-B	1.3	2.4	7/23	NA	NA	NA	Yes	NTX	No
IRON	5630	198000 J	T7-B	27804	27804	29/29	20000	OMOE	9.9	Yes	ASL	No
LEAD	16.1	241 J	112690-2WSD9(0.0-0.5)	58.9	58.9	29/29	35.8	TEC	6.73	Yes	ASL	Yes
MAGNESIUM	1570	9150	T8-B	4757	4757	29/29	NA	NA	NA	No	NUT	No
MANGANESE	55.3	366	112690-2WSD1(0.0-0.5)-D	214	214	29/29	460	OMOE	0.80	No	BSL	No
MERCURY	0.15 J	1.2 J	T6-B	0.30	0.36	15/22	0.18	TEC	6.67	Yes	ASL	Yes
NICKEL	6.6 J	61.5 J	T2-A	20.2	20	29/29	22.7	TEC	2.71	Yes	ASL	Yes
POTASSIUM	659	5170 J	T8-B	2749	2749	29/29	NA	NA	NA	No	NUT	No
SELENIUM	0.79	6.8 J	T7-B	1.2	1.8	16/29	1	NOAA	6.8	Yes	ASL	Yes
SILVER	0.17 J	0.96	T6-A	0.61	0.54	9/29	0.5	OMOE	1.9	Yes	ASL	Yes
SODIUM	114	6650	112690-2WSD8(0.0-0.5)	1723	1723	29/29	NA	NA	NA	No	NUT	No
VANADIUM	8.9 J	203 J	T7-B	55	55	29/29	57	NOAA	3.56	Yes	ASL	No
ZINC	35 J	702 J	T7-B	127	127	29/29	121	TEC	5.80	Yes	ASL	Yes
Miscellaneous Parameters (mg/kg)												
TOTAL ORGANIC CARBON	8420	91000	2WSD24 (0.0-1.0)	41848	41848	25/25	NA	NA	NA	No	NA	No

TABLE 5-2

OCCURENCE, DISTRIBUTION, AND SELECTION OF ECOLOGICAL CHEMICALS OF POTENTIAL CONCERN - SEDIMENT (PHASE I AND II RIs AND FFS)  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT  
 PAGE 3 OF 3

Chemical	Minimum Concentration <sup>(1)</sup>	Maximum Concentration <sup>(1)</sup>	Sample of Maximum Concentration	Average of All Samples <sup>(2)</sup>	Average of Positive Detections <sup>(3)</sup>	Frequency of Detection	Ecological Risk Screening					Retained for Food Chain Modeling?
							Screening Level	Source of Screening Level	Ecological Effects Quotient <sup>(4)</sup>	Retained as COPC (yes/no)	Rationale	

Shaded screening level indicates that the maximum detected concentration exceeds the screening level, shaded chemical name indicates that chemical is retained as a COPC or retained for food chain modeling

**Footnotes**

- 1 - Sample and duplicate are considered as two separate samples when determining the minimum and maximum concentrations detected and as one sample when determining the frequency of detection.
- 2 - Average of all analytical results are calculated using half of the detection limit for nondetects.
- 3 - Average of positive analytical results only.
- 4 - The ecological effects quotient is the maximum detected concentration divided by the screening level.
- 5 - Values are based on positive detections only
- 6 - Used gamma-BHC as a surrogate
- 7 - Used endrin as a surrogate
- 8 - Used heptachlor epoxide as a surrogate.
- 9 - Although these parameters are considered bioaccumulative, they are not included in the food chain model because the food chain model is conducted on the individual chemicals that make up the totals

Rationale Codes

For Selection as a COPC or for Further Evaluation:

- ASL = Above COPC Screening Level
- BSL = Below COPC Screening Level
- NTX = No Toxicity Data Available/Screening Level not Available
- NUT = Essential Nutrient

COPC = Chemical of Potential Concern

SCV = Secondary Chronic Value

SQB - Sediment Quality Benchmark

USEPA = United States Environmental Protection Agency

TEC = Threshold Effect Concentrations

NOAA = National Oceanic & Atmospheric Administration

OMOE = Ontario Ministry of Environment and Energy

NA = Not Available or Not Applicable

Sources in Order of Preference:

1. SQB - USEPA, 2003. Procedures for the Derivation of Equilibrium Partitioning Sediment Benchmarks (ESBs) for the Protection of Benthic Organisms, August (Dieldrin, EPA/600/R-02/010, Endrin, EPA/600/R-02/009)
2. TEC - MacDonald, D.D., C.G. Ingersoll, and T.A. Berger. 2000. Development and Evaluation of Consensus-Based Sediment Quality Guidelines for Freshwater Ecosystems. Archives of Environmental Contamination and Toxicology. Vol 39, pp. 20-31.
3. OMOE - OMOE 1993. Guidelines for the Protection and Management of Aquatic Sediment Quality in Ontario. Ministry of Environment and Energy. August.
4. USEPA - USEPA, 1996. ECO Update, Ecotox Thresholds. U.S. Environmental Protection Agency. Office of Solid Waste and Emergency Response. Intermittent Bulletin, Volume 3, Number 2. EPA540/F-95/038. January
5. NOAA - Buchman, M.F., 1999. NOAA Screening Quick Reference Tables, NOAA HAZMAT Report 99-1, Seattle, WA, Coastal Protection and Restoration Division, National Oceanic and Atmospheric Administration. <http://response.restoration.noaa.gov/cpr/sediment/squrt.html>
6. SCV - Jones, D.S., G.W. Suter II, and R.N. Hull. 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Sediment-Associated Biota 1997 Revision. Oak Ridge National Laboratory. ES/ER/TM-95/R4. November

TABLE 5-3

OCCURRENCE, DISTRIBUTION, AND SELECTION OF  
ECOLOGICAL CHEMICALS OF POTENTIAL CONCERN - SURFACE WATER  
SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT  
PAGE 1 OF 2

Chemical	Minimum Concentration <sup>(1)</sup>	Maximum Concentration <sup>(1)</sup>	Sample of Maximum Concentration	Average of All Samples <sup>(2)</sup>	Average of Positive Detections <sup>(3)</sup>	Frequency of Detection	Ecological Risk Screening				
							Screening Level	Source of Screening Level	Ecological Effects Quotient <sup>(4)</sup>	Retained as COPC (yes/no)	Rationale
<b>Volatile Organics (ug/L)</b>											
TETRACHLOROETHENE	2 J	2 J	2WSW12	5	2	1/9	98	ORNL	0.02	No	BSL
<b>Total Inorganics (ug/L)</b>											
ALUMINUM	66.9 J	20900	2WSW1	4875	5477	8/9	<b>87</b>	USEPA	240	Yes	ASL
ARSENIC	2.8	2.9	2WSW1	1.6	2.8	3/9	150	CTDEP	0.02	No	BSL
BARIUM	97.6	115	2WSW2	37.7	106	2/9	<b>4</b>	ORNL	29	Yes	ASL
BORON	121	369	2WSW2	135	222	5/9	NA	NA	NA	Yes	NTX
CALCIUM	272 J	46500	2WSW12	20988	20988	9/9	NA	NA	NA	No	NUT
COBALT	63.9	66.8	2WSW6	16.5	65.4	2/9	<b>23</b>	ORNL	2.9	Yes	ASL
COPPER	6.1 J	29.3 J	2WSW1	6.34	14.4	3/9	<b>4.8</b>	CTDEP	6.1	Yes	ASL
IRON	1070 J	11300	2WSW12	4737	7103	6/9	<b>1000</b>	USEPA	11	Yes	ASL
LEAD	2 J	5 J	2WSW6	3	3	4/5	<b>1.2</b>	CTDEP	4.2	Yes	ASL
MAGNESIUM	443	18500	2WSW1	8031	9029	8/9	NA	NA	NA	No	NUT
MANGANESE	43.7 J	1870	2WSW6	622	931	6/9	<b>120</b>	ST	16	Yes	ASL
MERCURY	0.21 J	0.21 J	2WSW9	0.12	0.21	1/7	0.77	CTDEP	0.27	No	BSL
NICKEL	11.1 J	84.7	2WSW1	23.4	59.3	3/9	<b>23.9</b>	CTDEP	2.93	Yes	ASL
POTASSIUM	447 J	15300	2WSW2	5926	5926	9/9	NA	NA	NA	No	NUT
SODIUM	19700	143000	2WSW2	47877	61357	7/9	NA	NA	NA	No	NUT
VANADIUM	4.3 J	4.3 J	2WSW10	1.8	4.3	1/9	20	ORNL	0.22	No	BSL
ZINC	47.6	334	2WSW6	87.1	190	4/9	<b>65</b>	CTDEP	5.14	Yes	ASL
<b>Filtered Inorganics (ug/L)</b>											
ALUMINUM	86.3	259	2WSW6	127	138	5/6	<b>87</b>	USEPA	2.98	Yes	ASL
BARIUM	83.3	91.2	2WSW2	35.6	87.3	2/6	<b>4</b>	ORNL	23	Yes	ASL
BORON	75.9 J	222	2WSW6	96.3	132.0	4/6	NA	NA	NA	Yes	NTX
CALCIUM	256 J	45300	2WSW12	20319	20319	6/6	NA	NA	NA	No	NUT
COPPER	4.5 J	4.5 J	2WSW7	1.9	4.5	1/6	4.8	CTDEP	0.94	No	BSL
IRON	110	19400	2WSW12	3988	3988	6/6	<b>1000</b>	USEPA	19.4	Yes	ASL
LEAD	1.8 J	6.1 J	2WSW7	1.9	4.0	2/5	<b>1.2</b>	CTDEP	5.08	Yes	ASL
MAGNESIUM	103 J	10800	2WSW2	5567	5567	6/6	NA	NA	NA	No	NUT
MANGANESE	9.6 J	571	2WSW12	275	275	6/6	<b>120</b>	ORNL	4.76	Yes	ASL
MERCURY	0.22 J	0.22 J	2WSW12	0.12	0.22	1/6	0.77	CTDEP	0.29	No	BSL
POTASSIUM	542 J	14700	2WSW2	6329	6329	6/6	NA	NA	NA	No	NUT
SODIUM	32300	133000	2WSW2	64277	77060	5/6	NA	NA	NA	No	NUT
VANADIUM	3.3 J	3.3 J	2WSW7	1.8	3.3	1/6	20	ORNL	0.17	No	BSL
ZINC	27.7	31.4	2WSW1	18.0	29.6	3/6	65	CTDEP	0.48	No	BSL
<b>Miscellaneous Parameters (mg/L)</b>											
HARDNESS	16	160	2WSW12	77	87	8/9	NA	NA	NA	NA	NA

Shaded criterion indicates that the maximum detected concentration exceeds one or more screening criteria and it indicates that the shaded chemical was retained as a COPC.

TABLE 5-3

OCCURRENCE, DISTRIBUTION, AND SELECTION OF  
 ECOLOGICAL CHEMICALS OF POTENTIAL CONCERN - SURFACE WATER  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT  
 PAGE 2 OF 2

Chemical	Minimum Concentration <sup>(1)</sup>	Maximum Concentration <sup>(1)</sup>	Sample of Maximum Concentration	Average of All Samples <sup>(2)</sup>	Average of Positive Detections <sup>(3)</sup>	Frequency of Detection	Ecological Risk Screening				
							Screening Level	Source of Screening Level	Ecological Effects Quotient <sup>(4)</sup>	Retained as COPC (yes/no)	Rationale

**Footnotes**

- 1 - Sample and duplicate are considered as two separate samples when determining the minimum and maximum concentrations detected concentrations and as one sample when determining the frequency of detection.
- 2 - Average of all analytical results are calculated using half of the detection limit for nondetects.
- 3 - Average of positive analytical results only.
- 4 - The ecological effects quotient is the maximum detected concentration divided by the screening level.

COPC= Chemical of potential concern

USEPA= United States Environmental Protection Agency Water Quality Criteria (USEPA, 2006)

CTDEP= Connecticut Department of Environmental Protection Water Quality Standard (CTDEP, 2002)

ORNL - Oak Ridge National Laboratory Benchmark (Suter and TSAO, 1996)

NA= Not Available or Not Applicable

Rationale Codes.

For Selection as a COPC or for Further Evaluation:

ASL = Above COPC Screening Level

BSL = Below COPC Screening Level

NTX = No Toxicity Data Available/Screening Level not Available

NUT = Essential Nutrient

TABLE 5-4

OCCURRENCE, DISTRIBUTION, AND SELECTION OF  
 ECOLOGICAL CHEMICALS OF POTENTIAL CONCERN - SURFACE WATER (AREA A LANDFILL SAMPLES)  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 1 OF 2

Chemical	Minimum Concentration <sup>(1)</sup>	Maximum Concentration <sup>(1)</sup>	Sample of Maximum Concentration	Average of All Samples <sup>(2)</sup>	Average of Positive Detections <sup>(3)</sup>	Frequency of Detection	Ecological Risk Screening					
							Screening Level	Source of Screening Level	Ecological Effects Quotient <sup>(4)</sup>	Retained as COPC (yes/no)	Rationale	
<b>Semivolatile Organics (ug/L)</b>												
BIS(2-ETHYLHEXYL)PHTHALATE	10 J	11 J	2-SW23-04-20051208	5.08	10.5	2/22	3	ORNL	3.7	Yes	ASL	
<b>Polyaromatic Hydrocarbons (ug/L)</b>												
2-METHYLNAPHTHALENE	0.19 J	1.6	2-SW19-03-20060829	0.18	0.65	3/22	72.16	USEPA FCV	0.02	No	BSL	
ACENAPHTHENE	0.21 V	3.8	2-SW19-03-20060829	0.32	1.06	5/22	55.85	USEPA FCV	0.1	No	BSL	
ACENAPHTHYLENE	0.2 J	0.2 J	2-SW19-03-20060829-D	0.10	0.15	1/22	306.9	USEPA FCV	0.001	No	BSL	
ANTHRACENE	0.2 J	0.21 J	2-SW19-04-20061212-D	0.11	0.19	3/22	20.73	USEPA FCV	0.0	No	BSL	
BENZO(A)ANTHRACENE	0.2 J	0.28 V	2-SW24-04-20051208	0.14	0.21	8/22	2.227	USEPA FCV	0.1	No	BSL	
BENZO(A)PYRENE	0.2 J	0.42 V	2-SW24-04-20051208	0.13	0.26	4/22	0.9573	USEPA FCV	0.4	No	BSL	
BENZO(B)FLUORANTHENE	0.2 J	0.78 V	2-SW24-04-20051208	0.15	0.35	4/22	0.6774	USEPA FCV	1.2	Yes	ASL	
BENZO(G,H,I)PERYLENE	0.25 J	0.37 V	2-SW24-04-20051208	0.15	0.27	4/22	0.4391	USEPA FCV	0.8	No	BSL	
BENZO(K)FLUORANTHENE	0.2 J	0.51 V	2-SW24-04-20051208	0.14	0.25	6/22	0.6415	USEPA FCV	0.8	No	BSL	
CHRYSENE	0.2 J	0.54 V	2-SW24-04-20051208	0.15	0.25	7/22	2.042	USEPA FCV	0.3	No	BSL	
DIBENZO(A,H)ANTHRACENE	0.2 J	0.22 J	2-SW19-04-20051207	0.11	0.18	2/22	0.2825	USEPA FCV	0.8	No	BSL	
FLUORANTHENE	0.2 J	1.1 V	2-SW24-04-20051208	0.21	0.30	12/22	7.109	USEPA FCV	0.2	No	BSL	
FLUORENE	0.2 J	2.1	2-SW19-03-20060829	0.21	0.59	5/22	39.3	USEPA FCV	0.1	No	BSL	
INDENO(1,2,3-CD)PYRENE	0.2 J	0.37 V	2-SW24-04-20051208	0.13	0.24	4/22	0.275	USEPA FCV	1.3	Yes	ASL	
NAPHTHALENE	0.21 J	6	2-SW19-03-20060829	0.37	3.06	2/22	2.43	USEPA FCV	2.5	Yes	ASL	
PHENANTHRENE	0.2 J	1.3	2-SW19-03-20060829	0.21	0.32	11/22	19.13	USEPA FCV	0.1	No	BSL	
PYRENE	0.19 J	0.86 V	2-SW24-04-20051208	0.18	0.28	10/22	10.11	USEPA FCV	0.1	No	BSL	
<b>Total Inorganics (ug/L)</b>												
ALUMINIUM	100 J	20000 V	2-SW24-04-20051208	1980	1980	22/22	87	USEPA WQC	230	Yes	ASL	
ANTIMONY	1 J	7	2-SW22-02-20050601	1.3	1.3	22/22	30	ORNL	0.2	No	BSL	
ARSENIC	1 J	127.4 V	2-SW24-04-20051208	12	12	22/22	150	CTDEP	0.8	No	BSL	
BARIUM	18	460 V	2-SW24-04-20051208	80	80	22/22	4	ST	115	Yes	ASL	
BERYLLIUM	5 J	5 J	2-SW18-04-20051207	2.6	5.0	6/22	0.66	ORNL	7.6	Yes	ASL	
CADMIUM	0.2 J	3.5	2-SW22-02-20050601	0.4	0.7	11/22	1.35	CTDEP	3	Yes	ASL	
CALCIUM	100 J	40000	2-SW19-03-20060829-D	13011	13011	22/22	NA	NA	NA	No	NUT	
CHROMIUM	1 J	38.8	2-SW22-02-20050601	5.0	6.3	17/22	11	CTDEP	4	Yes	ASL	
COBALT	1 J	52.6 V	2-SW24-04-20051208	4.5	4.5	22/22	23	ORNL	2.3	Yes	ASL	
COPPER	1 J	270.6	2-SW22-02-20050601	26	26	22/22	4.8	CTDEP	56	Yes	ASL	
IRON	50 J	350000 V	2-SW24-04-20051208	44126	44126	22/22	1000	USEPA WQC	350	Yes	ASL	
LEAD	0.5 J	130.8	2-SW22-02-20050601	14	14	22/22	1.2	CTDEP	109	Yes	ASL	
MAGNESIUM	1500	13000 V	2-SW24-04-20051208	3875	3875	22/22	NA	NA	NA	No	NUT	
MANGANESE	30	3600 V	2-SW24-04-20051208	401	401	22/22	120	ORNL	30	Yes	ASL	
MERCURY	0.2 J	0.5 J	2-SW22-02-20050601	0.2	0.3	8/22	0.77	CTDEP	0.6	No	BSL	
MOLYBDENUM	2 J	10.2 V	2-SW24-04-20051208	2.2	3.3	13/22	370	ORNL	0.03	No	BSL	
NICKEL	1 J	98.1	2-SW22-02-20050601	12	12	22/22	23.9	CTDEP	3.4	Yes	ASL	
POTASSIUM	2500 J	14000 V	2-SW24-04-20051208	4066	4854	17/22	NA	NA	NA	No	NUT	
SELENIUM	1 J	7	2-SW22-02-20050601	1.7	1.8	20/22	5	CTDEP	1.4	Yes	ASL	
SILVER	1 J	1 J	2-SW18-04-20051207	0.7	1.0	9/22	1.02	CTDEP	0.98	No	BSL	
SODIUM	2000 J	96000	2-SW19-03-20060829-D	44098	44098	22/22	NA	NA	NA	No	NUT	
THALLIUM	1 J	1 J	2-SW18-04-20051207	0.6	1.0	6/22	12	ORNL	0.1	No	BSL	
VANADIUM	1 J	83.2 V	2-SW24-04-20051208	14	14	22/22	20	ORNL	4	Yes	ASL	
ZINC	7	3346	2-SW22-02-20050601	347	347	22/22	65	CTDEP	51	Yes	ASL	

OCCURRENCE, DISTRIBUTION, AND SELECTION OF  
 ECOLOGICAL CHEMICALS OF POTENTIAL CONCERN - SURFACE WATER (AREA A LANDFILL SAMPLES)  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 2 OF 2

Chemical	Minimum Concentration <sup>(1)</sup>	Maximum Concentration <sup>(1)</sup>	Sample of Maximum Concentration	Average of All Samples <sup>(2)</sup>	Average of Positive Detections <sup>(3)</sup>	Frequency of Detection	Ecological Risk Screening						
							Screening Level	Source of Screening Level	Ecological Effects Quotient <sup>(4)</sup>	Retained as COPC (yes/no)	Rationale		
<b>Filtered Inorganics (ug/L)</b>													
<b>ALUMINUM</b>	100 J	100 J	2-SW18-04-20051207	66	100	7/22	<b>87</b>	USEPA WQC	1.1	Yes	ASL		
ANTIMONY	1 J	1 J	2-SW18-02-20050601	0.8	1.0	14/22	30	ORNL	0.03	No	BSL		
ARSENIC	1 J	3.3	2-SW23-03-20060830	1.1	1.1	21/22	150	CTDEP	0.02	No	BSL		
<b>BARIIUM</b>	14	100	2-SW19-03-20060829	33	33	22/22	<b>4</b>	ORNL	25	Yes	ASL		
<b>BERYLLIUM</b>	5 J	5 J	2-SW19-04-20051207-D	2.3	4.8	5/22	<b>0.66</b>	ORNL	7.6	Yes	ASL		
CADMIUM	0.2 J	0.2 J	2-SW19-04-20051207	0.1	0.2	4/22	1.35	CTDEP	0.1	No	BSL		
CALCIUM	5300	38000	2-SW19-03-20060829	12159	12159	22/22	NA	NA	NA	No	NUT		
CHROMIUM	1 J	1 V	2-SW18-04-20051207	0.8	1.0	12/22	11	CTDEP	0.1	No	BSL		
COBALT	1 J	16.1 V	2-SW24-04-20051208	1.9	2.2	19/22	23	ORNL	0.7	No	BSL		
<b>COPPER</b>	1 J	62.8	2-SW23-03-20060830	3.8	4.0	21/22	<b>4.8</b>	CTDEP	13	Yes	ASL		
<b>IRON</b>	50 J	32000 V	2-SW24-04-20051208	3192	3343	21/22	<b>1000</b>	USEPA WQC	32	Yes	ASL		
LEAD	0.5 J	1	2-SW20-03-20060824	0.4	0.5	15/22	1.2	CTDEP	0.8	No	BSL		
MAGNESIUM	1100	8400	2-SW24-02-20050602	3102	3102	22/22	NA	NA	NA	No	NUT		
<b>MANGANESE</b>	20	2200 V	2-SW24-04-20051208	235	235	22/22	<b>120</b>	ORNL	18	Yes	ASL		
MERCURY	0.2 J	0.2 J	2-SW19-04-20061212	0.1	0.2	4/22	0.77	CTDEP	0.3	No	BSL		
MOLYBDENUM	1.2	2 J	2-SW18-04-20051207	1.1	1.9	6/22	370	ORNL	0.01	No	BSL		
NICKEL	1 J	8.3 V	2-SW24-04-20051208	2.1	2.1	22/22	28.9	CTDEP	0.3	No	BSL		
POTASSIUM	2500 J	8800 V	2-SW24-04-20051208	3172	3872	16/22	NA	NA	NA	No	NUT		
SELENIUM	1 J	2 J	2-SW18-02-20050601	1.0	1.5	11/22	5	CTDEP	0.4	No	BSL		
SILVER	1 J	1 J	2-SW18-02-20050601	0.7	1.0	8/22	1.02	CTDEP	0.98	No	BSL		
SODIUM	12000	89000	2-SW19-03-20060829	43159	43159	22/22	NA	NA	NA	No	NUT		
THALLIUM	1 J	1 J	2-SW24-04-20051208	0.5	1.0	1/22	12	ORNL	0.1	No	BSL		
VANADIUM	1 J	1 J	2-SW18-02-20050601	0.8	1.0	15/22	20	ORNL	0.1	No	BSL		
<b>ZINC</b>	5.6	405.3	2-SW23-03-20060830	48	58	18/22	<b>65</b>	CTDEP	6.2	Yes	ASL		
<b>Miscellaneous Parameters (mg/L)</b>													
HARDNESS	21	130	2-SW19-03-20060829	51	51	22/22	NA	NA	NA	NA	NA		

## Footnotes

- 1 - Sample and duplicate are considered as two separate samples when determining the minimum and maximum concentrations detected and as one sample when determining the frequency of detection  
 2 - Average of all analytical results are calculated using half of the detection limit for nondetects  
 3 - Average of positive analytical results only  
 4 - The same maximum concentration occurred for multiple samples.

COPC= Chemical of potential concern

USEPA FCV= United States Environmental Protection Agency Final Chronic Value (USEPA, 2003)

USEPA WQC= United States Environmental Protection Agency Water Quality Criteria (USEPA, 2006)

CTDEP= Connecticut Department of Environmental Protection Water Quality Standard (CTDEP, 2002)

ORNL - Oak Ridge National Laboratory Benchmark (Suter and TSAO, 1996)

NA= Not Available or Not Applicable

Rationale Codes

For Selection as a COPC or for Further Evaluation

ASL = Above COPC Screening Level

BSL = Below COPC Screening Level

NTX = No Toxicity Data Available/Screening Level not Available

NUT = Essential Nutrient

Shaded criterion indicates that the maximum detected concentration exceeds one or more screening criteria and it indicates that the shaded chemical was retained as a COPC

TABLE 5-5

EXPOSURE PARAMETERS FOR THE TERRESTRIAL WILDLIFE MODEL  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT  
 PAGE 1 OF 2

Species/Exposure Inputs	Conservative Inputs		Average Inputs	
	Values	Units	Values	Units
<b>Bobwhite Quail</b>				
Body Weight = BW	1.540E-01	kg	1.751E-01	kg
Food Ingestion Rate = If	4.880E-03	kg/day	4.080E-03	kg/day
Water Ingestion Rate = lw	2.276E-02	L/day	1.926E-02	L/day
Soil Ingestion Rate = Is	6.783E-04	kg/day	2.489E-04	kg/day
Home Range = HR	Assume 100% on site		1.880E+01	acres
<b>Meadow Vole</b>				
Body Weight = BW	1.700E-02	kg	3.580E-02	kg
Food Ingestion Rate = If	3.756E-03	kg/day	3.488E-03	kg/day
Water Ingestion Rate = lw	7.513E-03	L/day	6.261E-03	L/day
Soil Ingestion Rate = Is	1.202E-04	kg/day	4.186E-05	kg/day
Home Range = HR	Assume 100% on site		6.590E-02	acres
<b>American Robin</b>				
Body Weight = BW	7.73E-02	kg	8.04E-02	kg
Food Ingestion Rate = If	1.25E-02	kg/day	1.19E-02	kg/day
Water Ingestion Rate = lw	1.21E-02	L/day	1.13E-02	L/day
Soil Ingestion Rate - Is	2.046E-03	kg/day	7.601E-04	kg/day
Home Range = HR	Assume 100% on site		6.100E-01	acres
<b>Short-Tailed Shrew</b>				
Body Weight = BW	1.500E-02	kg	1.610E-02	kg
Food Ingestion Rate = If	1.600E-03	kg/day	1.433E-03	kg/day
Water Ingestion Rate = lw	4.280E-03	L/day	3.600E-03	L/day
Soil Ingestion Rate - Is	4.801E-05	kg/day	1.289E-05	kg/day
Home Range = HR	Assume 100% on site		9.699E-01	acres
<b>Raccoon</b>				
Body Weight = BW	3.670E+00	kg	5.636E+00	kg
Food Ingestion Rate = If	2.370E-01	kg/day	1.840E-01	kg/day
Water Ingestion Rate = lw	4.680E-01	L/day	4.650E-01	L/day
Sediment Ingestion Rate = Is	2.228E-02	kg/day	1.730E-02	kg/day
Home Range = HR	Assume 100% on site		1.558E+03	acres
<b>Mallard Duck</b>				
Body Weight = BW	1.04E+00	kg	1.17E+00	kg
Food Ingestion Rate = If	8.20E-02	kg/day	7.83E-02	kg/day
Water Ingestion Rate = lw	6.76E-01	L/day	6.58E-02	L/day
Sediment Ingestion Rate = Is	2.706E-03	kg/day	2.583E-03	kg/day
Home Range = HR	Assume 100% on site		1.433E+03	km-radius

TABLE 5-5

EXPOSURE PARAMETERS FOR THE TERRESTRIAL WILDLIFE MODEL  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT  
 PAGE 2 OF 2

**Notes:**

The exposure factors were derived as presented in Attachment 2.

The soil/sediment ingestion rates were calculated by multiplying the food ingestion rates by the following incidental soil/sediment ingestion rates:

	Conservative	Average	Source
Bobwhite Quail	13.90%	6.10%	1, 2
Meadow Vole	3.20%	1.20%	1
American Robin	16.40%	6.40%	1, 4
Short-tailed Shrew	3%	0.90%	1
Raccoon	9.4%	9.4%	3
Mallard Duck	3.3%	3.3%	3

1 - U.S. Environmental Protection Agency, 2005. Ecological Soil Screening Level Guidance, Office of Emergency and Remedial Response. February.

2 - Based on the mourning dove.

3 - Beyer, N., E. Connor, and S. Gerould. 1994. Estimates of Soil Ingestion by Wildlife. Journal of Wildlife Management 58(2) pp. 375-382.

4 - Based on the American woodcock

TABLE 5-6

**TERRESTRIAL FOOD CHAIN MODEL - CONSERVATIVE SCENARIO  
SOIL INVERTIVOROUS AND HERBIVOROUS RECEPTORS  
SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT**

Chemical	Herbivorous Receptors EEQs				Soil Invertivorous Receptors EEQs			
	Bobwhite Quail		Meadow Vole		American Robin		Short-Tailed Shrew	
	NOAEL	LOAEL	NOAEL	LOAEL	NOAEL	LOAEL	NOAEL	LOAEL
<b>Semivolatile Organics</b>								
ACENAPHTHENE	7.8E-04	7.8E-05	2.6E-04	1.3E-04	1.8E-02	1.8E-03	1.2E-03	5.8E-04
ACENAPHTHYLENE	1.2E-03	1.2E-04	2.0E-04	2.0E-05	8.1E-03	8.1E-04	1.3E-04	1.3E-05
ANTHRACENE	9.9E-04	9.9E-05	1.2E-04	1.2E-05	5.3E-03	5.3E-04	5.9E-05	5.9E-06
BENZO(A)ANTHRACENE	1.5E-03	1.5E-04	6.7E-02	6.7E-03	2.7E-02	2.7E-03	1.8E-01	1.8E-02
BENZO(A)PYRENE	1.7E-03	1.7E-04	1.4E-02	1.4E-03	2.6E-02	2.6E-03	2.9E-02	2.9E-03
BENZO(B)FLUORANTHENE	3.9E-03	3.9E-04	1.0E-02	1.0E-03	3.7E-02	3.7E-03	1.0E-02	1.0E-03
BENZO(G,H,I)PERYLENE	1.8E-03	1.8E-04	2.7E-03	2.7E-04	1.7E-02	1.7E-03	2.7E-03	2.7E-04
BENZO(K)FLUORANTHENE	1.7E-03	1.7E-04	2.0E-03	2.0E-04	2.6E-02	2.6E-03	4.0E-03	4.0E-04
CHRYSENE	2.1E-03	2.1E-04	8.9E-02	8.9E-03	4.0E-02	4.0E-03	2.6E-01	2.6E-02
DIBENZO(A,H)ANTHRACENE	1.4E-04	1.4E-05	8.6E-04	8.6E-05	2.2E-03	2.2E-04	1.8E-03	1.8E-04
FLUORANTHENE	9.0E-03	9.0E-04	8.4E-03	4.2E-03	6.0E-02	6.0E-03	5.3E-03	2.7E-03
INDENO(1,2,3-CD)PYRENE	1.1E-03	1.1E-04	1.2E-03	1.2E-04	1.8E-02	1.8E-03	2.8E-03	2.8E-04
PHENANTHRENE	7.6E-03	7.6E-04	9.8E-02	9.8E-03	2.3E-02	2.3E-03	2.5E-02	2.5E-03
PYRENE	1.1E-02	1.1E-03	1.8E-02	1.1E-02	5.4E-02	5.4E-03	8.1E-03	4.8E-03
<b>Pesticides/PCBs</b>								
4,4'-DDD	2.9E-03	2.3E-03	2.0E-02	1.1E-02	3.6E-01	2.9E-01	3.6E-01	1.9E-01
4,4'-DDE	5.5E-04	4.4E-04	4.3E-03	2.3E-03	1.5E-01	1.2E-01	1.5E-01	8.1E-02
4,4'-DDT	1.3E-03	1.1E-03	9.6E-03	5.2E-03	2.7E-01	2.2E-01	2.7E-01	1.5E-01
ALPHA-CHLORDANE	7.3E-06	1.5E-06	8.2E-06	4.1E-06	1.2E-03	2.3E-04	3.5E-04	1.8E-04
AROCLOR-1260	9.2E-03	9.2E-04	4.2E-02	4.2E-03	5.3E+00	5.3E-01	9.3E+00	9.3E-01
GAMMA-CHLORDANE	5.3E-06	1.1E-06	6.0E-06	3.0E-06	8.6E-04	1.7E-04	2.6E-04	1.3E-04
<b>Inorganics</b>								
CADMIUM	7.4E-02	1.7E-02	6.6E-01	7.4E-02	4.5E+00	1.0E+00	5.6E+00	6.2E-01
CHROMIUM	2.2E-01	3.7E-02	6.9E-01	2.8E-02	2.9E+00	5.0E-01	1.5E+00	6.3E-02
COPPER	1.5E-01	1.7E-02	4.6E-01	3.3E-02	1.7E+00	2.0E-01	6.4E-01	4.6E-02
LEAD	4.2E-01	1.6E-02	3.8E-01	9.7E-03	6.1E+00	2.2E-01	1.0E+00	2.5E-02
MERCURY	1.8E+01	1.8E+00	2.4E+01	4.8E+00	2.7E+01	2.7E+00	3.3E+00	6.5E-01
SELENIUM	1.3E-01	6.6E-02	1.6E+00	9.5E-01	8.7E-01	4.3E-01	9.8E-01	5.9E-01
SILVER	1.1E-02	3.6E-04	7.6E-03	3.9E-04	7.9E-01	2.7E-02	1.7E-01	8.4E-03
VANADIUM	1.0E+00	2.0E-01	1.5E-01	7.9E-02	7.3E+00	1.5E+00	1.4E-01	7.5E-02
ZINC	1.9E-01	2.2E-02	1.0E-01	5.2E-02	4.9E+00	5.4E-01	2.8E-01	1.4E-01

Cells are shaded if the EEQ is greater than 1.0

NOAEL - No Observed Adverse Effects Level  
LOAEL - Lowest Observed Adverse Effects Level  
EEQ - Ecological Effects Quotient

TABLE 5-7

TERRESTRIAL FOOD CHAIN MODEL - CONSERVATIVE SCENARIO  
 SEDIMENT INVERTIVOROUS RECEPTORS  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT

Chemical	Sediment Invertivorous Receptors EEQs			
	Mallard Duck		Raccoon	
	NOAEL	LOAEL	NOAEL	LOAEL
<b>Semivolatile Organics</b>				
1,4-DICHLOROBENZENE	NV	NV	2.1E-04	2.1E-05
2-METHYLNAPHTHALENE	4.9E-03	4.9E-04	2.0E-04	2.0E-05
ACENAPHTHENE	1.0E-02	1.0E-03	1.0E-03	5.2E-04
ACENAPHTHYLENE	1.1E-02	1.1E-03	2.7E-04	2.7E-05
ANTHRACENE	6.5E-02	6.5E-03	1.2E-03	1.2E-04
BENZO(A)ANTHRACENE	7.3E-01	7.3E-02	7.7E+00	7.7E-01
BENZO(A)PYRENE	9.4E-01	9.4E-02	1.7E+00	1.7E-01
BENZO(B)FLUORANTHENE	1.5E+00	1.5E-01	6.6E-01	6.6E-02
BENZO(G,H,I)PERYLENE	6.2E-01	6.2E-02	1.5E-01	1.5E-02
BENZO(K)FLUORANTHENE	1.2E+00	1.2E-01	3.0E-01	3.0E-02
CHRYSENE	1.1E+00	1.1E-01	1.2E+01	1.2E+00
DIBENZO(A,H)ANTHRACENE	8.3E-03	8.3E-04	1.1E-02	1.1E-03
FLUORANTHENE	2.2E+00	2.2E-01	3.1E-01	1.5E-01
FLUORENE	2.7E-02	2.7E-03	3.9E-03	1.9E-03
INDENO(1,2,3-CD)PYRENE	6.2E-01	6.2E-02	1.5E-01	1.5E-02
NAPHTHALENE	2.1E-03	2.1E-04	5.2E-04	2.6E-04
PENTACHLOROPHENOL	9.3E-05	1.2E-05	1.7E-04	6.4E-05
PHENANTHRENE	9.7E-01	9.7E-02	1.7E+00	1.7E-01
PYRENE	1.1E+00	1.1E-01	2.7E-01	1.6E-01
<b>Pesticides/PCBs</b>				
4,4'-DDD	1.1E+00	8.9E-01	1.5E+00	8.2E-01
4,4'-DDE	4.3E+00	3.5E+00	5.5E+00	3.0E+00
4,4'-DDT	3.8E+00	3.1E+00	4.9E+00	2.6E+00
ALDRIN	NV	NV	4.3E-03	8.6E-04
ALPHA-CHLORDANE	1.1E-02	2.3E-03	4.4E-03	2.2E-03
AROCLOR-1260	9.4E+01	9.4E+00	2.1E+02	2.1E+01
BETA-BHC	1.5E-03	3.8E-04	1.8E-03	3.6E-04
DELTA-BHC	2.4E-03	6.0E-04	8.0E-02	8.0E-03
DIELDRIN	1.2E-01	1.0E-02	4.6E-01	5.5E-03
ENDOSULFAN I	3.5E-04	3.5E-05	2.0E-02	2.0E-03
ENDOSULFAN II	9.9E-04	9.9E-05	5.5E-02	5.5E-03
ENDOSULFAN SULFATE	4.5E-04	4.5E-05	2.5E-02	2.5E-03
ENDRIN	5.0E-01	5.0E-02	4.7E-02	4.7E-03
ENDRIN ALDEHYDE	5.1E-01	5.1E-02	4.7E-02	4.7E-03
ENDRIN KETONE	6.4E-01	6.4E-02	5.8E-02	5.8E-03
GAMMA-BHC (LINDANE)	5.6E-04	5.6E-05	1.2E-04	1.2E-05
GAMMA-CHLORDANE	4.2E-03	8.5E-04	1.6E-03	8.2E-04
HEPTACHLOR	NV	NV	1.2E-02	1.2E-03
HEPTACHLOR EPOXIDE	NV	NV	1.2E-02	1.2E-03
METHOXYCHLOR	NV	NV	2.5E-03	1.3E-03
<b>Inorganics</b>				
ARSENIC	3.6E-01	1.8E-01	2.9E-01	1.6E-01
CADMIUM	2.7E+00	6.2E-01	4.2E+00	4.6E-01
CHROMIUM	1.4E+00	2.4E-01	1.5E+00	6.0E-02
COPPER	1.8E+01	2.1E+00	1.0E+01	7.3E-01
LEAD	7.4E+00	2.7E-01	2.3E+00	5.9E-02
MERCURY	4.3E+01	4.3E+00	7.2E+00	1.4E+00
NICKEL	1.7E+00	6.2E-01	5.6E+00	6.5E-01
SELENIUM	1.4E+00	6.9E-01	2.4E+00	1.5E+00
SILVER	3.9E-02	1.3E-03	1.1E-02	5.7E-04
ZINC	2.9E+01	3.2E+00	2.2E+00	1.1E+00

Cells are shaded if the value is greater than 1.0

NOAEL - No Observed Adverse Effects Level

LOAEL - Lowest Observed Adverse Effects Level

EEQ - Ecological Effects Quotient

NV - No value could be calculated because toxicity data was not available for this chemical.

TABLE 6-1

ALTERNATE SOIL BENCHMARK COMPARISON  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUTT

Chemical	Maximum Concentration <sup>(1)</sup>	Average of All Samples <sup>(2)</sup>	Lower of Plant or Invertebrate Eco-SSL <sup>(3)</sup>	Canadian SQG Direct Contact <sup>(4)</sup>
<b>Inorganics (mg/kg)</b>				
CADMIUM	7.2	3.8	32	N/A
CHROMIUM	102	58.5	NA	64
COPPER	64.1 J	36.0	70	N/A
LEAD	128 J	42.4	120	N/A
MERCURY	0.69 J	0.25	NA	12
SELENIUM	2.4	0.92	NA	1
SILVER	4.5	1.5	560	N/A
VANADIUM	75 J	50.8	NA	130
ZINC	125 J	70.1	NA	200

NA - Not available

N/A - Not Applicable because an Eco-SSL value was available

**Footnotes**

1 - Sample and duplicate are considered as two separate samples when determining the minimum and maximum concentrations detected and as one sample when determining the frequency of detection.

2 - Average of all analytical results are calculated using half of the detection limit for nondetects.

3 - Eco-SSL - Ecological Soil Screening Level documents for individual chemicals

4 - Canadian SQG - Canadian Soil Quality Gurdeline documents for individual chemicals

TABLE 6-2

TERRESTRIAL FOOD CHAIN MODEL - LESS CONSERVATIVE SCENARIO  
SOIL INVERTIVOROUS AND HERBIVOROUS RECEPTORS  
SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT

Chemical	Herbivorous Receptors EEQs				Soil Invertivorous Receptors EEQs			
	Bobwhite Quail		Meadow Vole		American Robin		Short-Tailed Shrew	
	NOAEL	LOAEL	NOAEL	LOAEL	NOAEL	LOAEL	NOAEL	LOAEL
<b>Semivolatile Organics</b>								
ACENAPHTHENE	3.3E-04	3.3E-05	8.4E-05	4.2E-05	6.4E-03	6.4E-04	3.6E-04	1.8E-04
ACENAPHTHYLENE	5.9E-04	5.9E-05	6.5E-05	6.5E-06	2.0E-03	2.0E-04	2.9E-05	2.9E-06
ANTHRACENE	5.7E-04	5.7E-05	4.4E-05	4.4E-06	1.5E-03	1.5E-04	1.5E-05	1.5E-06
BENZO(A)ANTHRACENE	4.8E-04	4.8E-05	1.7E-02	1.7E-03	5.3E-03	5.3E-04	3.1E-02	3.1E-03
BENZO(A)PYRENE	6.5E-04	6.5E-05	4.1E-03	4.1E-04	6.9E-03	6.9E-04	6.9E-03	6.9E-04
BENZO(B)FLUORANTHENE	1.4E-03	1.4E-04	2.5E-03	2.5E-04	7.6E-03	7.6E-04	1.9E-03	1.9E-04
BENZO(G,H,I)PERYLENE	6.1E-04	6.1E-05	6.1E-04	6.1E-05	3.7E-03	3.7E-04	5.1E-04	5.1E-05
BENZO(K)FLUORANTHENE	6.3E-04	6.3E-05	5.5E-04	5.5E-05	6.4E-03	6.4E-04	8.9E-04	8.9E-05
CHRYSENE	6.3E-04	6.3E-05	2.2E-02	2.2E-03	7.7E-03	7.7E-04	4.5E-02	4.5E-03
DIBENZO(A,H)ANTHRACENE	7.1E-05	7.1E-06	3.3E-04	3.3E-05	7.6E-04	7.6E-05	5.7E-04	5.7E-05
FLUORANTHENE	2.2E-03	2.2E-04	1.4E-03	6.8E-04	8.1E-03	8.1E-04	6.4E-04	3.2E-04
INDENO(1,2,3-CD)PYRENE	4.1E-04	4.1E-05	3.4E-04	3.4E-05	4.8E-03	4.8E-04	6.7E-04	6.7E-05
PHENANTHRENE	4.6E-03	4.6E-04	3.7E-02	3.7E-03	6.4E-03	6.4E-04	6.4E-03	6.4E-04
PYRENE	3.2E-03	3.2E-04	3.4E-03	2.0E-03	8.4E-03	8.4E-04	1.1E-03	6.7E-04
<b>Pesticides/PCBs</b>								
4,4'-DDD	6.9E-04	5.5E-04	3.6E-03	1.9E-03	1.6E-01	1.3E-01	1.5E-01	8.0E-02
4,4'-DDE	2.6E-04	2.1E-04	1.4E-03	7.7E-04	1.1E-01	8.6E-02	9.8E-02	5.3E-02
4,4'-DDT	4.2E-04	3.4E-04	2.3E-03	1.2E-03	1.3E-01	1.1E-01	1.2E-01	6.6E-02
ALPHA-CHLORDANE	2.8E-06	5.6E-07	2.4E-06	1.2E-06	1.0E-03	2.1E-04	2.9E-04	1.5E-04
AROCLOR-1260	1.1E-03	1.1E-04	2.9E-03	2.9E-04	7.5E-01	7.5E-02	1.2E+00	1.2E-01
GAMMA-CHLORDANE	2.1E-06	4.1E-07	1.7E-06	8.7E-07	7.7E-04	1.5E-04	2.1E-04	1.1E-04
<b>Inorganics</b>								
CADMIUM	2.7E-02	6.2E-03	1.8E-01	2.0E-02	2.4E+00	5.6E-01	2.8E+00	3.1E-01
CHROMIUM	5.3E-02	8.9E-03	1.3E-01	5.2E-03	1.2E+00	2.0E-01	6.8E-01	2.8E-02
COPPER	5.9E-02	6.9E-03	1.4E-01	1.0E-02	7.6E-01	8.9E-02	2.9E-01	2.1E-02
LEAD	6.8E-02	2.5E-03	5.6E-02	1.4E-03	1.7E+00	6.4E-02	3.2E-01	8.1E-03
MERCURY	6.4E-01	6.4E-02	5.0E-01	1.0E-01	1.6E+01	1.6E+00	1.9E+00	3.8E-01
SELENIUM	3.0E-02	1.5E-02	2.3E-01	1.4E-01	3.4E-01	1.7E-01	3.9E-01	2.4E-01
SILVER	1.3E-03	4.4E-05	6.4E-04	3.2E-05	2.3E-01	7.8E-03	4.6E-02	2.3E-03
VANADIUM	2.3E-01	4.6E-02	2.0E-02	1.1E-02	2.3E+00	4.7E-01	5.6E-02	3.0E-02
ZINC	9.0E-02	9.9E-03	3.2E-02	1.6E-02	3.6E+00	3.9E-01	1.9E-01	9.6E-02

Cells are shaded if the EEQ is greater than 1.0

NOAEL - No Observed Adverse Effects Level

LOAEL - Lowest Observed Adverse Effects Level

EEQ - Ecological Effects Quotient

TABLE 6-3

TERRESTRIAL FOOD CHAIN MODEL - LESS CONSERVATIVE SCENARIO  
 SEDIMENT INVERTIVOROUS RECEPTORS  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT

Chemical	Sediment Invertivorous Receptors EEQs			
	Mallard Duck		Raccoon	
	NOAEL	LOAEL	NOAEL	LOAEL
<b>Semivolatile Organics</b>				
1,4-DICHLOROBENZENE	NV	NV	1.1E-04	1.1E-05
2-METHYLNAPHTHALENE	3.7E-03	3.7E-04	9.1E-05	9.1E-06
ACENAPHTHENE	2.8E-03	2.8E-04	1.7E-04	8.5E-05
ACENAPHTHYLENE	3.1E-03	3.1E-04	4.7E-05	4.7E-06
ANTHRACENE	1.5E-02	1.5E-03	1.5E-04	1.5E-05
BENZO(A)ANTHRACENE	3.6E-02	3.6E-03	2.3E-01	2.3E-02
BENZO(A)PYRENE	4.4E-02	4.4E-03	4.6E-02	4.6E-03
BENZO(B)FLUORANTHENE	5.5E-02	5.5E-03	1.5E-02	1.5E-03
BENZO(G,H,I)PERYLENE	3.3E-02	3.3E-03	4.8E-03	4.8E-04
BENZO(K)FLUORANTHENE	5.1E-02	5.1E-03	7.4E-03	7.4E-04
CHRYSENE	4.9E-02	4.9E-03	3.0E-01	3.0E-02
DIBENZO(A,H)ANTHRACENE	2.9E-03	2.9E-04	2.3E-03	2.3E-04
FLUORANTHENE	7.8E-02	7.8E-03	6.6E-03	3.3E-03
FLUORENE	1.4E-02	1.4E-03	1.2E-03	5.8E-04
INDENO(1,2,3-CD)PYRENE	3.2E-02	3.2E-03	4.7E-03	4.7E-04
NAPHTHALENE	1.6E-03	1.6E-04	2.3E-04	1.2E-04
PENTACHLOROPHENOL	7.9E-05	1.0E-05	8.7E-05	3.3E-05
PHENANTHRENE	4.2E-02	4.2E-03	4.5E-02	4.5E-03
PYRENE	5.1E-02	5.1E-03	7.2E-03	4.3E-03
<b>Pesticides/PCBs</b>				
4,4'-DDD	4.7E-02	3.8E-02	3.8E-02	2.1E-02
4,4'-DDE	2.2E-01	1.8E-01	1.6E-01	8.8E-02
4,4'-DDT	1.6E-01	1.3E-01	1.2E-01	6.7E-02
ALDRIN	NV	NV	2.2E-03	4.3E-04
ALPHA-CHLORDANE	8.3E-03	1.7E-03	1.9E-03	9.4E-04
AROCLOR-1260	5.5E+00	5.5E-01	7.1E+00	7.1E-01
BETA-BHC	1.3E-03	3.3E-04	9.1E-04	1.8E-04
DELTA-BHC	2.1E-03	5.1E-04	4.1E-02	4.1E-03
DIELDRIN	4.5E-02	4.0E-03	1.1E-01	1.2E-03
ENDOSULFAN I	1.3E-04	1.3E-05	4.2E-03	4.2E-04
ENDOSULFAN II	2.6E-04	2.6E-05	8.6E-03	8.6E-04
ENDOSULFAN SULFATE	2.4E-04	2.4E-05	7.8E-03	7.8E-04
ENDRIN	2.4E-01	2.4E-02	1.3E-02	1.3E-03
ENDRIN ALDEHYDE	1.7E-01	1.7E-02	9.1E-03	9.1E-04
ENDRIN KETONE	2.5E-01	2.5E-02	1.3E-02	1.3E-03
GAMMA-BHC (LINDANE)	4.8E-04	4.8E-05	5.9E-05	5.9E-06
GAMMA-CHLORDANE	1.4E-03	2.8E-04	3.2E-04	1.6E-04
HEPTACHLOR	NV	NV	6.1E-03	6.1E-04
HEPTACHLOR EPOXIDE	NV	NV	4.8E-03	4.8E-04
METHOXYCHLOR	NV	NV	1.3E-03	6.4E-04
<b>Inorganics</b>				
ARSENIC	3.5E-02	1.7E-02	2.1E-02	1.1E-02
CADMIUM	5.0E-02	1.1E-02	5.3E-02	5.9E-03
CHROMIUM	1.5E-01	2.5E-02	1.2E-01	4.8E-03
COPPER	1.4E+00	1.6E-01	5.0E-01	3.5E-02
LEAD	2.5E-01	9.2E-03	6.8E-02	1.7E-03
MERCURY	3.6E+00	3.6E-01	3.7E-01	7.4E-02
NICKEL	1.1E-01	3.8E-02	2.3E-01	2.6E-02
SELENIUM	2.0E-01	1.0E-01	2.1E-01	1.3E-01
SILVER	2.1E-02	7.0E-04	3.6E-03	1.9E-04
ZINC	1.2E+00	1.3E-01	5.3E-02	2.6E-02

Cells are shaded if the value is greater than 1.0

NOAEL - No Observed Adverse Effects Level

LOAEL - Lowest Observed Adverse Effects Level

EEQ - Ecological Effects Quotient

NV - No value could be calculated because toxicity data was not available for this chemical.

**APPENDIX C – ATTACHMENT 1**  
**CHEMICAL CONCENTRATION FIGURES**

## General Legend for the Concentration Figures

### Green Symbols

The locations designated with a green symbol have detected concentrations (or one-half of the reporting limit for non-detected chemicals) that are less than their respective screening level. The screening levels are the Threshold Effects Levels (TECs) from MacDonald et al., (2000).

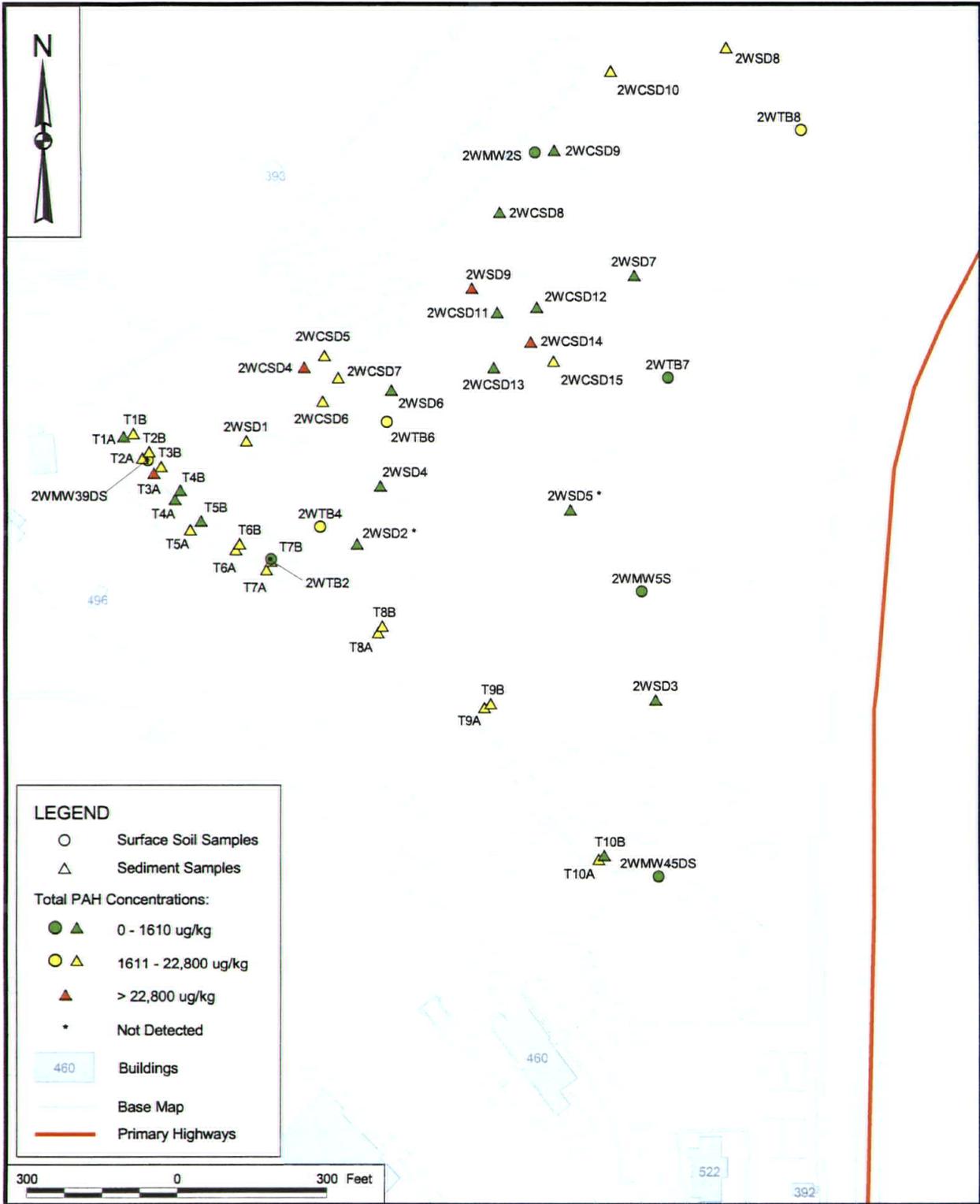
### Yellow Symbols

The locations designated with a yellow symbol have detected concentrations (or one-half of the reporting limit for non-detected chemicals) that are greater than their respective screening level, but less than their respective Probable Effects Concentration (PEC) from MacDonald et al., (2000).

### Red Symbols

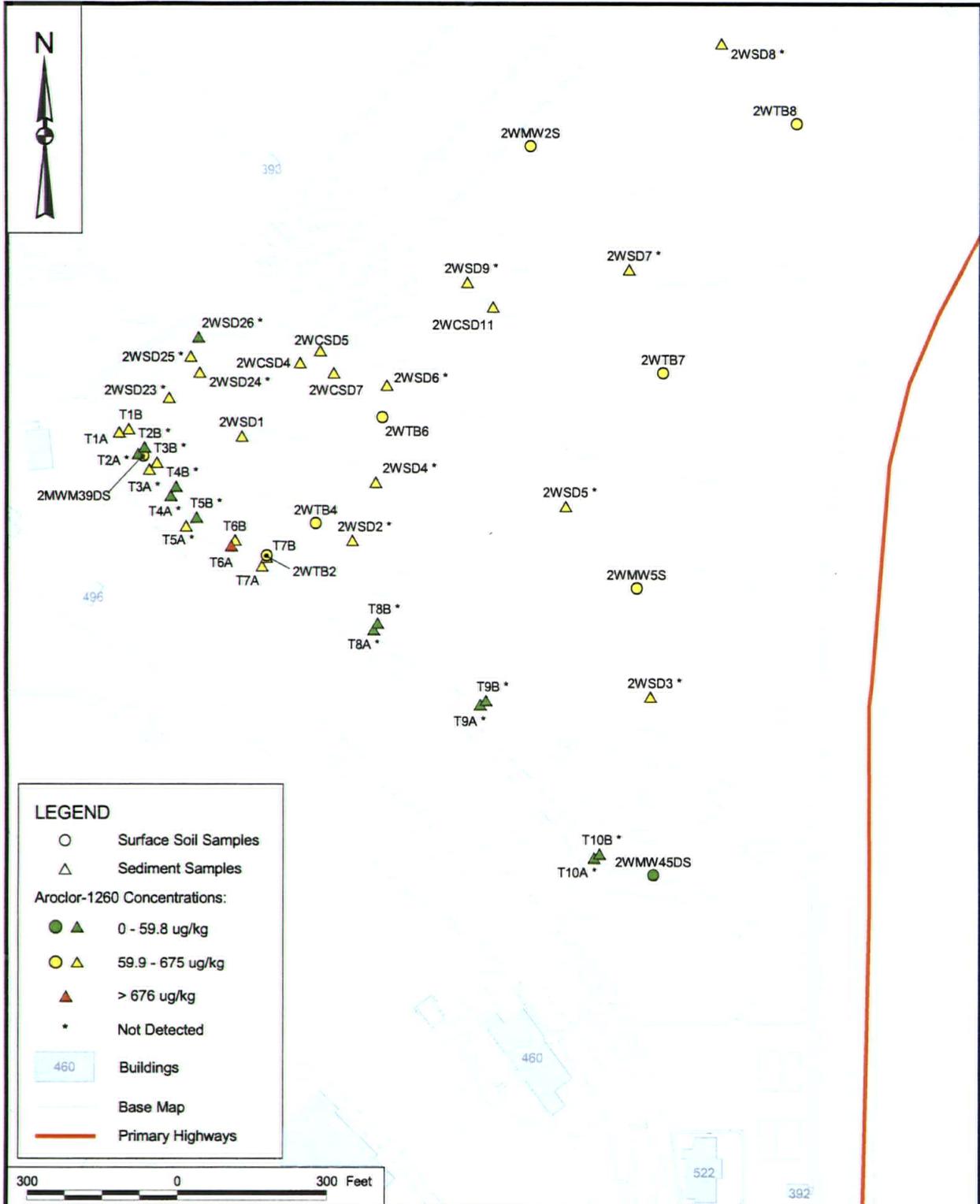
The locations designated with a red symbol have detected concentrations (or one-half of the reporting limit for non-detected chemicals) that are greater than their respective PEC.

For the total organic carbon (TOC) plot, the colors were selected to represent ranges of the TOC concentrations based on professional judgment. They are not meant to represent any particular "risk" threshold.



DRAWN BY J. ENGLISH	DATE 6/28/07	Tetra Tech NUS, Inc.	CONTRACT NUMBER CTO 0439	OWNER NO. ---	
CHECKED BY A. BERNHARDT	DATE 8/07/07		APPROVED BY <i>AJB</i>	DATE 8-12-07	
COST/SCHEDULE-AREA		<b>TOTAL PAH SAMPLE RESULTS</b> AREA A WETLAND NAVAL SUBMARINE BASE NEW LONDON GROTON, CONNECTICUT	APPROVED BY ---	DATE ---	
SCALE AS NOTED			DRAWING NO.	FIGURE 1	REV 0

P:\GIS\NLOMAP\RIAREA A SITE LOCATION.APR AREA A TOTAL PAH RESULTS LAYOUT 8/07/07 JEE



**LEGEND**

- Surface Soil Samples
- △ Sediment Samples

**Aroclor-1260 Concentrations:**

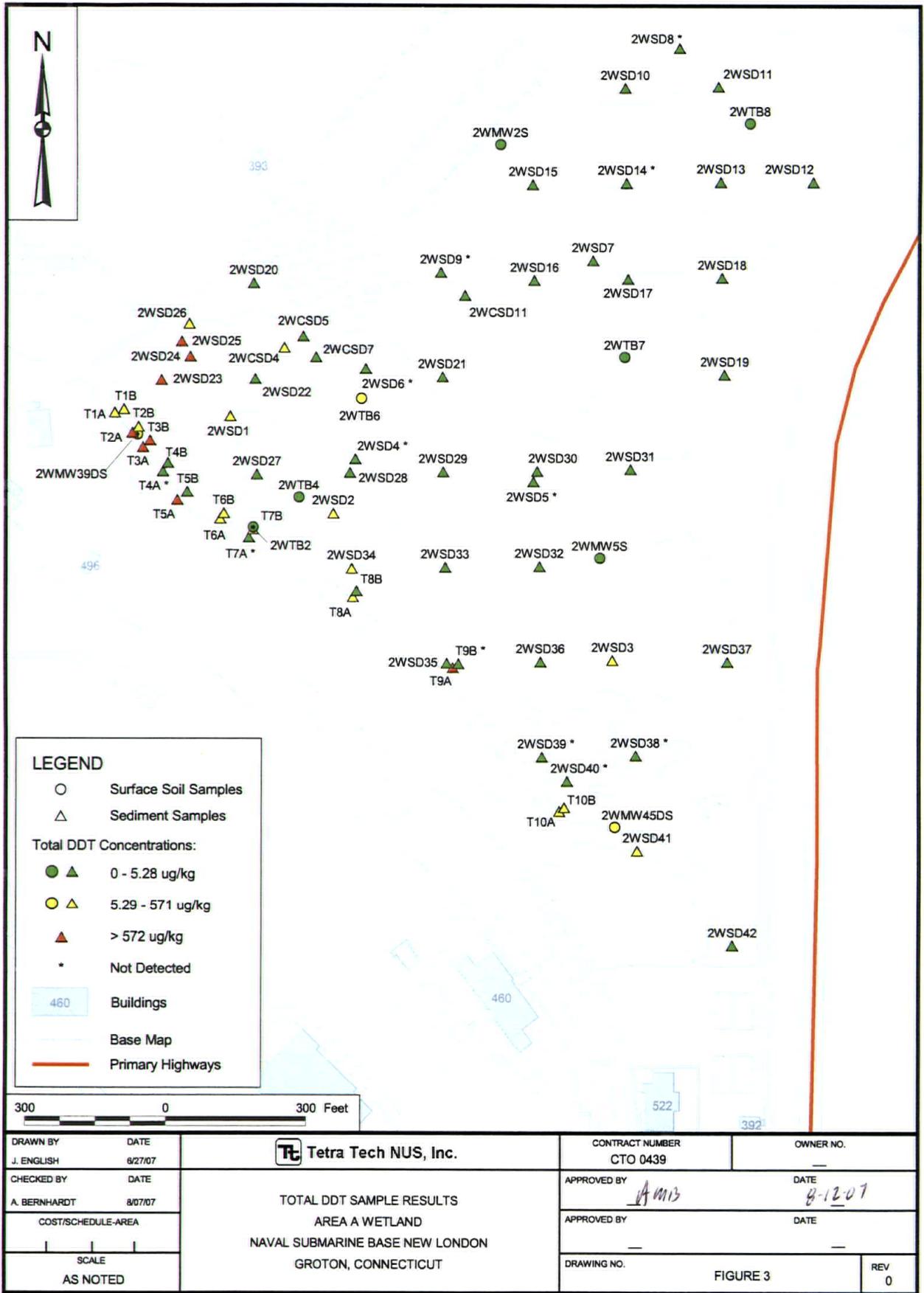
- ▲ 0 - 59.8 ug/kg
- ▲ 59.9 - 675 ug/kg
- ▲ > 676 ug/kg
- Not Detected

- 460 Buildings
- Base Map
- Primary Highways

300 0 300 Feet

DRAWN BY J. ENGLISH CHECKED BY A. BERNHARDT COST/SCHEDULE-AREA SCALE AS NOTED	DATE 6/26/07 DATE 7/13/07	Tetra Tech NUS, Inc.  <b>AROCOLOR-1260 SAMPLE RESULTS</b> AREA A WETLAND NAVAL SUBMARINE BASE NEW LONDON GROTON, CONNECTICUT	CONTRACT NUMBER CTO 0439  APPROVED BY <i>AJB</i> APPROVED BY DRAWING NO. FIGURE 2	OWNER NO. DATE 8-12-07 DATE REV 0
---	------------------------------------	---	--	--

P:\GIS\NLO\NAP\AREA A SITE LOCATION.APR AREA A AROCLOR-1260 RESULTS LAYOUT 8/07/07 JEE



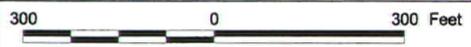
**LEGEND**

- Surface Soil Samples
- △ Sediment Samples

Total DDT Concentrations:

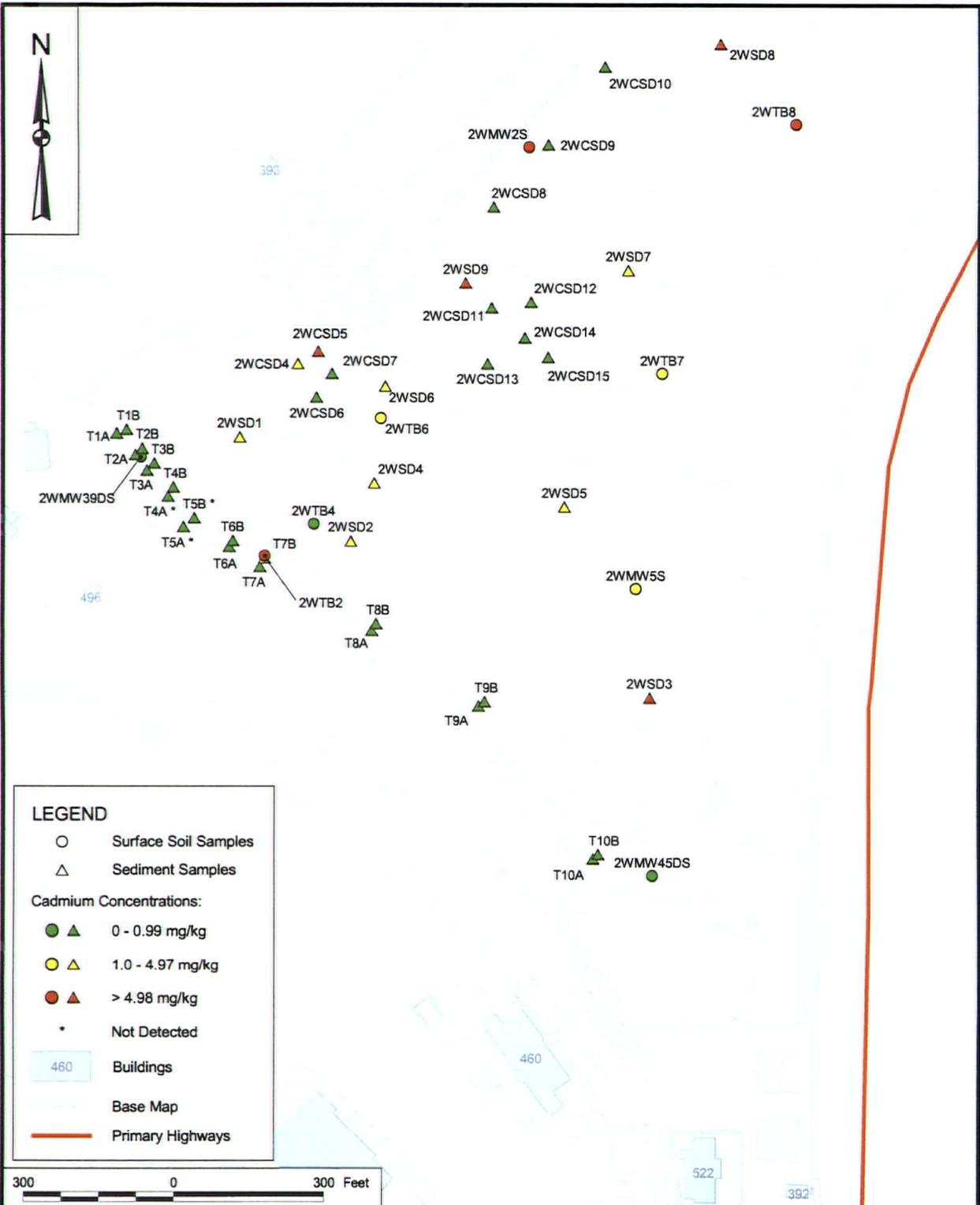
- ▲ 0 - 5.28 ug/kg
- ▲ 5.29 - 571 ug/kg
- ▲ > 572 ug/kg
- \* Not Detected

- 460 Buildings
- Base Map
- Primary Highways



DRAWN BY J. ENGLISH CHECKED BY A. BERNHARDT COST/SCHEDULE-AREA SCALE AS NOTED	DATE 8/27/07 DATE 8/07/07 DATE 8/07/07	Tetra Tech NUS, Inc.  <b>TOTAL DDT SAMPLE RESULTS</b> AREA A WETLAND NAVAL SUBMARINE BASE NEW LONDON GROTON, CONNECTICUT	CONTRACT NUMBER CTO 0439  APPROVED BY <i>AM13</i>  APPROVED BY _____  DRAWING NO. FIGURE 3	OWNER NO. _____  DATE 8-12-07  DATE _____  REV 0
---	---	---	--	--

P:\GIS\INLON\MAPPING\APR\AREA A SITE LOCATION.APR AREA A TOTAL DDT RESULTS LAYOUT 8/07/07 JEE



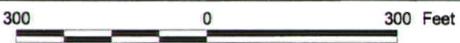
**LEGEND**

- Surface Soil Samples
- △ Sediment Samples

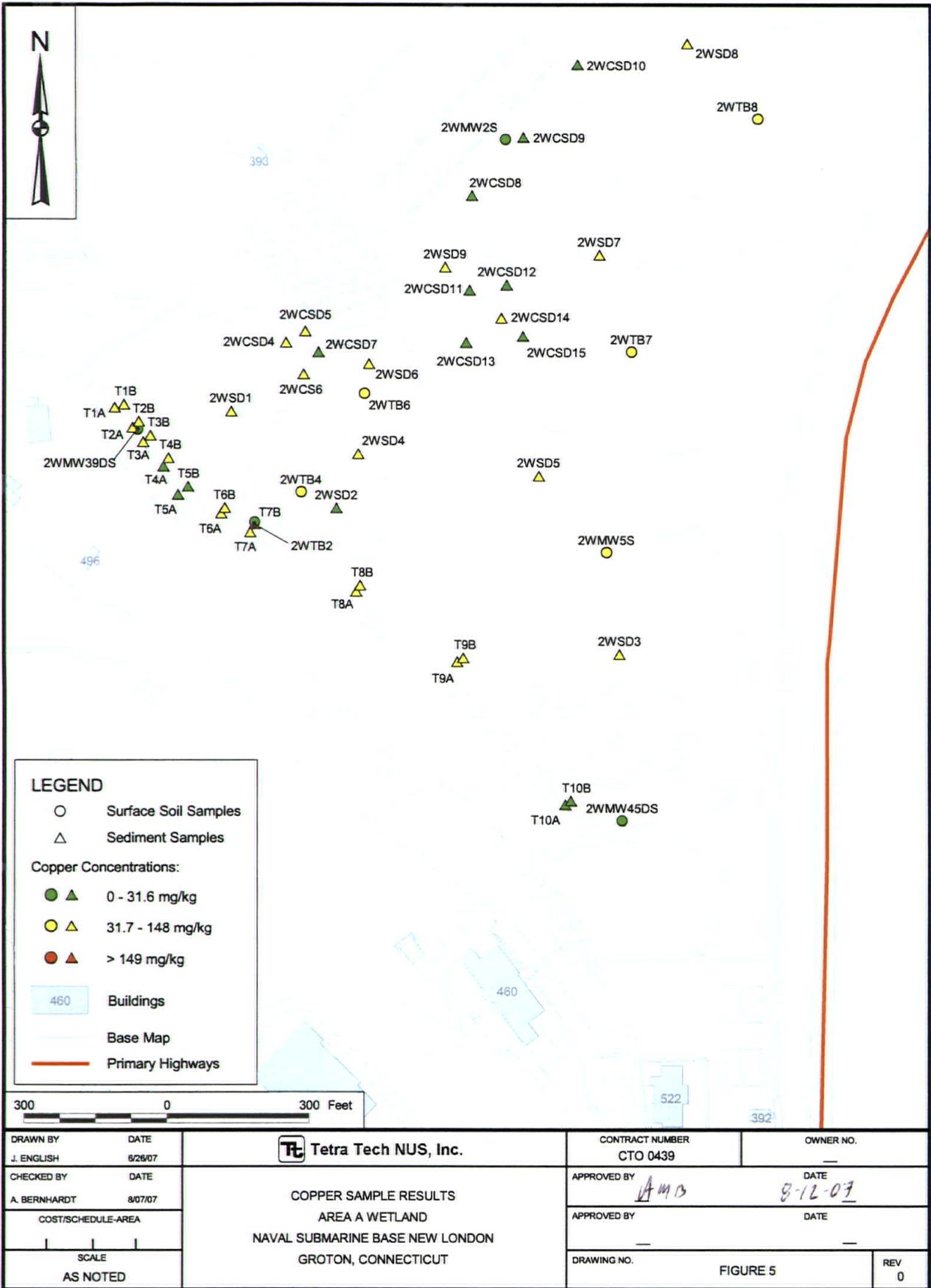
Cadmium Concentrations:

- ▲ 0 - 0.99 mg/kg
- ▲ 1.0 - 4.97 mg/kg
- ▲ > 4.98 mg/kg
- \* Not Detected

- 460 Buildings
- Base Map
- Primary Highways



DRAWN BY J. ENGLISH DATE 6/26/07	Tetra Tech NUS, Inc.  <b>CADMIUM SAMPLE RESULTS</b> AREA A WETLAND NAVAL SUBMARINE BASE NEW LONDON GROTON, CONNECTICUT	CONTRACT NUMBER CTO 0439	OWNER NO. 
CHECKED BY A. BERNHARDT DATE 8/07/07		APPROVED BY <i>AUB</i>	DATE 8-12-07
COST/SCHEDULE-AREA 		APPROVED BY 	DATE 
SCALE AS NOTED		DRAWING NO. FIGURE 4	REV 0



**LEGEND**

- Surface Soil Samples
- △ Sediment Samples

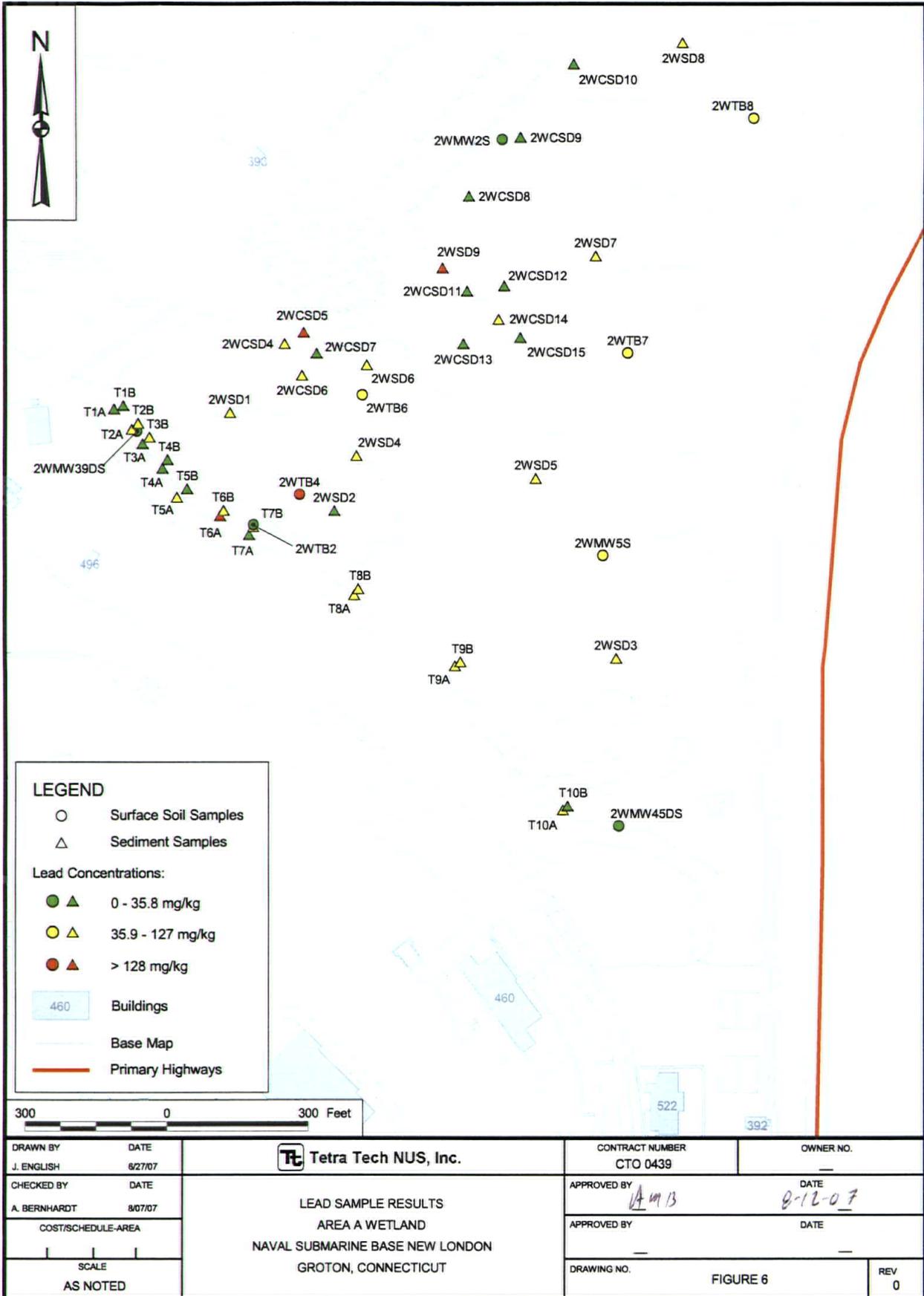
**Copper Concentrations:**

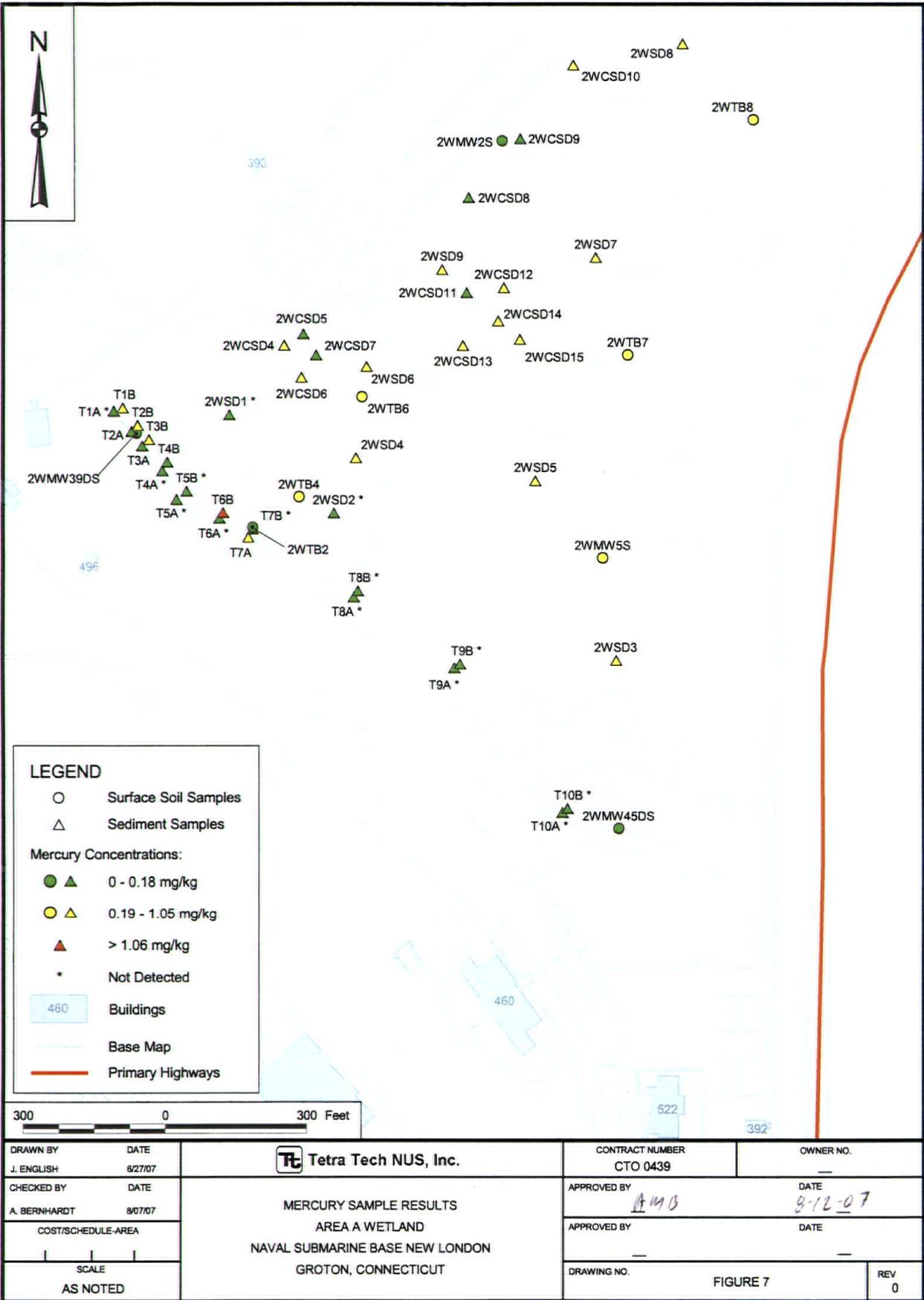
- ▲ 0 - 31.6 mg/kg
- ▲ 31.7 - 148 mg/kg
- ▲ > 149 mg/kg

- 460 Buildings
- Base Map
- Primary Highways

300 0 300 Feet

DRAWN BY J. ENGLISH		DATE 6/26/07		Tetra Tech NUS, Inc.		CONTRACT NUMBER CTO 0439		OWNER NO.					
CHECKED BY A. BERNHARDT		DATE 8/07/07				APPROVED BY <i>AMB</i>		DATE 8-12-07					
COST/SCHEDULE-AREA				<b>COPPER SAMPLE RESULTS</b> AREA A WETLAND NAVAL SUBMARINE BASE NEW LONDON GROTON, CONNECTICUT						APPROVED BY		DATE	
SCALE AS NOTED										DRAWING NO.		FIGURE 5	



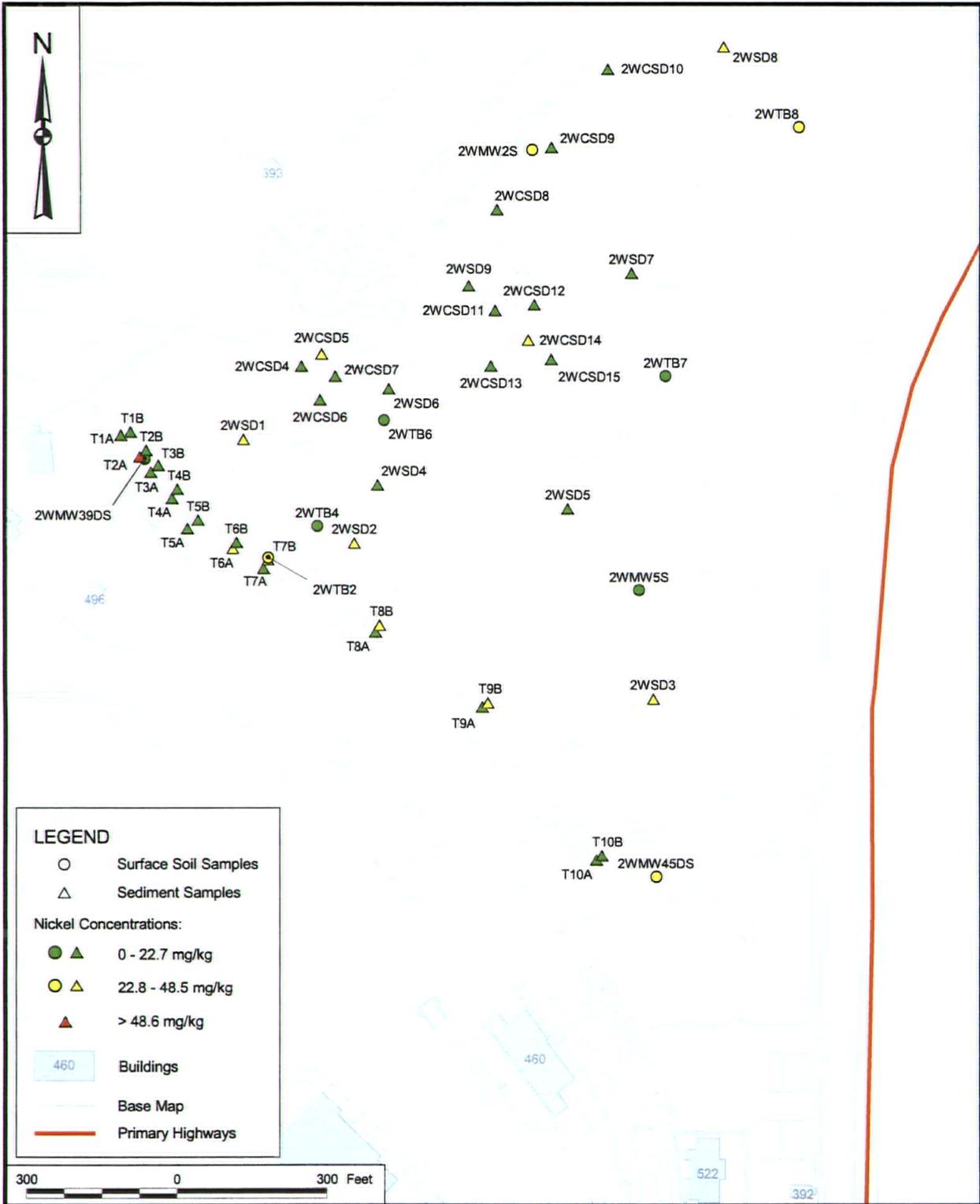


**LEGEND**

- Surface Soil Samples
- △ Sediment Samples
- Mercury Concentrations:**
- ▲ 0 - 0.18 mg/kg
- ▲ 0.19 - 1.05 mg/kg
- ▲ > 1.06 mg/kg
- \* Not Detected
- 460 Buildings
- Base Map
- Primary Highways

300 0 300 Feet

DRAWN BY J. ENGLISH CHECKED BY A. BERNHARDT COST/SCHEDULE-AREA SCALE AS NOTED	DATE 8/27/07 DATE 8/07/07 DATE DATE	Tetra Tech NUS, Inc.  <b>MERCURY SAMPLE RESULTS</b> AREA A WETLAND NAVAL SUBMARINE BASE NEW LONDON GROTON, CONNECTICUT	CONTRACT NUMBER CTO 0439  APPROVED BY <i>AMB</i>  APPROVED BY DRAWING NO.	OWNER NO. DATE 8-12-07 DATE FIGURE 7	REV 0
---	--	---	--	--	----------



**LEGEND**

- Surface Soil Samples
- △ Sediment Samples

Nickel Concentrations:

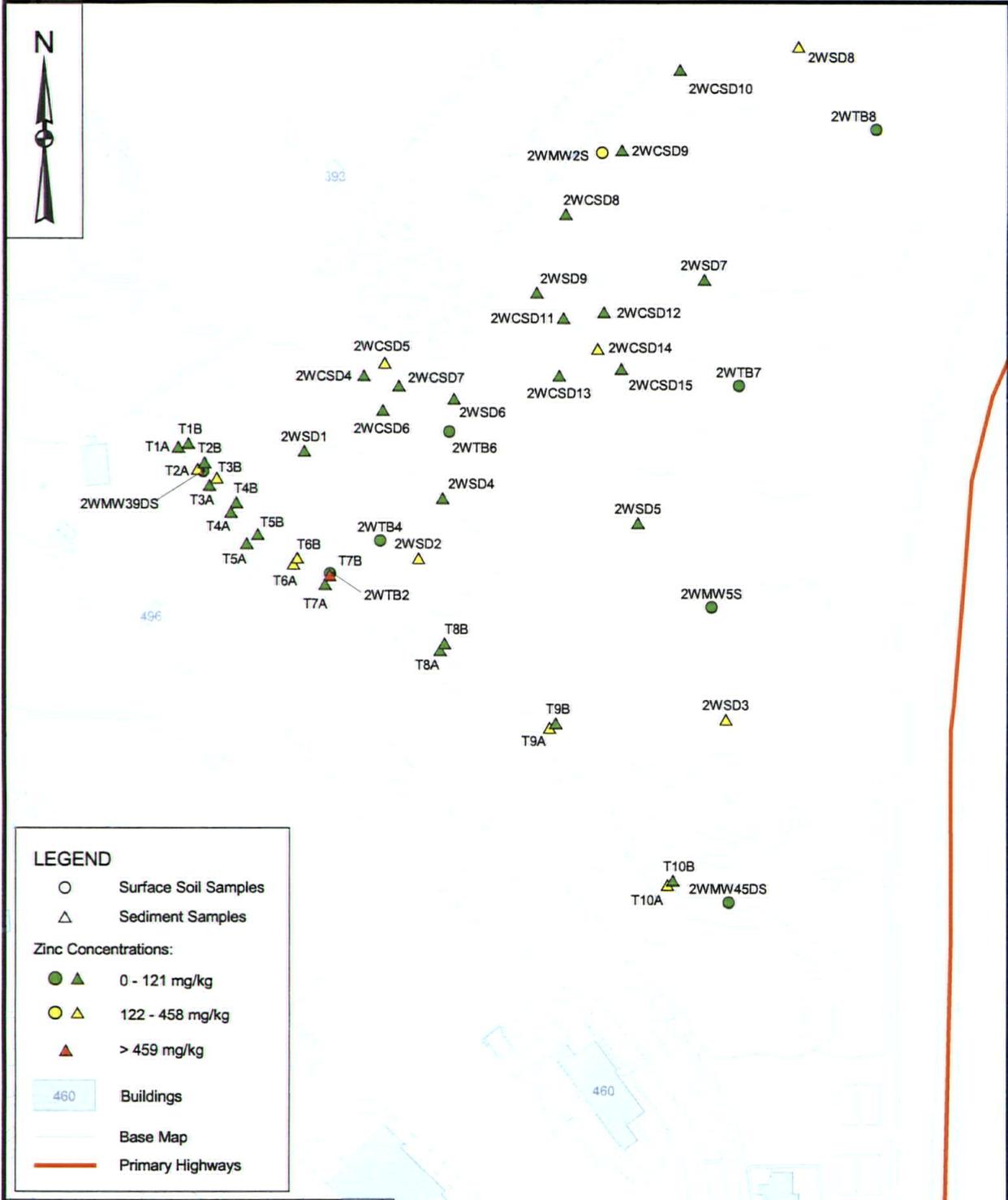
- ▲ 0 - 22.7 mg/kg
- ▲ 22.8 - 48.5 mg/kg
- ▲ > 48.6 mg/kg

- 460 Buildings
- Base Map
- Primary Highways



DRAWN BY J. ENGLISH CHECKED BY A. BERNHARDT COST/SCHEDULE-AREA SCALE AS NOTED	DATE 6/27/07 DATE 8/07/07  T Tetra Tech NUS, Inc.  NICKEL SAMPLE RESULTS AREA A WETLAND NAVAL SUBMARINE BASE NEW LONDON GROTON, CONNECTICUT	CONTRACT NUMBER CTO 0439 APPROVED BY <i>A M B</i> APPROVED BY DRAWING NO. FIGURE 8	OWNER NO.  DATE 8-12-07 DATE  REV 0
---	---	--	--

P:\GIS\NLOMAP\AREA A SITE LOCATION.APR AREA A NICKEL RESULTS LAYOUT 8/07/07 JEE



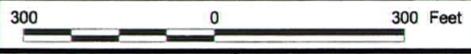
**LEGEND**

- Surface Soil Samples
- △ Sediment Samples

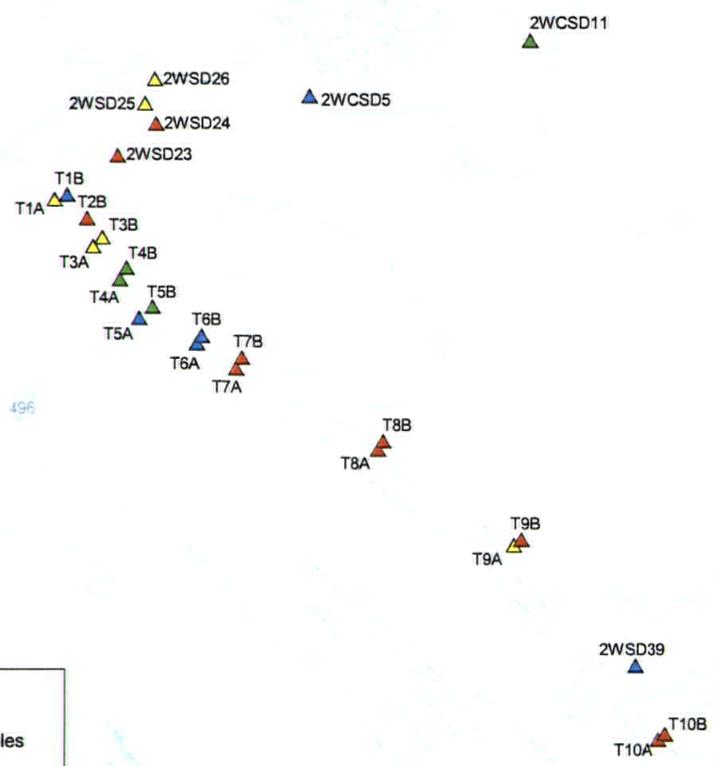
Zinc Concentrations:

- ▲ 0 - 121 mg/kg
- ▲ 122 - 458 mg/kg
- ▲ > 459 mg/kg

- 460 Buildings
- Base Map
- Primary Highways

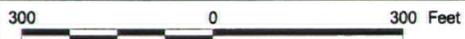


DRAWN BY J. ENGLISH	DATE 6/28/07	<b>Tetra Tech NUS, Inc.</b>	CONTRACT NUMBER CTO 0439	OWNER NO. —
CHECKED BY A. BERNHARDT	DATE 8/07/07		APPROVED BY <i>A M B</i>	DATE 8-12-07
COST/SCHEDULE-AREA		<b>ZINC SAMPLE RESULTS</b> AREA A WETLAND NAVAL SUBMARINE BASE NEW LONDON GROTON, CONNECTICUT	APPROVED BY —	DATE —
SCALE AS NOTED			DRAWING NO. FIGURE 9	REV 0



**LEGEND**

- △ Surface Soil Samples
- TOC Concentrations:
  - ▲ 0 - 10,000 mg/kg
  - ▲ 10,000 - 25,000 mg/kg
  - ▲ 26,000 - 50,000 mg/kg
  - ▲ > 50,000 mg/kg
- 460 Buildings
- Base Map
- Primary Highways



DRAWN BY J. ENGLISH	DATE 8/27/07	<b>Tetra Tech NUS, Inc.</b>	CONTRACT NUMBER CTO 0439	OWNER NO. —
CHECKED BY A. BERNHARDT	DATE 8/07/07		APPROVED BY <i>A.M.S.</i>	DATE 8-12-07
COST/SCHEDULE-AREA		<b>TOTAL ORGANIC CARBON SAMPLE RESULTS</b> <b>AREA A WETLAND</b> <b>NAVAL SUBMARINE BASE NEW LONDON</b> <b>GROTON, CONNECTICUT</b>	APPROVED BY —	DATE —
SCALE AS NOTED			DRAWING NO. FIGURE 10	REV 0

**APPENDIX C – ATTACHMENT 2**

**EXPOSURE PARAMETERS FOR FOOD CHAIN MODELS**

**RECEPTOR PROFILES  
SITE 2B - AREA A WETLAND  
NSB-NLON, GROTON, CONNECTICUT**

The following sections present the receptor profiles for the short-tailed shrew, American robin, meadow vole, northern bobwhite quail, raccoon, and mallard duck. The majority of the information for the profiles was obtained from the Wildlife Exposure Factors Handbook (USEPA, 1993). The data for the incidental soil ingestion rates were obtained from the Estimates of Soil Ingestion by Wildlife (Beyer, 1994) or the USEPA Ecological Soil Screening Guidance (USEPA, February 2005).

The food and water ingestion rates are listed in g/g (of body weight)-day on a wet weight basis but were converted to dry weight for the ERA. The home ranges are presented in hectares in USEPA (December 1993) but were converted to acres by multiplying the number of hectares by 2.471. Also note that the estimated percent of soil in the diets are listed in dry weight. The attached table presents the calculation of the exposure parameters and how the calculations were done.

**Short-Tailed Shrew (*Blarina brevicauda*)**

Shrews inhabit a wide variety of habitats and are common in areas with abundant vegetative cover. They need cool, moist habitats because of their high metabolic and water-loss rates. The short-tailed shrew is primarily carnivorous, eating insects such as earthworms, slugs, and snails.

The adult body weight for the short-tailed shrew in various habitats ranged from 0.015 to 0.01921 kg with an average of 0.0161 kg. The listed food ingestion rates for shrews are 0.49 and 0.62 g/g-day (wet-weight). The water ingestion rate was listed as 0.223 g/g-day. The food and water ingestion rates in kg/day and L/day, respectively, were calculated as shown in the attached table. The food ingestion rates were then multiplied by 0.16, which is the percent solids of worms (Sample et al., 1997) to convert the ingestion rate from a wet-weight value to a dry-weight value. The incidental soil ingestion rate was calculated by multiplying the ingestion rate by the percentage of soil that is incidentally ingested (assumed 3 percent for conservative food chain model and 0.9 percent for the average food chain model) from USEPA (February 2005). 3 percent is the 90<sup>th</sup> percentile value and 0.9 percent is the 50<sup>th</sup> percentile value from USEPA (February 2005).

The home range for the shrew (0.9699 acres) was calculated using data from a tamarek bog in Manitoba (only value available).

### **American Robin (*Turdus migratorius*)**

American robins' habitats include parks, lawns, moist forests, swamps, open woodlands, and orchards. Robins forage on the ground in open areas, along habitat edges, or the edges of streams. They also may forage above ground in shrubs and within the lower branches of trees. In the months preceding and during the breeding season, robins feed primarily on invertebrates and on some fruits. During the rest of the year their diet consists primarily of fruits.

The adult body weight for the American robin in New York woodlands and forests and in Pennsylvania ranged from 0.0773 to 0.0862 kg with an average of 0.0804 kg. The only listed food ingestion rates were for robins in Kansas (1.52 g/g-day) and California (0.89 g/g-day), with an average of 1.205 g/g-day. The water ingestion rate was estimated as 0.14 g/g-day. Studies calculating ingestion rates for the robin included in the USEPA (December 1993) are based on a diet comprised of berries. However, because it is assumed that 100 percent of the robin's diet is worms for the food chain models, the ingestion rate for the robin was calculated using field metabolism scaling as presented on the attached table (Nagy et al., 1999). These are the values that were used in the food chain model for this site. The incidental soil ingestion rate was calculated by multiplying the ingestion rate by the percentage of soil that is incidentally ingested (assumed 16.4 percent for conservative food chain model and 6.4 percent for the average food chain model) from USEPA (February 2005). The 16.4 percent and 6.4 percent values are based on the American woodcock.

The home range for the robin was calculated using data from Tennessee and a New York dense conifer forest. The values ranged from 0.27 to 1.04 acres with an average home range of 0.61 acres.

### **Meadow Vole (*Microtus pennsylvanicus*)**

Meadow voles inhabit grassy fields, marshes, and bogs; however, they prefer fields with more grass, more cover, and fewer woody plants. They typically consume green succulent vegetation, sedges, seeds, roots, bark, fungi, insects, and animal matter. However, green succulent vegetation makes up the majority of their diet.

The adult body weight for the vole ranges from 0.017 to 0.0524 kg with an average of 0.0358 kg. The only listed food ingestion rates for voles range from 0.30 to 0.35 g/g-day (wet-weight), with an average of 0.325 g/g-day. The water ingestion rates are 0.14 (estimated) and 0.21 g/g-day, with an average of 0.175 g/g-day. The food and water ingestion rates in kg/day and L/day, respectively, were calculated as shown in the attached table. The food ingestion rates were then multiplied by 0.30, which is the percent solids of young grass (Sample et al., 1997) to convert the ingestion rate from a wet-weight value to a dry-weight value. The incidental soil ingestion rate was calculated by multiplying the ingestion rate by the percentage of soil that is incidentally ingested (assumed 3.2 percent for conservative food chain model and 1.2 percent for the average

food chain model) from USEPA (February 2005). 3.2 percent is the 90<sup>th</sup> percentile value and 1.2 percent is the 50<sup>th</sup> percentile value from USEPA (February 2005).

The home range for the meadow vole ranges from 0.000494 to 0.2051 acres with an average home range of 0.0659 acres.

### **Northern Bobwhite Quail (*Colinus virginianus*)**

Quails inhabit grasslands, idle fields, pastures, and large clumps of grasses. Bobwhite quails forage in areas with open vegetation, some bare ground, and light litter. Seeds from weeds, woody plants, and grasses comprise the majority of an adult's diet, although green vegetation has been found to dominate the diet of this species in winter in the southern areas of the United States.

The adult body weight for the bobwhite quail ranges from 0.154 to 0.1939 kg with an average of 0.1751 kg. The listed food ingestion rates for quails range from 0.067 to 0.093 g/g-day (wet-weight), with an average of 0.078 g/g-day. The water ingestion rate is estimated as 0.10 and 0.11 g/g-day, and measured as 0.10 to 0.13 g/g-day, for an average water ingestion rate of 0.11 g/g-day. The food and water ingestion rates in kg/day and L/day, respectively, were calculated as shown in the attached table. The food ingestion rates were then multiplied by 0.30, which is the percent solids of young grass (Sample et al., 1997) to convert the ingestion rate from a wet-weight value to a dry-weight value. The incidental soil ingestion rate was calculated by multiplying the ingestion rate by the percentage of soil that is incidentally ingested (assumed 13.9 percent for conservative food chain model and 6.1 percent for the average food chain model) from USEPA (February 2005). The 13.9 percent and 6.1 percent values are based on the mourning dove.

The home range for the quail ranges from 8.9 to 41.3 acres with an average home range of 18.8 acres.

### **Raccoon (*Procyon lotor*)**

Raccoons are found near virtually every aquatic habitat, particularly in hardwood swamps, mangroves, floodplain forests, and freshwater and saltwater marshes. They are also common in suburban residential areas. They use surface waters for both drinking and foraging. They feed primarily on fleshy fruits, nuts, acorns, and corn, but also eat grains, insects, frogs, crayfish, eggs, and virtually any animal and vegetable matter.

The adult body weight for the raccoon ranges from 3.67 to 7.6 kg, with an average of 5.64 kg. The water ingestion rate ranges from 0.082 to 0.083 g/g. The food and water ingestion rates in kg/day and L/day, respectively, were calculated as shown in the attached table. The incidental sediment ingestion rate is calculated by multiplying the ingestion rate by the percentage of sediment that is incidentally ingested (9.4 percent), as cited in Beyer (1994).

The home range for the conservative model is assumed to be equal to the size of the site indicating that the raccoon will spend all of its time at the site. The typical home range sizes for the raccoon are 96 to 6,325 acres for an average home range of 1,558 acres.

### **Mallard Duck (*Anas platyrhynchos*)**

The mallard duck is a surface-feeding duck in that it feeds in shallow water, sifting and filtering through soft mud for food. Mallards are found in freshwater and saltwater wetlands nesting in areas that are dense with grassy vegetation approximately a half meter high. The mallard feeds mostly on seeds and grains of aquatic plants and aquatic invertebrates depending on the season, foraging and dabbling through sediment.

The male mallard duck is generally heavier than the female mallard duck with the average body weights ranging from 1.043 kg to 1.246 kg, with an average of 1.17 kg. The food and water ingestion rates in kg/day and L/day, respectively, were calculated as shown in the attached table. The incidental sediment ingestion rate is calculated by multiplying the ingestion rate by the percentage of sediment that is incidentally ingested (3.3 percent), as cited in Beyer (1994).

Based on data from Minnesota wetlands/river, mallard ducks have home ranges from 98.84 acres to 3558 with an average home range of 1433 acres.

## References:

Beyer, N., E. Connor, and S. Gerould. 1994. Estimates of Soil Ingestion by Wildlife. Journal of Wildlife Management 58(2) pp. 375-382.

Nagy, K.A., I.A. Girard, and T.K. Brown. 1999. Energetics of Free-Ranging Mammals, Reptiles, and Birds. Annu. Rev. Nutr. 19. pp. 247-277.

Sample, B.E., M.S. Aplin, R.A. Efroymson, G.W., Suter II, and C.J.E. Welsh. 1997. Methods and Tools for Estimation of the Exposure of Terrestrial Wildlife to Contaminants. Oak Ridge National Laboratory. October. ORNL/TM-13391.

USEPA (U.S. Environmental Protection Agency), 1993. Wildlife Exposure Factors Handbook. U.S. Environmental Protection Agency. Office of Research and Development. Washington, D.C. December 1993. EPA/600/R-93/187a.

USEPA, 2005. Ecological Soil Screening Level for Antimony, Interim Final. Office of Emergency and Remedial Response. OSWER Directive 9285.7-61. February.

DRY WEIGHT DERIVATION OF BODY WEIGHT, FOOD INTAKE, AND WATER INTAKE FACTORS FOR TERRESTRIAL FOOD CHAIN MODELS  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 1 OF 3

Species/Factor	Data from EPA (1993)		Study Average	Derivation of Factors for Modeling	
	Age/Sex/Cond./Seas.	Value		Calculation of Values	Notes
<b>American Robin</b>					
Body Weight (g)	A B	77.3	77.3	Minimum Value	0.0773 kg
	A M nonbreeding	86.2		Maximum Value	0.0862 kg
	A F nonbreeding	83.6	84.9	Overall Study Average	0.0804 kg
	A M breeding	77.4			
	A F breeding	80.6	79		
Food Ingestion Rate (g/g-day)	B B free-living	0.89		<i>For Eating Mostly Fruit</i>	
	- B free-living	1.52		Conservative value	0.0281 kg/day    Maximum ingestion rate * Average Body weight * 0.23 <sup>(1)</sup>
				Average value	0.0223 kg/day    Average ingestion rate * Average Body weight * 0.23 <sup>(1)</sup>
					<sup>(1)</sup> - 0.23 = percent solids in fruit to convert to a dry weight ingestion rate
	Overall Study Average		1.21	<i>Based on Metabolic Scaling</i>	
			0.01247 kg/day	Used maximum body weight in below equation	
			0.01188 kg/day	Used average body weight in below equation	
				Food ingestion rates were calculated from Nagy et al., (1999) for insectivores as follows $FI = (9.7 \cdot BW(g)^{0.705}) / 18kJ/g/1000$	
Water Ingestion Rate (g/g-day)	A B	0.14		Conservative value	0.012 L/day    Ingestion rate * Maximum Body weight
				Average value	0.011 L/day    Ingestion rate * Average Body weight
<b>Short-Tailed Shrew</b>					
Body Weight (g)	A B	15	15	Minimum Value	0.0150 kg
	M summer	19.21	17.27	Maximum Value	0.01921 kg
	F summer	17.4		Overall Study Average	0.01613 kg
	M fall	16.87			
	M fall	15.58			
Food Ingestion Rate (g/g-day)	A B	0.49		Conservative value	0.0016 kg/day    Maximum ingestion rate * Average Body weight * 0.16 <sup>(1)</sup>
	A B	0.62		Average value	0.00143 kg/day    Average ingestion rate * Average Body weight * 0.16 <sup>(1)</sup>
	Overall Study Average		0.555		<sup>(1)</sup> - 0.16 = percent solids in earthworms to convert to a dry weight ingestion rate
Water Ingestion Rate (g/g-day)	A B	0.223		Conservative value	0.00428 L/day    Ingestion rate * Maximum Body weight
				Average value	0.00360 L/day    Ingestion rate * Average Body weight

DRY WEIGHT DERIVATION OF BODY WEIGHT, FOOD INTAKE, AND WATER INTAKE FACTORS FOR TERRESTRIAL FOOD CHAIN MODELS  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 2 OF 3

Species/Factor	Data from EPA (1993)		Study Average	Derivation of Factors for Modeling		
	Age/Sex/Cond./Seas.	Value		Calculation of Values	Notes	
<b>Meadow Vole</b>						
Body Weight (g)	A M summer	40	36.7	Minimum Value	0.017 kg	
	A F summer	33.4		Maximum Value	0.052 kg	
				Overall Study Average	0.0358 kg	
	A M spring	52.4	48.0			
	A F spring	43.5				
	A B spring	26	21.2			
	A B summer	24.3				
	A B fall	17				
	A B winter	17.5				
	A M	35.5	37.3			
A F	39					
Food Ingestion Rate (g/g-day)		0.3	0.33	Conservative value:	0.003756 kg/day	Maximum ingestion rate * Average Body weight * 0.3 <sup>(1)</sup>
		0.35		Average value	0.003488 kg/day	Average ingestion rate * Average Body weight * 0.3 <sup>(1)</sup>
Water Ingestion Rate	A B	0.21	0.18	Conservative value:	0.007513 L/day	Maximum ingestion rate * Average Body weight
	A B	0.14		Average value	0.006261 L/day	Average ingestion rate * Average Body weight
<b>Northern Bobwhite Quail</b>						
Body Weight (g)	A B fall	189.9	191	Minimum Value	0.154 kg	
	A B winter	193.9		Maximum Value	0.194 kg	
	A B spring	190		Overall Study Average	0.1751 kg	
	A M winter	181	177			
	A M summer	163				
	A F winter	183				
	A F summer	180				
	A M winter	161	157			
	A M summer	154				
	A F winter	157				
A F summer	157					
Food Ingestion Rate (g/g-day)	A B winter	0.093	0.078	Conservative value	0.00488 kg/day	Maximum ingestion rate * Average Body weight * 0.3 <sup>(1)</sup>
	A B spring	0.067		Average value	0.00408 kg/day	Average ingestion rate * Average Body weight * 0.3 <sup>(1)</sup>
	A B summer	0.079				
	A B fall	0.072				
Water Ingestion Rate (g/g-day)	A M summer	0.1	0.11	Conservative value	0.0227616 L/day	Maximum ingestion rate * Average Body weight
	A F summer	0.13		Average value	0.0192598 L/day	Average ingestion rate * Average Body weight
	A M summer	0.11				
	A F summer	0.1				

<sup>(1)</sup> - 0.30 = percent solids in grass to convert to a dry weight ingestion rate

DRY WEIGHT DERIVATION OF BODY WEIGHT, FOOD INTAKE, AND WATER INTAKE FACTORS FOR TERRESTRIAL FOOD CHAIN MODELS  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 3 OF 3

Species/Factor	Data from EPA (1993)		Study Average	Derivation of Factors for Modeling		
	Age/Sex/Cond./Seas.	Value		Calculation of Values		Notes
<b>Raccoon</b>						
Body Weight (kg)	A M	7.6	6.7	Minimum Value	3.67 kg	
	A F parous	6.4		Maximum Value	7.6 kg	
	A F nulliparous	6		Overall Study Average	5.636 kg	
	A M	6.76	6.25			
	A F	5.74				
	A M	4.31	3.99			
A F	3.67					
Food Ingestion Rate (g/g-day)			No Value	Conservative value	0.237 kg/day	Used maximum body weight in below equation
				Average value	0.184 kg/day	Used average body weight in below equation
						Food ingestion rates were calculated from Nagy et al., (1999) for carnivores as follows $FI = ((2.23) \cdot BW(g)^{0.65}) / 1000$
Water Ingestion Rate (g/g-day)	A M	0.082	0.083	Conservative value	0.468 L/day	Maximum ingestion rate * Average Body weight
	A F	0.083		Average value	0.465 L/day	Average ingestion rate * Average Body weight
<b>Mallard</b>						
Body Weight (kg)	A M	1.225	1.134	Minimum Value	1.043 kg	
	A F	1.043		Maximum Value	1.246 kg	
				Overall Study Average	1.166 kg	
	A M	1.246	1.171			
	A F	1.095				
	A M	1.237	1.163			
A F	1.088					
A F	1.197	1.197				
Food Ingestion Rate (g/g-day)				Conservative value	0.08200 kg/day	Used maximum body weight in below equation
				Average value	0.07826 kg/day	Used average body weight in below equation
						Food ingestion rates were calculated from Nagy et al., (1999) for insectivores as follows: $FI = ((9.7 \cdot BW(g)^{0.705}) / 18kJ/g) / 1000$
Water Ingestion Rate (g/g-day)	A M	0.058	0.0565	Conservative value	0.067628 L/day	Maximum ingestion rate * Average Body weight
	A F	0.055		Average value	0.065879 L/day	Average ingestion rate * Average Body weight

Notes:  
 A = Adult  
 F = Female, M = Male, B = Both  
 BW = Body Weight

# Lipid Data By Organism Group

Lipid Data By Organism Groups							
Group	Wet/Dry	% Lipid (GM)	(n)	RMSE	Not Used	Min Value	Max Value
Birds	Wet	47.048	6	11.127	10	1.900	91.200
	Dry	N/A	0	N/A	2	12.700	12.800
	???	9.418	2	1.167	8	1.141	9.680
Crustacea - Freshwater	Wet	4.956	5	1.254	8	0.660	11.590
	Dry	*11.364	37	10.227	2	1.000	26.700
	???	1.351	2	0.229	1	0.718	3.000
Crustacea - Marine	Wet	2.984	8	1.935	29	0.470	17.700
	Dry	16.121	81	37.020	1	1.600	52.600
	???	5.954	11	3.579	3	0.213	9.600
Echinoderms	Wet	N/A	0	N/A	0	0	0
	Dry	5.554	20	1.897	8	0.001	32.100
	???	N/A	0	N/A	0	0	0
Fish - Bottom Feeders	Wet	4.342	41	10.573	46	0.050	52.000
	Dry	7.950	2	3.380	15	3.830	18.000
	???	7.444	77	5.286	44	0.180	45.000
Fish - Mid-Water Feeders	Wet	6.676	56	7.200	51	0.053	72.000
	Dry	N/A	0	N/A	6	9.300	51.100
	???	26.316	10	3.613	29	0.020	66.000
Fish - Plankton Feeders	Wet	N/A	0	N/A	9	3.300	11.600
	Dry	N/A	0	N/A	4	15.000	21.430
	???	3.071	6	3.003	3	1.015	14.000
Mammals - Marine	Wet	32.169	2	71.428	12	3.500	92.000
	Dry	N/A	0	N/A	0	0	0
	???	N/A	0	N/A	8	1.200	79.100
Misc	Wet	N/A	0	N/A	4	0.100	1.000
	Dry	5.200	1	4.700	0	5.200	5.200
	???	N/A	0	N/A	0	0	0
Molluscs - Freshwater	Wet	1.358	34	0.215	19	0.160	9.000
	Dry	*10.500	4	0.691	7	5.800	12.200

	???	N/A	0	N/A	1	0.900	0.900
Molluscs - Marine/Estuarine	Wet	1.419	12	0.092	20	0.150	6.310
	Dry	4.963	9	2.326	40	1.385	26.700
	???	7.615	4	0.682	11	0.300	9.400
Turtles/Amphibians	Wet	10.480	1	2.234	0	10.480	10.480
	Dry	N/A	0	N/A	0	0	0
	???	N/A	0	N/A	0	0	0
Worms - Freshwater	Wet	1.272	8	1.729	7	0.550	13.000
	Dry	*6.464	30	2.852	4	0.600	13.200
	???	2.864	12	9.367	1	0.130	3.700
Worms - Terrestrial	Wet	1.210	1	0.061	0	1.210	1.210
	Dry	N/A	0	N/A	0	0	0
	???	N/A	0	N/A	0	0	0

**Please Click Your Back Button To Select A Specific Organism**

Data shown are **Grand Means (GM) and Root Mean Square Error (RMSE)** except where (n) = 1. Those data are simple means and standard deviation from the citation. The *Not Used* column indicates available records that were missing (n) or error data and could not be used to calculate the grand mean. N/A indicates no data available to calculate a grand mean. Minimum and maximum data are calculated from ALL entries in the database

The column *Type* indicates the type of error reported for these data. SE was converted to SD for statistical calculations.

The error type abbreviations are *SD* = standard deviation, *SE* = standard error, *CI* = confidence interval, *SEM* = standard error of the mean, *CV* = Coefficient of Variation, *PE* = propagated error and *NA* = not available (unknown),

The column *Wet/Dry* indicates wet or dry weight. *Wet* = all values were wet weight, *Dry* = all values were dry weight, *Both* = sediment was dry weight, tissue was wet weight, *???* = Unknown, not specified in reference.

The column *Used?* indicates whether or not the %Lipid data were used in the Grand Mean and Error calculations. Only data containing mean, error, and number were used in Grand Mean and RMS Error calculations.

These data have been compiled by personnel of The US Army Engineer Research and Development Center, Waterways Experiment Station, Environmental Laboratory (CEERD-EP-R). It is strongly suggested that users verify that the displayed data are appropriate for their use before basing any decisions on them.

[Return to BSAF Home](#)

[Copy to spreadsheet instructions](#)

Lipid Data were last updated on March 12, 2007

Please contact Charlie Lutz with problems, suggestions, additions, etc. --- [Send E-mail](#)

Powered by dBase Plus software (© dBI Inc.)



**APPENDIX C – ATTACHMENT 3**  
**CHEMICAL SPECIFIC BIOACCUMULATION AND TOXICITY VALUES**

## ATTACHMENT TABLE 3-1

## TOXICITY REFERENCE VALUES FOR TERRESTRIAL FOOD CHAIN MODELS

## SITE 2B - AREA A WETLAND

## NSB-NLON, GROTON, CONNECTICUT

PAGE 1 OF 2

PARAMETER	Mammal		Bird	
	NOAEL	LOAEL	NOAEL	LOAEL
<b>SEMIVOLATILES</b>				
2-Methylnaphthalene	41	410	2	20
Acenaphthene	17.5	35	2	20
Acenaphthylene	70	700	2	20
Anthracene	100	1000	2	20
Benzo(a)anthracene	0.17	1.7	2	20
Benzo(a)pyrene	1	10	2	20
Benzo(b)fluoranthene	4	40	2	20
Benzo(g,h,i)perylene	7.2	72	2	20
Benzo(k)fluoranthene	7.2	72	2	20
Chrysene	0.17	1.7	2	20
Dibenzo(a,h)anthracene	1.33	13.3	2	20
Dibenzofuran	NV	NV	2	20
1,4-Dichlorobenzene	30	300	NV	NV
Fluoranthene	12.5	25	2	20
Fluorene	12.5	25	2	20
Indeno(1,2,3-cd)pyrene	7.2	72	2	20
Naphthalene	7.1	14.2	2	20
Pentachlorophenol	8.42	22.65	6.73	52.01
Phenanthrene	1	10	2	20
Pyrene	7.5	12.5	2	20
<b>PESTICIDES/PCBs</b>				
4,4'-DDD	0.147	0.274	0.227	0.281
4,4'-DDE	0.147	0.274	0.227	0.281
4,4'-DDT	0.147	0.274	0.227	0.281
Aldrin	0.2	1	NV	NV
Alpha-Chlordane	4.58	9.16	2.14	10.7
Aroclor-1260	0.068	0.68	0.18	1.8
beta-BHC	0.4	2	0.56	2.25
delta-BHC	0.014	0.14	0.56	2.25
Dieldrin	0.015	1.27	0.0709	0.8
Endosulfan I	0.15	1.5	10	100
Endosulfan II	0.15	1.5	10	100
Endosulfan Sulfate	0.15	1.5	10	100
Endrin	0.092	0.92	0.01035	0.1035
Endrin Aldehyde	0.092	0.92	0.01	0.1
Endrin Ketone	0.092	0.92	0.01	0.1
Gamma-BHC (Lindane)	8	80	2	20
Gamma-Chlordane	4.58	9.16	2.14	10.7
Heptachlor	0.1	1	NV	NV
Heptachlor Epoxide	0.1	1	NV	NV
Methoxychlor	4	8	NV	NV

**ATTACHMENT TABLE 3-1**

**TOXICITY REFERENCE VALUES FOR TERRESTRIAL FOOD CHAIN MODELS  
SITE 2B - AREA A WETLAND  
NSB-NLON, GROTON, CONNECTICUT  
PAGE 2 OF 2**

PARAMETER	Mammal		Bird	
	NOAEL	LOAEL	NOAEL	LOAEL
<b>INORGANICS</b>				
Arsenic	2.47	4.55	2.24	4.51
Cadmium	0.8	6.9	1.5	6.3
Chromium	2.40	58.17	2.66	15.63
Copper	5.8	81.4	4.05	34.76
Lead	4.7	186.4	1.63	44.63
Mercury	0.032	0.16	0.0064	0.064
Nickel	1.70	14.77	6.71	18.57
Selenium	0.2	0.33	0.4	0.8
Silver	6.02	118.6	2.02	60.47
Vanadium	4.16	7.74	0.34	1.69
Zinc	160	320	14.49	130.9

**Notes:**

The sources of these NOAELS and LOAELS are presented in Attachment Table 3-2.

The NOAELS and LOAELS in the source table were divided by 10 if a subchronic study was the basis for the value. Also, if only a NOAEL was available, the value was multiplied by 10 to estimate the LOAEL. If only a LOAEL was available, the value was divided by 10 to estimate the NOAEL.

ATTACHMENT TABLE 3-2

SOURCES AND ENDPOINTS FOR NOAELS AND LOAELS FOR TERRESTRIAL WILDLIFE  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 1 OF 3

Parameters	Concentration (mg/kg-day)	Endpoint	Effect	Chronic/ Subchronic	Species	Primary Reference	Source of Reference
<b>Semivolatiles Organics</b>							
Acenaphthene	175	NOAEL	systemic	subchronic	mouse	USEPA, 1989a	IRIS, 2002
Acenaphthene	350	LOAEL	systemic	subchronic	mouse	USEPA, 1989a	IRIS, 2002
Acenaphthylene	700	NOAEL	mortality/BW	subchronic	mouse	USEPA, 1989a	PRC, 1996
Anthracene	1000	NOAEL	systemic	subchronic	mouse	USEPA, 1989b	IRIS, 2002
Anthracene	1000	NOAEL	reproductive	Subchronic	mouse	USEPA, 1989b	IT Corp, 1997
Benzo(a)anthracene	17.1	NOAEL	carcinogenic	acute	mouse	Clayson et al., 1968	PRC, 1996
Benzo(a)pyrene	10	LOAEL	reproductive	chronic	mouse	Mackenzie and Angevine, 1981	Sample et al., 1996
Benzo(b)fluoranthene	40	LOAEL	carcinogenic	chronic	rodent	Lo and Sandi, 1978	PRC, 1996
Benzo(g,h,i)perylene	72	LOAEL	carcinogenic	chronic	rodent	Simms and Overcash, 1983	PRC, 1996
Benzo(k)fluoranthene	72	LOAEL	carcinogenic	chronic	rodent	Lo and Sandi, 1978	PRC, 1996
Bis(2-ethylhexyl)phthalate	18 33	NOAEL	reproductive	chronic	mouse	Lamb et al., 1987	Sample et al., 1996
Bis(2-ethylhexyl)phthalate	183 3	LOAEL	reproductive	chronic	mouse	Lamb et al., 1987	Sample et al., 1996
Butylbenzylphthalate	159	NOAEL	systemic	subchronic	rat	NTP, 1985a	IRIS, 2002
Butylbenzylphthalate	470	LOAEL	systemic	subchronic	rat	NTP, 1985a	IRIS, 2002
Chrysene	17 1	NOAEL	carcinogenic	acute	mouse	Clayson et al., 1968	PRC, 1996
Dibenzo(a,h)anthracene	13 3	LOAEL	carcinogenic	chronic	mouse	Lorenz and Stewart, 1948	PRC, 1996
1,2-Dichlorobenzene	120	NOAEL	systemic	chronic	rat	NTP, 1985b	IRIS, 2002
1,4-Dichlorobenzene	300	LOAEL	mortality	chronic	rat	NTP, 1987	ATSDR, 1998
Diethyl phthalate	4583	NOAEL	reproductive	chronic	mouse	Lamb et al., 1987	Sample et al., 1996
Di-n-butylphthalate	550	NOAEL	reproductive	chronic	mouse	Lamb et al., 1987	Sample et al., 1996
Di-n-butylphthalate	1833	LOAEL	reproductive	chronic	mouse	Lamb et al., 1987	Sample et al., 1996
7,12-Dimethylbenz(a)anthracene	2	NOAEL	systemic	chronic	nestling/starlings	Trust et al., 1994	
7,12-Dimethylbenz(a)anthracene	20	LOAEL	systemic	chronic	nestling/starlings	Trust et al., 1994	
Fluoranthene	125	NOAEL	systemic	subchronic	mouse	USEPA, 1988	IRIS, 2002
Fluoranthene	250	LOAEL	systemic	subchronic	mouse	USEPA, 1988	IRIS, 2002
Fluorene	125	NOAEL	systemic	subchronic	mouse	USEPA, 1989c	IRIS, 2002
Fluorene	250	LOAEL	systemic	subchronic	mouse	USEPA, 1989c	IRIS, 2002
Hexachloroethane	1	NOAEL	systemic	subchronic	rat	Gorzinski et al., 1985	IRIS, 2002
Hexachloroethane	15	LOAEL	systemic	subchronic	rat	Gorzinski et al., 1985	IRIS, 2002
Indeno(1,2,3-cd)pyrene	72	LOAEL	carcinogenic	chronic	rodent	Simms and Overcash, 1983	PRC, 1996
Isophorone	150	NOAEL	systemic	subchronic	dog	Nor-Am Agric Products, 1972	IRIS, 2002
Isophorone	179	LOAEL	systemic	chronic	rat	NTP, 1984	IRIS, 2002
2-Methylnaphthalene	41	NOAEL	mortality	chronic	rat	Schmahl, 1955	ATSDR, 1989
2-Methylphenol	50	NOAEL	neurotoxicity	subchronic	rat	USEPA, 1996 and 1997	IRIS, 2002
2-Methylphenol	150	LOAEL	neurotoxicity	Subchronic	rat	USEPA, 1996 and 1997	IRIS, 2002
Naphthalene	71	NOAEL	systemic	subchronic	rat	BCL, 1980	IRIS, 2002
Naphthalene	142	LOAEL	systemic	subchronic	rat	BCL, 1980	IRIS, 2002
Pentachlorophenol	0 24	NOAEL	reproductive	chronic	rat	Schwetz et al., 1978	Sample et al., 1996
Pentachlorophenol	2 4	LOAEL	reproductive	chronic	rat	Schwetz et al., 1978	Sample et al., 1996
Phenol	60	NOAEL	body weight	subchronic	rat	NTP, 1983	IRIS, 2002
Phenol	120	LOAEL	body weight	subchronic	rat	NTP, 1983	IRIS, 2002
Pyrene	75	NOAEL	systemic	subchronic	mouse	USEPA, 1989c	IRIS, 2002
Pyrene	125	LOAEL	systemic	subchronic	mouse	USEPA, 1989c	IRIS, 2002
2,4,5-Trichlorophenol	100	NOAEL	systemic	subchronic	rat	McCullister, et al., 1961	IRIS, 2002
2,4,5-Trichlorophenol	300	LOAEL	systemic	subchronic	rat	McCullister, et al., 1961	IRIS, 2002
2,4,6-Trichlorophenol	4 2	NOAEL	reproductive	subchronic	rat	Exon and Koller, 1985	ASTDR, 1999
2,4,6-Trichlorophenol	42	LOAEL	reproductive	subchronic	rat	Exon and Koller, 1985	ASTDR, 1999
<b>Herbicides</b>							
Dinoseb	1	LOAEL	reproductive	chronic	rat	Dow Chemical, 1981	IRIS, 2002
2,4-D	1	NOAEL	systemic	chronic	rat	Dow Chemical, 1983	IRIS, 2002
2,4-D	5	LOAEL	systemic	chronic	rat	Dow Chemical, 1983	IRIS, 2002
2,4,5-T	3	NOAEL	reproductive	chronic	rat	Kociba et al., 1979	IRIS, 2002
2,4,5-T	10	LOAEL	reproductive	chronic	rat	Kociba et al., 1979	IRIS, 2002
<b>Pesticides</b>							
Aldrin	0 2	NOAEL	reproductive	chronic	rat	Treon and Cleveland, 1955	Sample et al., 1996
Aldrin	1	LOAEL	reproductive	chronic	rat	Treon and Cleveland, 1955	Sample et al., 1996
BHC (mixed isomers)	0 137	LOAEL	reproductive	chronic	mink	Bleavins et al., 1984	Sample et al., 1996
BHC (mixed isomers)	0 563	NOAEL	reproductive	chronic	Japanese quail	Vos et al., 1971	Sample et al., 1996
BHC (mixed isomers)	2 25	LOAEL	reproductive	chronic	Japanese quail	Vos et al., 1971	Sample et al., 1996
beta-BHC	4	NOAEL	systemic	subchronic	rat	Van Velsen et al., 1986	Sample et al., 1996
beta-BHC	20	LOAEL	systemic	subchronic	rat	Van Velsen et al., 1986	Sample et al., 1996
gamma-BHC (lindane)	8	NOAEL	reproductive	chronic	rat	Palmer et al., 1978	Sample et al., 1996
gamma-BHC (lindane)	20	LOAEL	reproductive	chronic	mallard duck	Chakravarty and Lahiri, 1986	Sample et al., 1996
Chlordane	2 14	NOAEL	mortality	chronic	red-winged blackbird	Stickel et al., 1983	Sample et al., 1996
Chlordane	10 7	LOAEL	mortality	chronic	red-winged blackbird	Stickel et al., 1983	Sample et al., 1996
Chlordane	4 58	NOAEL	reproduction	chronic	mouse	WHO, 1984	Sample et al., 1996

## ATTACHMENT TABLE 3-2

SOURCES AND ENDPOINTS FOR NOAELS AND LOAELS FOR TERRESTRIAL WILDLIFE  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 2 OF 3

Parameters	Concentration (mg/kg-day)	Endpoint	Effect	Chronic/ Subchronic	Species	Primary Reference	Source of Reference
Chlordane	9.16	LOAEL	reproduction	chronic	mouse	WHO, 1984	Sample et al., 1996
4,4'-DDT	0.8	NOAEL	reproductive	chronic	rat	Fitzhugh, 1948	Sample et al., 1996
4,4'-DDT	4	LOAEL	reproductive	chronic	rat	Fitzhugh, 1948	Sample et al., 1996
4,4'-DDT	0.052 <sup>(1)</sup>	LOAEL	reproduction	chronic	brown pelican	Anderson et al., 1975	USEPA, 1995
4,4'-DDT	0.009 <sup>(1)</sup>	NOAEL	reproduction	chronic	brown pelican	Anderson et al., 1975	USEPA, 1995
Dieldrin	0.077	NOAEL	reproduction	chronic	barn owl	Mendenhall et al., 1983	Sample et al., 1996
Dieldrin	0.2	LOAEL	reproduction	chronic	rat	Treon and Cleveland, 1955	Sample et al., 1996
Endosulfan	1.5	NOAEL	reproduction	subchronic	rat	Dikshith et al., 1984	ATSDR, 1993
Endosulfan	10	NOAEL	reproduction	chronic	gray partridge	Abiola, 1992	Sample et al., 1996
Endrin	0.92	LOAEL	reproduction	chronic	mouse	Good and Ware, 1969	Sample et al., 1996
Endrin	0.1035	LOAEL	reproduction	chronic	screech owl	Fleming et al., 1982	Sample et al., 1996
Heptachlor	1	LOAEL	reproduction	chronic	mink	Crum et al., 1993	Sample et al., 1996
Methoxychlor	4	NOAEL	reproduction	chronic	rat	Gray et al., 1988	Sample et al., 1996
Methoxychlor	8	LOAEL	reproduction	chronic	rat	Gray et al., 1988	Sample et al., 1996
Toxaphene	8	NOAEL	reproduction	chronic	rat	Kennedy et al., 1973	Sample et al., 1996
Aroclor-1242	0.685	LOAEL	reproduction	chronic	mink	Bleavins et al., 1980	Sample et al., 1996
Aroclor-1242	0.41	NOAEL	reproduction	chronic	screech owl	McLane and Hughes, 1980	Sample et al., 1996
Aroclor-1248	0.1	LOAEL	reproductive	chronic	rhesus monkey	Barsotti et al., 1976	Sample et al., 1996
Aroclor-1254	1.8	LOAEL	reproductive	chronic	pheasant	Dahlgren et al., 1972	Sample et al., 1996
Aroclor-1254	0.68	LOAEL	reproduction	chronic	mouse	McCoy et al., 1995	Sample et al., 1996
<b>Dioxins/Furans</b>							
1,2,3,6,7,8-HXCDF	0.0016	NOAEL	systemic	subchronic	rat	Poiger et al., 1989	Sample et al., 1996
1,2,3,6,7,8-HXCDF	0.016	LOAEL	systemic	subchronic	rat	Poiger et al., 1989	Sample et al., 1996
1,2,3,4,8-PCDF	0.48	NOAEL	systemic	subchronic	rat	Poiger et al., 1989	Sample et al., 1996
1,2,3,7,8-PCDF	0.0016	NOAEL	systemic	subchronic	rat	Poiger et al., 1989	Sample et al., 1996
1,2,3,7,8-PCDF	0.016	LOAEL	systemic	subchronic	rat	Poiger et al., 1989	Sample et al., 1996
2,3,4,7,8-PCDF	0.00016	NOAEL	systemic	subchronic	rat	Poiger et al., 1989	Sample et al., 1996
2,3,4,7,8-PCDF	0.0016	LOAEL	systemic	subchronic	rat	Poiger et al., 1989	Sample et al., 1996
2,3,7,8-TCDD	0.000001	NOAEL	reproduction	chronic	rat	Murray et al., 1979	Sample et al., 1996
2,3,7,8-TCDD	0.00001	LOAEL	reproduction	chronic	rat	Murray et al., 1979	Sample et al., 1996
2,3,7,8-TCDD	0.000014	NOAEL	reproduction	chronic	pheasant	Nosek et al., 1992	Sample et al., 1996
2,3,7,8-TCDD	0.00014	LOAEL	reproduction	chronic	pheasant	Nosek et al., 1992	Sample et al., 1996
<b>Explosives</b>							
1,3-Dinitrobenzene	0.4	NOAEL	reproduction	subchronic	rat	Cody et al., 1981	IRIS, 2002
1,3-Dinitrobenzene	1.07	NOAEL	reproductive	subchronic	rat	Cody et al., 1981	IRIS, 2002
2,4-Dinitrotoluene	0.2	NOAEL	neurotoxicity	chronic	dog	Ellis et al., 1985	IRIS, 2002
2,4-Dinitrotoluene	1.5	LOAEL	neurotoxicity	chronic	dog	Ellis et al., 1985	IRIS, 2002
m, o, and p-Nitrotoluene	200	LOAEL	systemic	chronic	rat	Ciss et al., 1980	HEAST, 1997
HMX	30	NOAEL	mortality	subchronic	mouse	Everett and Maddock, 1985	Talmage et al., 1999
HMX	75	LOAEL	mortality	subchronic	mouse	Everett and Maddock, 1985	Talmage et al., 1999
RDX	7	NOAEL	reproduction	chronic	mouse	Lish et al., 1984	Talmage et al., 1999
RDX	35	LOAEL	reproduction	chronic	mouse	Lish et al., 1984	Talmage et al., 1999
Tetryl	13	NOAEL	reproduction	subchronic	rat	Reddy et al., 1994	Talmage et al., 1999
Tetryl	62	LOAEL	reproduction	subchronic	rat	Reddy et al., 1994	Talmage et al., 1999
1,3,5-Trinitrobenzene	2.68	NOAEL	systemic	chronic	rat	Reddy et al., 1996	IRIS, 2002
1,3,5-Trinitrobenzene	13.31	LOAEL	systemic	chronic	rat	Reddy et al., 1996	IRIS, 2002
2,4,6-Trinitrotoluene	160	LOAEL	reproductive	subchronic	rat	Dilley et al., 1982	Talmage et al., 1999
2,4,6-Trinitrotoluene	7	NOAEL	systemic	subchronic	bobwhite quail	Gogal et al., (in draft)	USCHPPM, 2000
2,4,6-Trinitrotoluene	178	LOAEL	systemic	subchronic	bobwhite quail	Gogal et al., (in draft)	USCHPPM, 2000
<b>Inorganics</b>							
Aluminum	109.7	NOAEL	reproductive	chronic	ringed dove	Carrere et al., 1986	Sample et al., 1996
Aluminum	19.3	LOAEL	reproductive	chronic	mouse	Ondreich et al., 1966	Sample et al., 1996
Antimony	1.25	LOAEL	lifespan	chronic	mouse	Schroeder et al., 1968	Sample et al., 1996
Arsenic	1.261	LOAEL	reproductive	chronic	mouse	Schroeder and Mitchner, 1971	Sample et al., 1996
Arsenic	2.46	NOAEL	mortality	chronic	brown-headed cowbird	USFWS, 1969	Sample et al., 1996
Arsenic	7.38	LOAEL	mortality	chronic	brown-headed cowbird	USFWS, 1969	Sample et al., 1996
Barium	5.1	NOAEL	growth	chronic	rat	Perry et al., 1983	Sample et al., 1996
Barium	198	LOAEL	mortality	subchronic	rat	Borzelleca et al., 1988	Sample et al., 1996
Barium	208.26	NOAEL	mortality	subchronic	chicks	Johnson et al., 1960	Sample et al., 1996
Barium	416.53	LOAEL	mortality	subchronic	chicks	Johnson et al., 1960	Sample et al., 1996
Beryllium	0.66	NOAEL	systemic	chronic	rat	Schroeder and Mitchner, 1975b	Sample et al., 1996
Cadmium	1	NOAEL	reproductive	chronic	rat	Sutou et al., 1980	Sample et al., 1996
Cadmium	10	LOAEL	reproductive	chronic	rat	Sutou et al., 1980	Sample et al., 1996
Cadmium	1.45	NOAEL	reproductive	chronic	mallard duck	White and Finely, 1978	Sample et al., 1996
Cadmium	20	LOAEL	reproductive	chronic	mallard duck	White and Finely, 1978	Sample et al., 1996
Chromium(III)	1	NOAEL	reproductive	chronic	black duck	Haselline et al., 1985	Sample et al., 1996
Chromium(III)	5	LOAEL	reproductive	chronic	black duck	Haselline et al., 1985	Sample et al., 1996

ATTACHMENT TABLE 3-2

SOURCES AND ENDPOINTS FOR NOAELS AND LOAELS FOR TERRESTRIAL WILDLIFE  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 3 OF 3

Parameters	Concentration (mg/kg-day)	Endpoint	Effect	Chronic/ Subchronic	Species	Primary Reference	Source of Reference
Chromium(VI)	3.28	NOAEL	BW/food cons	chronic	rat	Mackenzie et al., 1958	Sample et al., 1996
Chromium(VI)	131.4	LOAEL	mortality	subchronic	rat	Steven et al., 1976	Sample et al., 1996
Cobalt	12	LOAEL	growth	chronic	rat	Domingo et al., 1985	Eng. Field Activity, 1998
Copper	11.71	NOAEL	reproductive	chronic	mink	Aulerich et al., 1982	ATSDR, 1989
Copper	15.14	LOAEL	reproductive	chronic	mink	Aulerich et al., 1982	ATSDR, 1989
Copper	46.97	NOAEL	mortality	chronic	chicks	Mehring et al., 1960	Sample et al., 1996
Copper	61.72	LOAEL	mortality	chronic	chicks	Mehring et al., 1960	Sample et al., 1996
Cyanide	68.7	NOAEL	reproductive	chronic	rat	Tewe and Maner, 1981	Sample et al., 1996
Iron	500	LOAEL	unknown	chronic	rabbit	NAS, 1980	
Iron	1000	LOAEL	unknown	chronic	poultry	NAS, 1980	
Lead	8	NOAEL	reproductive	chronic	rat	Azar et al., 1973	Sample et al., 1996
Lead	80	LOAEL	reproductive	chronic	rat	Azar et al., 1973	Sample et al., 1996
Lead	1.13	NOAEL	reproductive	chronic	Japanese quail	Edens et al., 1976	Sample et al., 1996
Lead	11.3	LOAEL	reproductive	chronic	Japanese quail	Edens et al., 1976	Sample et al., 1996
Manganese	977	NOAEL	growth	chronic	Japanese quail	Laskey and Edens, 1985	Sample et al., 1996
Manganese	88	NOAEL	reproductive	chronic	rat	Laskey et al., 1982	Sample et al., 1996
Manganese	284	LOAEL	reproductive	chronic	rat	Laskey et al., 1982	Sample et al., 1996
Mercury	0.064	LOAEL	reproductive	chronic	mallard duck	Heinz, 1979	Sample et al., 1996
Mercury	0.032	NOAEL	reproductive	chronic	rat	Verschuuren et al., 1976	Sample et al., 1996
Mercury	0.16	LOAEL	reproductive	chronic	rat	Verschuuren et al., 1976	Sample et al., 1996
Nickel	40	NOAEL	reproductive	chronic	rat	Ambrose et al., 1976	Sample et al., 1996
Nickel	80	LOAEL	reproductive	chronic	rat	Ambrose et al., 1976	Sample et al., 1996
Nickel	77.4	NOAEL	mortality	chronic	mallard duck	Cain and Pafford, 1981	Sample et al., 1996
Nickel	107	LOAEL	mortality	chronic	mallard duck	Cain and Pafford, 1981	Sample et al., 1996
Selenium	0.4	NOAEL	reproductive	chronic	mallard duck	Heinz et al., 1989	Sample et al., 1996
Selenium	0.8	LOAEL	reproductive	chronic	mallard duck	Heinz et al., 1989	Sample et al., 1996
Selenium	0.2	NOAEL	reproductive	chronic	rat	Rosenfeld and Beath, 1954	Sample et al., 1996
Selenium	0.33	LOAEL	reproductive	chronic	rat	Rosenfeld and Beath, 1954	Sample et al., 1996
Silver	54.4	LOAEL	survival	chronic	chicks	Petersen and Jensen, 1975	
Silver	18.1	LOAEL	systemic	subchronic	mouse	Rungby and Danscher, 1984	ATSDR, 1989
Thallium	0.74	LOAEL	reproductive	subchronic	rat	Formigli et al., 1986	Sample et al., 1996
Tin	23.4	NOAEL	reproductive	chronic	mouse	Davis et al., 1987	Sample et al., 1996
Tin	35	LOAEL	reproductive	chronic	mouse	Davis et al., 1987	Sample et al., 1996
Tin	6.76	NOAEL	reproductive	chronic	Japanese quail	Schlatterer et al., 1993	Sample et al., 1996
Tin	16.9	LOAEL	reproductive	chronic	Japanese quail	Schlatterer et al., 1993	Sample et al., 1996
Vanadium	2.1	LOAEL	reproductive	chronic	rat	Domingo et al., 1986	Sample et al., 1996
Vanadium	11.38	NOAEL	mortality, BW	chronic	mallard duck	White and Dieter, 1978	Sample et al., 1996
Zinc	160	NOAEL	reproductive	chronic	rat	Schlucker and Cox, 1968	Sample et al., 1996
Zinc	320	LOAEL	reproductive	chronic	rat	Schlucker and Cox, 1968	Sample et al., 1996
Zinc	14.49	NOAEL	reproductive	chronic	white leghorn hen	Stahl et al., 1990	Sample et al., 1996
Zinc	130.9	LOAEL	reproductive	chronic	white leghorn hen	Stahl et al., 1990	Sample et al., 1996

Notes: The NOAELS and LOAELS for the PAHs that do not have values are based on the benzo(a)pyrene values

The NOAELS and LOAELS for the PAHs for birds were based on 7,12-dimethylbenz(a)anthracene

1 - The LOAEL of 0.052 mg/kg-day was the recalculated LOAEL for the referenced study by assuming a lag time for the reduction of DDT (see USEPA, 1995)

The NOAEL of 0.009 was calculated by dividing the original LOAEL of the referenced study (0.027 mg/kg-day) by an uncertainty factor of 3

## SOURCES AND ENPOINTS FOR NOAELS AND LOAELS FOR TERRESTRIAL WILDLIFE

Page 1 of 8

Abiola, F.A. 1992. "Ecotoxicity of Organochloride Insecticides: Effects of Endosulfan on Birds Reproduction and Evaluation of its Induction Effects in Partridge, *Perdix perdix*". L. Rev. Vet. Med. 143:443-450.

Agency for Toxic Substances and Disease Registry (ATSDR). 1999. Toxicological profile for chlorophenols. Atlanta, GA: U.S. Department of Health and Human Services, Public Health Service.

Agency for Toxic Substances and Disease Registry (ATSDR). 1998. Toxicological profile for 1,4-dichlorobenzene. Atlanta, GA: U.S. Department of Health and Human Services, Public Health Service.

Agency for Toxic Substances and Disease Registry (ATSDR). 1993. Toxicological Profile for Endosulfan. Prepared by Clement Associates for the US Department of Health and Human Services.

Agency for Toxic Substances and Disease Registry (ATSDR). 1991. Toxicological Profile for Arsenic - Draft. Prepared by Life Systems, Inc, for the US Department of Health and Human Services.

Agency for Toxic Substances and Disease Registry (ATSDR). 1989. Toxicological Profile for Copper - Draft. Prepared by Syracuse Research Corporation for the US Department of Health and Human Services.

Agency for Toxic Substances and Disease Registry (ATSDR). 1989. Toxicological Profile for Naphthalene/2-methylnaphthalene - Draft. Prepared by Life Systems, Inc, for the US Department of Health and Human Services.

Agency for Toxic Substances and Disease Registry (ATSDR). 1989. Toxicological Profile for Polycyclic Aromatic Hydrocarbons - Draft. Prepared by Clement Associates for the US Department of Health and Human Services.

Agency for Toxic Substances and Disease Registry (ATSDR). 1989. Toxicological Profile for Silver - Draft. Prepared by Clement Associates for the US Department of Health and Human Services.

Ambrose, A.M., D.S. Larson, J.R. Borzelleca and G.R. Hennigar, Jr. 1976. "Long-Term Toxicologic Assessment of Nickel in Rats and Dogs". J. Food Science Technology. 13:181-187. Cited in IRIS, Accessed September 2002 (Nickel).

Anderson, D. W., R. W. Risebrough, L. A. Woods, Jr., L. R. DeWeese, and W. G. Edgecomb. 1975. Brown pelicans: improved reproduction off the southern California coast. *Science* 190: 806-808.

Aulerich, R.J., R.K., Ringer, M.R., Bleavins, et. al. 1982. "Effects of Supplemental Dietary Copper on Growth, Reproductive Performance and Kit Survival of Standard Dark Mink and the Acute Toxicity of Copper to Mink". J. Animal Sci. 55:337-343.

Azar, A., H.J. Trochimowicz and M.E. Maxfield. 1973. "Review of Lead Studies in Animals Carried out at Haskell Laboratory - Two Year Feeding Study and Response to Hemorrhage Study". In Barth D., A. Berlin, R. Engel, P. Recht and J. Smeets, Ed. Environmental Health Aspects of Lead: Proceedings International Symposium; October 1972; Amsterdam, The Netherlands. Commission of the European Communities, Luxemburg. p. 199-208.

Barsotti, D. A., R. J. Marlar and J. R. Allen. 1976. Reproductive dysfunction in Rhesus monkeys exposed to low levels of polychlorinated biphenyls (Aroclor 1248). *Fd. Cosmet. Toxicol.* 14: 99-103.

Battelle's Columbus Laboratories (BCL). 1980. Unpublished subchronic toxicity study: Naphthalene (C52904), Fischer 344 rats. Prepared by Battelle Laboratories under NTP Subcontract No. 76-34-106002. Available from the Center for Environmental Research Information, (301) 345-2870.

## SOURCES AND ENPOINTS FOR NOAELS AND LOAELS FOR TERRESTRIAL WILDLIFE

### Page 2 of 8

Bleavins, M.R., C.S. Sisodia, and T.K. Mukkur. 1980. "The Effects of Methyl Mercury, Tetrethyl Lead, and Sodium Arsenite on the Humoral Immune Response in Mice". *Toxicol. Appl. Pharmacol.* 52:245-254.

Bleavins, M. R., R. J. Aulerich, and R. K. Ringer. 1980. Polychlorinated biphenyls (Aroclors 1016 and 1242): Effect on survival and reproduction in mink and ferrets. *Arch. Environ. Contam. Toxicol.* 9: 627-635.

Bleavins, M. R., R. J. Aulerich, and R. K. Ringer. 1984. Effects of chronic dietary hexachlorobenzene exposure on the reproductive performance and survivability of mink and European ferrets. *Arch. Environ. Contam. Toxicol.* 13: 357-365.

Borzelleca, J. F., L. W. Condie, Jr., and J. L. Egle, Jr. 1988. Short-term toxicity (one-and ten-day gavage) of barium chloride in male and female rats. *J. American College of Toxicology.* 7: 675-685.

Cain, B. W. and E. A. Pafford. 1981. Effects of dietary nickel on survival and growth of Mallard ducklings. *Arch. Environm. Contam. Toxicol.* 10: 737-745.

Carriere, D., K. Fischer, D. Peakall, and P. Angehrn. 1986. Effects of dietary aluminum in combination with reduced calcium and phosphorus on the ring dove (*Streptopelia risoria*). *Water, Air, and Soil Poll.* 30: 757-764.

Chakravarty, S. and P. Lahiri. 1986. Effect of lindane on eggshell characteristics and calcium level in the domestic duck. *Toxicology.* 42: 245-258.

Ciss, M., N. Huyen, H. Dutertre, N. Phu-Lich, and R. Truhaut. 1980. Toxicologic study of nitrotoluenes: long term toxicity. *Dakar Med.* 25(4):293-302. French.

Clayson et al., 1968. IARC Monographs 1972-present. 1973 V3-55. Cited in Hazardous Substances Databank, 1995.

Cody, T.E., S. Witherup, L. Hastings, K. Stemmes, and R.T. Christian. 1981. "1,3-Dinitrobenzene: Toxic Effects in Vivo and in Vitro". *J. Toxicol. Environ. Health.* 7(5): 829-847.

Crum, J. A., S. J. Bursian, R. J. Aulerich, P. Polin, and W. E. Braselton. 1993. The reproductive effects of dietary heptachlor in mink (*Mustela vison*). *Arch. Environ. Contam. Toxicol.* 24: 156-164.

Dahlgren, R.B., R.L. Linder, and C.W. Carlson. 1972. "Polychlorinated Biphenyls: Their Effects on Pinned Pheasants". *Environ. Health Perspect.* 1:89-101.

Davis, A., R. Barale, G. Brun et al., 1987. Evaluation of the genetic and embryotoxic effects of bis(tri-n-butyltin)oxide (TBTO), a broad-spectrum pesticide, in multiple in vivo and in vitro short-term tests. *Muta. Res.* 188:65-95.

Dikshith, T.S.S., R.B. Raizada, M.K. Srivastava, and B.S. Kaphalia. 1984. "Response of Rats to Repeated Oral Administration of Endosulfan". *Ind. Health.* 22:295-304.

Dilley, J.V., C.A. Tyson, R.J. Spanggord, D.P. Sasmore, G.W. Newell, and J.C. Dacre. 1982. Short-term oral toxicity of 2,4,6-trinitrotoluene in mice, rats, and dogs. *J. Toxicol Environ Health* 9:565-585.

Domingo, J.L., J.L. Paternaia, J.M. Llobet, and J. Corbella. 1986. "Effects of Vanadium on Reproduction, Gestation, Parturition and Lactation in Rats upon Oral Administration". *Life Sci.* 39:819-824.

Domingo, J.L., J.L. Paternain, J.M. Llobet, et al., 1985. "Effects of Cobalt on Postnatal Development and Late Gestation in Rats Upon Oral Administration." *Rev. Esp. Fisiol.* Volume 41. Pages 293-298.

## SOURCES AND ENPOINTS FOR NOAELS AND LOAELS FOR TERRESTRIAL WILDLIFE

Page 3 of 8

Dow Chemical Company. 1983. Accession No. 251473. Available from EPA. Write to FOI, EPA, Washington, DC 20460.

Dow Chemical Company. 1981. MRID No. 00152675. Available from EPA. Write to FOI, EPA, Washington D.C. 20460.

Edens, F., W. E. Benton, S. J. Bursian, and G. W. Morgan. 1976. Effect of Dietary Lead on Reproductive Performance in Japanese Quail, *Coturnix coturnix japonica*. *Toxicol. Appl. Pharmacol.* 38: 307-314.

Ellis, H.V., C.B. Hong, C.C. Lee, J.C. Dacre and J.P. Glennon. 1985. Subchronic and chronic toxicity studies of 2,4-dinitrotoluene. Part I. Beagle dogs. *J. Am. College Toxicol.* 4(4): 233-242.

Engineering Field Activity, West, 1998. Development to Toxicity Reference Values for Conducting Ecological Risk Assessments at Naval Facilities in California, Interim Final. EFA West, Naval Facilities Engineering Command, United States Navy. San Bruno, California.

Everett D.J. and S.M. Maddock. 1985. HMX: 13-week toxicity study in mice by dietary administration. AD A171602. Final report to the U.S. Army. Inveresk Research International, Ltd., Musselburgh, Scotland.

Exon, J.H. and L.D. Keller. 1985. Toxicity of 2-chlorophenol, 2,4-dichlorophenol, and 2,4,6-trichlorophenol, In: Jolley RL, ed. *Water Chlorination: Chemistry, environmental impact and health effects*. Volume 5. Fifth Conference on Water Chlorination: Environmental Impact and Health Effects, Williamsburg, VA, June 1984, 307-330.

Fitzhugh, O.G., 1948. "Use of DDT Insecticides on Food Products". *Ind. Eng. Chem.* 40:704-705.

Fleming, W. J., M. A. Ross McLane, E. Cromartie. 1982. Endrin decreases screech owl productivity. *J. Wildl. Manage.* 46:462-468

Formigli, L., R. Scelsi, P. Poggi, C. Gregotti, A. DiNucci, E. Sabbioni, L. Gottardi, and L. Manzo. 1986. "Thallium-Induced Testicular Toxicity in the Rat". *Environ. Res.* 40:531-539.

Good, E.E., and G.W. Ware. 1969. "Effects of Insecticides on Reproduction in the Laboratory Mouse, IV. Endrin and Dieldrin". *Toxicol. Appl. Pharmacol.* 14:201-203.

Gorzinski, S.J., R.J. Nolan, S.B. McCollester, R.J. Kociba and J.L. Mattsson. 1985. Subchronic oral toxicity, tissue distribution and clearance of hexachloroethane in the rat. *Drug Chem. Toxicol.* 8(3): 155-169.

Gray, L. E., Jr., J. Ostby, R. Sigmon, J. Ferrell, G. Rehnberg, R. Linder, R. Cooper, J. Goldman, and J. Laskey. 1988. The development of a protocol to assess reproductive effects of toxicants in the rat. *Reprod. Toxicol.* 2: 281-287.

Haseltine, S.D., L. Sileo, D.J. Hoffman, and B.D. Mulhern. 1985. Effects of chromium on reproduction and growth in black ducks.

Health Effects Assessment Summary Tables (HEAST). 1994 and 1997. USEPA, Office of Solid Waste and Emergency Response. EPA 540/R-94/020.

Heinz, G.H., D.J. Hoffman, and L.G. Gold. 1989. "Impaired Reproduction of Mallards Fed and Organic Form of Selenium". *J. Wildl. Mgmt.* 53: 418-428.

Heinz, G. H. 1979. "Methyl Mercury: Reproductive and Behavioral Effects on Three Generations of Mallard Ducks." *J. Wildl. Mgmt.* 43: 394-401.

## SOURCES AND ENPOINTS FOR NOAELS AND LOAELS FOR TERRESTRIAL WILDLIFE

Page 4 of 8

IT Corporation. 1997 (November). Predictive Ecological Risk Assessment Methodology. Environmental Restoration Program, Sandia National Laboratory, New Mexico. Sandia National Laboratory. Albuquerque, NM. Appendix A, Table A.1.

Johnson, D., Jr., A. L. Mehring, Jr., and H. W. Titus. 1960. Tolerance of chickens for barium. *Proc. Soc. Exp. Biol. Med.* 104: 436-438.

Kennedy, G. L., Jr., J. P. Frawley., and J. C. Calandra. 1973. Multigeneration reproductive effects of three pesticides. *Toxicol. Appl. Pharmacol.* 25: 589-596.

Kociba, R.J., D.G. Keyes, R.W. Lisowe, et al. 1979. Results of a two-year chronic toxicity and oncogenic study of rats ingesting diets containing 2,4,5- trichlorophenoxyacetic acid (2,4,5-T). *Food Cosmet. Toxicol.* 17: 205-221.

Lamb, J.C., IV, R.E. Chapin, J. Teague, A.D. Lawton, and J.R. Reel. 1987. "Reproductive Effects of Four Phthalic Acid Esters in the Mouse". *Toxicol. Appl. Pharmacol.* 88:255-269.

Laskey, J. W., and F. W. Edens. 1985. Effects of chronic high-level manganese exposure on male behavior in the Japanese Quail (*Coturnix coturnix japonica*). *Poult. Sci.* 64: 579-584.

Laskey, J.W., G.L. Rehnberg, J.F. Hein, and S.D. Carter. 1982. "Effects of Chronic Manganese (MN<sub>3</sub>O<sub>4</sub>) Exposure on Selected Reproductive Parameters in Rats.: *J. Toxicol. Environ. Health.* 9:677-687.

Lincer, J.L. 1975. DDE-Induced Eggshell –Thinning in the American kestrel: A Comparison of the Field Situation and Laboratory Results. *J. Appl. Ecology* 12:781-793.

Lish P.M., B.S. Levine, E.M. Furedi, E.M Sagartz, and V.S. Rac. 1984. Determination of the chronic mammalian toxicological effects of RDX: twenty-four month chronic toxicity/carcinogenicity study of hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) in the B6C3F1 hybrid mouse. Phase VI. Vol. 1. AD A160774. IIT Research Institute, Chicago, IL. U.S. Army Medical Research and Development Command, Frederick, MD.

Lo, MT and Sandi, E. 1978. Polycyclic Aromatic Hydrocarbons in Foods. *Residue Review*, 69:35-86. Cited in U.S. Fish and Wildlife Service. Polycyclic Aromatic Hydrocarbons Hazards to Fish, Wildlife, and Invertebrates: A Synoptic Review. May 1987.

Lorenz E. and H.L. Stewart. 1948. Tumors of alimentary tract in mice fed carcinogenic hydrocarbons in mineral oil emulsions. *J. Natl. Cancer Inst.* 9:173.

MacKenzie, R.D., R.U. Byerrum, C.F. Decker, C.A. Hoppert and R.F. Langham. 1958. "Chronic Toxicity Studies. II. Hexavalent and Trivalent Chromium Administered in Drinking Water to Rats". *Am. Med. Assoc. Arch. Ind. Health.* 18:232-234.

MacKenzie, K.M., and D.M. Angevine. 1981. "Infertility in Mice Exposed in Utero to Benzo(a)pyrene. *Biol. Reprod.* 24:183-191.

McCoy, G, M. F. Finlay, A. Rhone, K. James, and G. P. Cobb. 1995. Chronic polychlorinated biphenyls exposure on three generations of oldfield mice (*Peromyscus polionotus*): effects on reproduction, growth, and body residues. *Arch. Environ. Contam. Toxicol.* 28: 431-435

McCollister, D.D., D.T. Lockwood and V.K. Rowe. 1961. Toxicologic information on 2,4,5-trichlorophenol. *Toxicol. Appl. Pharmacol.* 3: 63-70.

## SOURCES AND ENPOINTS FOR NOAELS AND LOAELS FOR TERRESTRIAL WILDLIFE

Page 5 of 8

McLane, M.A.R., and D.L. Hughes. 1980. "Reproductive Success of Screech Owls Fed Aroclor-1248". *Arch Environ. Contam. Toxicol.* 9:661-665.

Mehring, A. L. Jr., J. H. Brumbaugh, A. J. Sutherland, and H. W. Titus. 1960. The tolerance of growing chickens for dietary copper. *Poult. Sci.* 39: 713-719.

Mendenhall, V. M., E. E. Klaas, and M. A. R. McLane. 1983. Breeding success of barn owls (*Tyto alba*) fed low levels of DDE and dieldrin. *Arch. Environ. Contam. Toxicol.* 12: 235-240.

Murray, F. J., F. A. Smith, K. D. Nitschke, C. G. Humiston, R. J. Kociba, and B. A. Schwetz. 1979. Three-generation reproduction study of rats given 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) in the diet. *Toxicol. Appl. Pharmacol.* 50: 241-252.

National Academy of Sciences (NAS). 1980. Mineral Tolerance of Domestic Animals. National Research Council, Commission on Natural Resources, Committee on Animal Nutrition.

Nor-Am Agricultural Products, Inc. 1972. MRID No. 00123976. Available from EPA. Write to FOI, EPA, Washington, DC 20460.

Nosek, J. A., S. R. Craven, J. R. Sullivan, S. S. Hurley, and R. E. Peterson. 1992. Toxicity and reproductive effects of 2,3,7,8-tetrachlorodibenzo-p-dioxin in ring-necked pheasants. *J. Toxicol. Environ. Health.* 35: 187-198.

NTP (National Toxicology Program). 1987. Toxicology and carcinogenesis studies of 1,4-Dichlorobenze (CAS No. 106-46-7) in F344/N rats and B6C3F1 mice (gave studies). Research Triangle Park, NC: National Toxicology Program. NTP TR 319. NIH Publication No. 87-2575.

NTP (National Toxicology Program). 1985a. Twenty-Six Week Subchronic Study and Modified Mating Trial in F344 Rats. Butyl Benzyl Phthalate. Final Report. Project No. 12307-02, -03. Hazelton Laboratories America, Inc. Unpublished Study.

NTP (National Toxicology Program). 1985b. Toxicology and carcinogenesis studies of 1,2-dichlorobenzene (o-dichlorobenzene) (CAS No. 95-50-1) in F344/N rats and B6C3F1 mice (gavage studies). NTP TR 255. NIH Publ. No. 86-2511.

NTP (National Toxicology Program). 1984. NTP Technical Report on the Toxicology and Carcinogenesis Studies of Isophorone in F344/N Rats and B63F1 Mice (Gavage). NIH Publication No. 84-2547, NTP-83-168.

NTP (National Toxicology Program). 1983. Teratologic evaluation of phenol in CD rats and mice. Report prepared by Research Triangle Institute, Research Triangle Park, NC. NTIS PB83-247726. Gov. Rep. Announce. Index. 83(25): 6247.

Ondreicka, R.E., E. Ginter, and J. Kortus. 1966. Chronic Toxicity of Aluminum in rats and Mice and its Effects on Phosphorus Metabolism." *Brit. J. Indust. Med.* 23:305-313.

Palmer, A. K., D. D. Cozens, E. J. F. Spicer, and A. N. Worden. 1978. Effects of lindane upon reproductive functions in a 3-generation study in rats. *Toxicology.* 10: 45-54.

Perry, H. M., E. F. Perry, M. N. Erlanger, and S. J. Kopp. 1983. Cardiovascular effects of chronic barium ingestion. In: Proc. 17th Ann. Conf. Trace Substances in Environ. Health, vol. 17. U. of Missouri Press, Columbia, MO.

## SOURCES AND ENPOINTS FOR NOAELS AND LOAELS FOR TERRESTRIAL WILDLIFE

Page 6 of 8

- Petersen, R.P. and L.S. Jensen. 1975. Interrelationship of dietary silver with copper in chick. *Poultry Science*, 54(3): 771-775.
- Poiger, H., N. Pluess, and C. Schlatter. 1989. Subchronic toxicity of some chlorinated dibenzofurans to rats. *Chemosphere*. 18: 265-275.
- PRC Environmental Management, Inc. 1996. (August). Region 5 Ecological Data Quality Levels. Final Report. United States Environmental Protection Agency. Chicago, Illinois.
- Reddy, T.V., F.B. Daniel, G.R. Olson, B. Wiechman and G. Reddy. 1996. Chronic toxicity studies of 1,3,5-trinitrobenzene in Fischer 344 rats. U.S. Army, Fort Detrick, MD. (Final Report)
- Reddy, T.V., F.B. Daniel, M. Robinson, G.R. Olson, B. Weichman, and G. Reddy. 1994. Subchronic toxicity studies on 1,3,5-trinitrobenzene, 1,3-dinitrobenzene, and tetryl in rats: subchronic toxicity evaluation of *N*-methyl-*N*-2,4,6-tetranitroaniline (tetryl) in Fischer-344 rats. U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH. National Technical Information Service, Springfield, VA.
- Rosenfeld, I., and O.A. Beath. 1954. Selenium: Geobotany, Biochemistry, Toxicity and Nutrition. Academic Press, New York. p. 198-208.
- Rungby, J., and G. Danscher. 1984. "Hypoactivity in Silver Exposed Mice." *Acta. Pharmacol. et Toxicol.* 55:398-401. Cited in ASTDR, 1989 (Silver).
- Sample, B.E., D.M. Opresko, and G.W. Suter II. 1996. Toxicological Benchmarks for Wildlife: 1996 Revision. Oak Ridge National Laboratory. June. ES/ER/TM-86/R3.
- Schlatterer, B., T. M. M. Coenen, E. Ebert, R. Grau, V. Hilbig, and R. Munk. 1993. "Effects of Bis(Tri-*n*-butyltin)Oxide in Japanese Quail Exposed During Egg Laying Period: an Interlaboratory Comparison Study." *Arch. Environ. Contam. Toxicol.* 24: 440-448.
- Schlicker, S.A. and D.H. Cox. 1968. "Maternal Dietary Zinc, and Development and Zinc, Iron, and Copper Content of the Rat Fetus". *J. Nutr.* 95:287-294.
- Schmahl, D. 1955. "The Testing of Naphthalene and Anthracene for Carcinogenic Effects on Rats". *Z. Krebsforsch* 60:697-710.
- Schroeder, H.A. and M. Mitchener. 1975. Life-term studies in rats: effects of aluminum, barium, beryllium, and tungsten. *J. Nutr.* 105: 421-427.
- Schroeder, H.A. and M. Mitchener. 1971. "Toxic Effects of Trace Elements on the Reproduction of Mice and Rats". *Arch. Environ. Health* 23:102-106.
- Schroeder, H. A., M. Mitchener, J. J. Balassa, M. Kanisawa, and A. P. Nason. 1968. Zirconium, niobium, antimony, and fluorine in mice: effects on growth, survival and tissue levels. *J. Nutr.* 95: 95-101.
- Schwetz, B. A., J. F. Quast, P. A. Keeler, C. G. Humiston, and R. J. Kociba. 1978. Results of two-year toxicity and reproduction studies on pentachlorophenol in rats. pp 301-309 in K. R. Rao, ed., *Pentachlorophenol: Chemistry, Pharmacology, and Environmental Toxicology*. Plenum Press, New York. 401 pp.
- Simms, R.C. and R.M. Overcash. 1983. Fate of Polynuclear Aromatic Compounds In Soil Plant Systems. *Residue Review*, 88: 1-68. Cited in U.S. Fish and Wildlife Service. Polycyclic Aromatic Hydrocarbons Hazards to Fish, Wildlife, and Invertebrates: A Synoptic Review. May 1987.

## SOURCES AND ENPOINTS FOR NOAELS AND LOAELS FOR TERRESTRIAL WILDLIFE

Page 7 of 8

Stahl, J. L., J. L. Greger, and M. E. Cook. 1990. Breeding-hen and progeny performance when hens are fed excessive dietary zinc. *Poult. Sci.* 69: 259-263.

Steven, J. D., L. J. Davies, E. K. Stanley, R.A. Abbott, M. Ihnat, L. Bidstrup, and J. F. Jaworski. 1976. Effects of chromium in the Canadian environment. NRCC No. 151017. 168 pp.

Stickel, L.F., W.H. Stickel, R.A. Dryland, and D.L. Hughes. 1983. "Oxychlorane, HCS-3260, and Nonachlor in Birds: Lethal Residues and Loss Rates". *J. Toxicol. Environ. Health.* 12:611-622.

Sutou, S., K. Yamamoto, H. Sendota, and M. Sugiyama. 1980. Toxicity, fertility, teratogenicity, and dominant lethal tests in rats administered cadmium subchronically. I. Fertility, teratogenicity, and dominant lethal tests. *Ecotoxicol. Environ. Safety.* 4:51-56.

Talmage, Sylvia S., Dennis M. Opresko, Christopher J. Maxwell, Christopher J.E. Welsh, F. Michael Cretella, Patricia H. Reno, and F. Bernard Daniel, 1999. "Nitroaromatic Munition Compounds: Environmental Effects and Screening Values." *Rev. Environ. Contam. Toxicol.* 161:1-156.

Tewe, O.O., and J.H. Maner. 1981. "Long-Term and Carry-Over Effect of Dietary Inorganic Cyanide (KCN) in the Life Cycle Performance and Metabolism of Rats". *Toxicol. Appl. Pharmacol.* 58:1-7.

Treon, J. F. and F. P. Cleveland. 1955. Toxicity of certain chlorinated hydrocarbon insecticides for laboratory animals, with special reference to aldrin and dieldrin. *Ag. Food Chem.* 3: 402-408.

Trust, K.A., A. Fairbrother, and M.J. Hooper. 1994. Effects of 7,12-Dimethylbenz(a)anthracene on Immune Function and Mixed-Function Oxygenase Activity in the European Starling. *Environ. Tox. And Chem.*, Vol. 13, No. 5, pp. 821-830.

USEPA. 1989a. Mouse oral subchronic study with acenaphthene. Study conducted by Hazelton Laboratories, Inc., for the Office of Solid Waste, Washington, DC.

USEPA. 1989b. Subchronic toxicity in mice with anthracene. Final Report. Hazelton Laboratories America, Inc. Prepared for the Office of Solid Waste, Washington, DC.

USEPA. 1989c. 13-Week Mouse Oral Subchronic Toxicity Study. Prepared by Toxicity Research Laboratories, Ltd., Muskegon, MI for the Office of Solid Waste, Washington, DC. (Fluorene).

USEPA, 1988. 13-Week Mouse Oral Subchronic Study. Prepared by Toxicity Research Labs, LTD. Muskegon, MI. for the Office of Solid Waste. Cited in IRIS, Accessed September 2002. (Fluoranthene)

USEPA. 1986. o, m, p-Cresol. 90-Day oral subchronic toxicity studies in rats. Office of Solid Waste, Washington, DC.

USEPA. 1987. o, m, p-Cresol. 90-Day oral subchronic neurotoxicity study in rats. Office of Solid Waste, Washington, DC.

USEPA (U.S. Environmental Protection Agency), 1995. Great Lakes Water Quality Initiative Criteria Documents for the Protection of Wildlife, DDT; Mercury; 2,3,7,8-TCDD; and PCBs. Office of Water, Washington, DC. EPA-820/B-95-008 March.

USFWS (United States Fish and Wildlife Service. 1969. Bureau of sport fisheries and wildlife. Publication 74, pp. 56-57.

## SOURCES AND ENPOINTS FOR NOAELS AND LOAELS FOR TERRESTRIAL WILDLIFE

Page 8 of 8

Van Velsen, F. L., L. H. J. C. Danse, F. X. R. Van Leeuwen, J. A. M. A. Dormans, and M. J. Van Logten. 1986. The subchronic oral toxicity of the beta-isomer of hexachlorocyclohexane in rats. *Fund. Appl. Toxicol.* 6: 697-712.

Verschuuren, H. G., R. Kroes, E. M. Den Tonkelaar, J. M. Berkvens, P. W. Helleman, A. G. Rauws, P. L. Schuller, and G. J. Van Esch. 1976. "Toxicity of Methyl Mercury Chloride in Rats. II. Reproduction Study." *Toxicol.* 6: 97-106.

Vos, J. G., H. L. Van Der Maas, A. Musch, and E. Ram. 1971. Toxicity of hexachlorobenzene in Japanese quail with special reference to porphyria, liver damage, reproduction, and tissue residues. *Toxicol. Appl. Pharmacol.* 18: 944-957.

White, D.H., and M.P. Dieter. 1978. "Effects of Dietary Vanadium in Mallard Ducks". *J. Toxicol. Environ. Health* 4:43-50.

White, D.H., and M.T. Finley. 1978. "Uptake and Retention of Dietary Cadmium in Mallard Ducks". *Environ. Res.* 17:53-59

World Health Organization (WHO). 1984. Chlordane. Environmental Health Criteria 34. World Health Organization, Geneva, Switzerland.

**NOTE: Some of the references in this list are not referenced in the proceeding table. This reference list also includes other toxicity values not used in the development of the terrestrial reference values.**

**BIOACCUMULATION FACTORS**  
**SITE 2B - AREA A WETLAND**  
**NSB-NLON, GROTON, CONNECTICUT**

This appendix presents the bioaccumulation factors (BAFs) and biota-sediment accumulation factors (BSAFs) that were used in the food chain models. The following sources of BAFs and BSAFs were used in the ecological risk assessment for most of the chemicals:

- Plant and Soil Invertebrate BAFs: EPA Guidance for Developing Ecological Soil Screening Levels, Attachment 4-1 (USEPA, 2005)
- Plant BAFs (organic chemicals): Toxicity and Chemical-Specific Factors Database (ORNL, 2006).
- Plant BAFs (metals): Empirical Model for the Uptake of Inorganic Chemicals from Soil by Plants (ORNL, September 1998).
- Soil Invertebrate BAFs: Development and Validation of Bioaccumulation Models for Earthworms (Sample et al., 1998).
- Sediment Invertebrate BSAFs: Biota Sediment Accumulation Factors for Invertebrates: Review and recommendations for the Oak Ridge Reservation. (ORNL, August 1998).
- Fish BSAFs: The Incidence and Severity of Sediment Contamination in Surface Waters of the United States, Volume 1: National Sediment Quality Survey (USEPA, 2004).

Attachment Table 3-3 presents the BAFs and BSAFs that were used in the surrogate species' food-chain models for the individual constituents that were detected in the Area A Wetland. Note that dry weight BAFs and BSAFs were used for this ERA.

The EPA Guidance for Developing Ecological Soil Screening Levels (Eco SSLs) was the source of the BAFs for most of the chemicals. The majority of the BAFs are actually regression equations that are used to calculate the tissue concentration from the soil concentration.

Attachment Table 3-4 presents the derivation of the soil to earthworm BAFs for PAHs. The BAFs for the PAHs in the Eco-SSL guidance document are based on equilibrium partitioning. The article on which the BAFs in the Eco-SSI document are based (Jager et al., 2003) indicates that

the equilibrium partitioning may overestimate BAFs by up to two orders of magnitude. Two sets of BAF studies were found for PAHs based on empirical data (see Appendix Table 3-2). In Ma et al., (1998), BAFs were calculated for PAHs in different soil types and the BAFs were presented as values for total PAHs. The second study, reported in Beyer (1990), presented average soil and earthworm concentrations for individual PAHs. The data were used to calculate the BAFs by dividing the PAH concentrations in the earthworm samples by the PAH concentrations in the soil samples. For the conservative food chain model, 1.606 [the maximum BAF from Ma et al., (1998)] was used while the average BAF of 0.609 was used for the average food chain model. These values were used because they are more conservative than those from Beyer (1990) and were based on more than one study. Although the selected BAFs are about one order of magnitude lower than those in the Eco-SSL document, the BAFs from Ma et al., were used for because they likely provide a more representative assessment of bioaccumulation because they are based on empirical data.

The average BAFs for pesticides were calculated from field studies summarized in various studies. The sources of the BAF are listed in the footnotes in Attachment Table 3-5. BAFs were calculated by dividing the worm concentration by the soil concentration (if the BAFs were not calculated within the study). The BAFs were either presented on a wet-weight or dry weight basis. Wet weight BAFs were derived by multiplying the dry weight BAF by 0.16, which is the percent solids of soil invertebrates (Sample et. al., 1997), while dry-weight BAFs were derived by dividing the wet weight BAF by 0.16. For this ERA, the dry-weight BAFs were used in the food chain model.

An average BAF was calculated for each pesticide, when data from more than one study were available. The average BAF was used for both the conservative and average food chain models.

The BSAFs above were used to calculate chemical concentrations in the tissues of sediment invertebrates from the chemical concentrations in the sediment. The BSAFs for invertebrates were used metals and PCBs. However, invertebrate BSAFs are not available for other organic chemicals so fish BSAFs were used for the organic chemicals except PCBs.

**References:**

Beyer, W.N. and D.C. Gish. 1980. Persistence in Earthworms and Potential Hazards to Birds of soil Applied DDT, Dieldrin, and Heptachlor. *J. Appl. Ecol.* 17:295-307. Cited in Beyer, 1990.

Beyer, Nelson. 1990. Evaluating Soil Contamination. U.S. Department of the Interior, Fish and Wildlife Service. Biological Report 90(2). July.

Davis, B.N.K. 1971. Laboratory Studies on the Uptake of Dieldrin and DDT by Earthworms. *Soil Biol. Biochem.* 3:221-233. Cited in Beyer, 1990.

Gish, C.D., 1970. Organochlorine Insecticide Residues in Soils and Soil Invertebrates from Agricultural Lands. *Pestic. Monit. J.* 3:241-252. Cited in Beyer, 1990.

Jager, T. 1998. Mechanistic Approach for Estimating Bioconcentration of Organic Chemicals in Earthworms. *Environ. Toxicol. Chem.* 17: 2080-2090.

Jeffries, D.J., and B.N.K. Davis. 1968. Dynamics of Dieldrin in Soil, Earthworms, and Song Thrushes. *J. Wildlife. Manage.* 32:441-456. Cited in Beyer, 1990.

Ma, Wei-Chun, Andre Van Kleunen, Jaap Immerzeel, and P. Gert-Jan de Maagd. 1998. Bioaccumulation of Polycyclic Aromatic Hydrocarbons by Earthworms: Assessment of Equilibrium Partitioning Theory in *In Situ* Studies and Water Experiments. *Environmental Toxicology and Chemistry*, Vol. 17, No. 9. pp. 1730-1737.

ORNL (Oak Ridge National Laboratory). 1998. Empirical Model for the Uptake of Inorganic Chemicals from Soil by Plants. BJC/OR-133. September.

ORNL (Oak Ridge National Laboratory). 1998. Biota Sediment Accumulation Factors for Invertebrates: Review and recommendations for the Oak Ridge Reservation. BJC/OR-112. August.

ORNL. 2006. Toxicity and Chemical-Specific Factors Database. Oak Ridge National Laboratory Web Page, [http://risk.lsd.ornl.gov/cgi-bin/tox/TOX\\_9801](http://risk.lsd.ornl.gov/cgi-bin/tox/TOX_9801).

Sample, B.E., J.J. Beauchamp, R.A. Efroymson, G.W., Suter II, and T.L. Ashwood. 1998. Development and Validation of Bioaccumulation Models for Earthworms. Oak Ridge National Laboratory. June. ES/ER/TM-220.

USEPA (U.S. Environmental Protection Agency), 2004. The Incidence and Severity of Sediment Contamination in Surface Waters of the United States, Volume 1: National Sediment Quality Survey: Second Edition. Office of Science and Technology. Washington, D.C. EPA 823-R-04-007. November.

USEPA, 2005. Guidance for Developing Ecological Soil Screening Level. Office of Solid Waste and Emergency and Response. OSWER Directive 92857-55. February.

Venter, J.M., and A.J. Reinecke. 1985. Dieldrin and Growth and Development of the Earthworm *Eisenia Fetida* (Oligochaeta). Bull. Environ, Contam. Toxicol. 35:652-659. Cited in Beyer, 1990.

Wheatley, G.A., and J.A. Hardman. 1968. Organochlorine Insecticide Residues in Earthworms from Arable Soils. J. Sci. Food. Agric. 19:219-225. Cited in Beyer, 1990.

## ATTACHMENT TABLE 3-3

DRY WEIGHT BAFS AND/OR BSAFS FOR PLANTS, EARTHWORMS, AND FISH  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 1 OF 2

Chemicals	Plant BAFs <sup>(1,2,3)</sup>		Earthworm BAFs <sup>(3,4,5)</sup>		Fish BSAFs <sup>(6)</sup>		Sediment Invertebrate BSAFs <sup>(7)</sup>	
	Conservative <sup>(8)</sup>	Average <sup>(8)</sup>	Conservative <sup>(8)</sup>	Average <sup>(8)</sup>	Conservative <sup>(8)</sup>	Average <sup>(8)</sup>	Conservative <sup>(8)</sup>	Average <sup>(8)</sup>
<b>Semivolatile Organics</b>								
1,4-Dichlorobenzene	4.10E-01	4.10E-01	1.00E+00	1.00E+00	1.00E+00	1.00E+00	1.00E+00	1.00E+00
2-Methylnaphthalene	???	???	1.00E+00	1.00E+00	1.00E+00	1.00E+00	1.00E+00	1.00E+00
Acenaphthene	Eco-SSL	Eco-SSL	6.69E-01	2.56E-01	2.90E-01	2.90E-01	1.00E+00	1.00E+00
Acenaphthylene	Eco-SSL	Eco-SSL	6.69E-01	2.56E-01	2.90E-01	2.90E-01	1.00E+00	1.00E+00
Anthracene	Eco-SSL	Eco-SSL	6.69E-01	2.56E-01	2.90E-01	2.90E-01	1.00E+00	1.00E+00
Benzo(a)anthracene	Eco-SSL	Eco-SSL	6.69E-01	2.56E-01	2.90E-01	2.90E-01	1.00E+00	1.00E+00
Benzo(a)pyrene	Eco-SSL	Eco-SSL	6.69E-01	2.56E-01	2.90E-01	2.90E-01	1.00E+00	1.00E+00
Benzo(b)fluoranthene	Eco-SSL	Eco-SSL	6.69E-01	2.56E-01	2.90E-01	2.90E-01	1.00E+00	1.00E+00
Benzo(g,h,i)perylene	Eco-SSL	Eco-SSL	6.69E-01	2.56E-01	2.90E-01	2.90E-01	1.00E+00	1.00E+00
Benzo(k)fluoranthene	Eco-SSL	Eco-SSL	6.69E-01	2.56E-01	2.90E-01	2.90E-01	1.00E+00	1.00E+00
Chrysene	Eco-SSL	Eco-SSL	6.69E-01	2.56E-01	2.90E-01	2.90E-01	1.00E+00	1.00E+00
Dibenzo(a,h)Anthracene	Eco-SSL	Eco-SSL	6.69E-01	2.56E-01	2.90E-01	2.90E-01	1.00E+00	1.00E+00
Dibenzofuran	1.50E-01	1.50E-01	6.69E-01	2.56E-01	1.00E+00	1.00E+00	1.00E+00	1.00E+00
Fluoranthene	Eco-SSL	Eco-SSL	6.69E-01	2.56E-01	2.90E-01	2.90E-01	1.00E+00	1.00E+00
Fluorene	1.10E-01	1.10E-01	6.69E-01	2.56E-01	2.90E-01	2.90E-01	1.00E+00	1.00E+00
Indeno(1,2,3-cd)pyrene	Eco-SSL	Eco-SSL	6.69E-01	2.56E-01	2.90E-01	2.90E-01	1.00E+00	1.00E+00
Naphthalene	4.60E-01	4.60E-01	6.69E-01	2.56E-01	2.90E-01	2.90E-01	1.00E+00	1.00E+00
Pentachlorophenol	1.40E-02	1.40E-02	1.00E+00	1.00E+00			1.00E+00	1.00E+00
Phenanthrene	Eco-SSL	Eco-SSL	6.69E-01	2.56E-01	2.90E-01	2.90E-01	1.00E+00	1.00E+00
Pyrene	Eco-SSL	Eco-SSL	6.69E-01	2.56E-01	2.90E-01	2.90E-01	1.00E+00	1.00E+00
<b>Pesticides/ PCBs</b>								
4,4'-DDD	Eco-SSL	Eco-SSL	Eco-SSL	Eco-SSL	2.80E-01	2.80E-01	1.00E+00	1.00E+00
4,4'-DDE	Eco-SSL	Eco-SSL	Eco-SSL	Eco-SSL	7.70E+00	7.70E+00	1.00E+00	1.00E+00
4,4'-DDT	Eco-SSL	Eco-SSL	Eco-SSL	Eco-SSL	1.67E+00	1.67E+00	1.00E+00	1.00E+00
Aldrin	6.90E-01	6.90E-01	3.30E+00	3.30E+00	1.80E+00	1.80E+00	1.00E+00	1.00E+00
Alpha-Chlordane	2.50E-02	2.50E-02	5.00E+00	5.00E+00	4.77E+00	4.77E+00	1.00E+00	1.00E+00
Aroclor-1260	2.90E-03	2.90E-03	1.59E+01	6.67E+00	1.85E+00	1.85E+00	6.41E+01	3.62E+01
Dieldrin	8.20E-02	8.20E-02	6.64E+00	6.64E+00	1.80E+00	1.80E+00	1.00E+00	1.00E+00
Gamma-Chlordane	2.50E-02	2.50E-02	5.00E+00	5.00E+00	2.22E+00	2.22E+00	1.00E+00	1.00E+00
Heptachlor	1.20E-01	1.20E-01	1.00E+01	1.00E+01	1.80E+00	1.80E+00	1.00E+00	1.00E+00
Endosulfan I	3.30E-01	3.30E-01	1.00E+00	1.00E+00	1.80E+00	1.80E+00	1.00E+00	1.00E+00
Endosulfan II	3.30E-01	3.30E-01	1.00E+00	1.00E+00	1.80E+00	1.80E+00	1.00E+00	1.00E+00
Endosulfan Sulfate	3.30E-01	3.30E-01	1.00E+00	1.00E+00	1.80E+00	1.80E+00	1.00E+00	1.00E+00
Endrin	8.20E-02	8.20E-02	3.60E+00	3.60E+00	1.80E+00	1.80E+00	1.00E+00	1.00E+00
Endrin Aldehyde	8.20E-02	8.20E-02	3.60E+00	3.60E+00	1.80E+00	1.80E+00	1.00E+00	1.00E+00
Endrin Ketone	8.20E-02	8.20E-02	3.60E+00	3.60E+00	1.80E+00	1.80E+00	1.00E+00	1.00E+00
Heptachlor Epoxide	2.80E-02	2.80E-02	3.00E+00	3.00E+00	1.80E+00	1.80E+00	1.00E+00	1.00E+00
Methoxychlor	1.10E-01	1.10E-01	1.00E+00	1.00E+00	1.80E+00	1.80E+00	1.00E+00	1.00E+00
beta-BHC	1.80E-01	1.80E-01	5.00E+00	5.00E+00	1.80E+00	1.80E+00	1.00E+00	1.00E+00
delta-BHC	9.00E-01	9.00E-01	5.00E+00	5.00E+00	1.80E+00	1.80E+00	1.00E+00	1.00E+00
gamma-BHC (Lindane)	2.70E-01	2.70E-01	5.00E+00	5.00E+00	1.80E+00	1.80E+00	1.00E+00	1.00E+00

ATTACHMENT TABLE 3-3

DRY WEIGHT BAFS AND/OR BSAFs FOR PLANTS, EARTHWORMS, AND FISH  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 2 OF 2

Chemicals	Plant BAFs <sup>(1,2,3)</sup>		Earthworm BAFs <sup>(3,4,5)</sup>		Fish BSAFs <sup>(6)</sup>		Sediment Invertebrate BSAFs <sup>(7)</sup>	
	Conservative <sup>(8)</sup>	Average <sup>(8)</sup>	Conservative <sup>(8)</sup>	Average <sup>(8)</sup>	Conservative <sup>(8)</sup>	Average <sup>(8)</sup>	Conservative <sup>(8)</sup>	Average <sup>(8)</sup>
<b>Inorganics</b>								
Arsenic	Eco-SSL	Eco-SSL	Eco-SSL	Eco-SSL	1.00E+00	1.00E+00	6.90E-01	1.43E-01
Cadmium	Eco-SSL	Eco-SSL	Eco-SSL	Eco-SSL	1.00E+00	1.00E+00	7.99E+00	6.00E-01
Chromium	Eco-SSL	Eco-SSL	Eco-SSL	Eco-SSL	1.00E+00	1.00E+00	4.68E-01	1.00E-01
Copper	Eco-SSL	Eco-SSL	Eco-SSL	Eco-SSL	1.00E+00	1.00E+00	5.25E+00	1.56E+00
Lead	Eco-SSL	Eco-SSL	Eco-SSL	Eco-SSL	1.00E+00	1.00E+00	6.07E-01	7.10E-02
Mercury	5.00E+00	6.52E-01	Regression	Regression	1.00E+00	1.00E+00	2.87E+00	1.14E+00
Nickel	Eco-SSL	Eco-SSL	Eco-SSL	Eco-SSL	1.00E+00	1.00E+00	2.32E+00	4.86E-01
Selenium	Eco-SSL	Eco-SSL	Eco-SSL	Eco-SSL	1.00E+00	1.00E+00	1.00E+00	1.00E+00
Silver	Eco-SSL	Eco-SSL	Eco-SSL	Eco-SSL	1.00E+00	1.00E+00	1.00E+00	1.00E+00
Vanadium	Eco-SSL	Eco-SSL	Eco-SSL	Eco-SSL	1.00E+00	1.00E+00	1.00E+00	1.00E+00
Zinc	Eco-SSL	Eco-SSL	Eco-SSL	Eco-SSL	1.00E+00	1.00E+00	7.53E+00	1.94E+00

**Notes:**

BAF - Bioaccumulation Factor

BSAF - Biota Sediment Accumulation Factor

1 - ORNL (2006) for organics; only one value is available for conservative and average exposures

2 - ORNL, (September, 1998) for inorganics; conservative value is 90th percentile, average value is median value

3 - Where "Eco-SSL" is given, values were calculated using equations from USEPA (2005), Attachment 4-1,

Tables 4a (for inorganics) and 4b (for organics).

4 - Sample et al., (June, 1998) for mercury and Aroclor-1260; conservative value is 90th percentile; average value is median value

5 - See Attachment Tables 3-4 and 3-5 for PAH and pesticide BAFs, respectively.

6 - USEPA, November 2004; only one value is available for conservative and average exposures. Values for organic chemicals are the same for wet-weight and dry weight.

These were used as surrogates for invertebrate BASFs for the organics, except Aroclor-1260.

7 - ORNL (August, 1998); conservative value is 90th percentile; average value is median value

8 - Conservative and average refers to the exposure scenarios for which the uptake factors are used

Default value of 1 is assigned to parameters without uptake factors

ATTACHMENT TABLE 3-4

SOIL TO EARTHWORM BIOACCUMULATION FACTORS FOR PAHS  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT

Soil	Wet-Weight BAFs <sup>(1)</sup>	Dry-Weight BAFs <sup>(2)</sup>	Soil Type
OT1	0.081	0.506	Silty clay loam
OT2	0.026	0.163	Light sandy loam
OT3	0.105	0.656	Silty clay loam
OT4	0.257	1.606	Silty clay loam
OT5	0.192	1.200	Silty clay loam
OT6	0.091	0.569	Silty clay loam
GP1	0.069	0.431	Silty clay loam
GP2	0.072	0.450	Silty clay loam
GP3	0.062	0.388	Silty clay loam
GP4	0.11	0.688	Silty clay
GP5	0.042	0.263	Silty clay
GP6	0.062	0.388	Silty clay
<b>Minimum BAF</b>	<b>0.026</b>	<b>0.163</b>	
<b>Maximum BAF</b>	<b>0.257</b>	<b>1.606</b>	
<b>Average BAF</b>	<b>0.097</b>	<b>0.609</b>	

Notes:

Source of data is Ma et al., (1998)

1 - BAFs from the study are based on wet weight and normalized to the percent of organic carbon and percent lipids.

2 - These BAFs were calculated by dividing the wet weight BAF by 0.16 (percent solids of an earthworm)

Chemical	Earthworm Bioaccumulation Factors <sup>(1)</sup>			
	Soil Concentration (mg/kg)	Earthworm Concentration (mg/kg)	BAF (dry weight)	BAF (wet weight) <sup>(2)</sup>
Acenaphthylene	ND	ND	ND	ND
Anthanthrene	1.2	0.11	0.092	0.015
Anthracene	0.92	0.047	0.051	0.008
Benzo(a)anthracene	2	0.25	0.13	0.020
Benzo(a)pyrene	3.8	1.3	0.34	0.055
Benzo(b)fluoranthene	2.6	0.83	0.32	0.051
Benzo(e)pyrene	2.1	0.91	0.43	0.069
Benzo(g,h,i)perylene	4.5	1.1	0.24	0.039
Benzo(k)fluoranthene	1.5	0.38	0.25	0.041
Chrysene	2	0.35	0.18	0.028
Dibenzo(a,i)pyrene	1.4	0.44	0.31	0.050
Dibenzo(a,j)anthracene	0.87	0.32	0.37	0.059
Fluoranthene	2.4	0.19	0.079	0.013
Fluorene	ND	ND	ND	ND
Indeno(1,2,3-cd)pyrene	3.1	1.3	0.42	0.067
Naphthalene	ND	ND	ND	ND
Perylene	1.2	0.3	0.25	0.040
Phenanthrene	2.3	0.28	0.12	0.019
Pyrene	2.5	0.23	0.092	0.015
Triphenylene	1.3	0.87	0.67	0.107
		<b>Minimum BAF</b>	<b>0.051</b>	<b>0.0082</b>
		<b>Maximum BAF</b>	<b>0.669</b>	<b>0.107</b>
		<b>Average BAF</b>	<b>0.256</b>	<b>0.041</b>

Notes:

ND = No data available

1 - Source of data is Table 25 in Beyer (1990)

2 - Wet weight BAF was calculated by multiplying the dry weight BAF by 0.16 (percent solids of an earthworm)

ATTACHMENT TABLE 3-5

SOIL TO EARTHWORM BIOACCUMULATION FACTORS FOR PESTICIDES  
SITE 2B - AREA A WETLAND  
NSB-NLON, GROTON, CONNECTICUT

Parameter	Study Values					Calculated Values		Reference	Comments
	Worm Concentration		Soil Conc. (dry weight)	Dry Weight BAF	Wet Weight BAF	Final Dry Weight <sup>(1)</sup> BAF	Final Wet Weight <sup>(2)</sup> BAF		
	Dry Weight	Wet Weight							
<i>Dieldrin</i>	NA	NA	NA	8	NA	8	1.28	1	soil type unknown (11-year field study)
	NA	NA	NA	2.4	NA	2.4	0.38	2	compost (lab) (17 ppm dieldrin in compost)
	NA	NA	NA	5.6	NA	5.6	0.90	2	compost (lab) (17 ppm dieldrin in compost)
	NA	18.4	25	NA	0.74	4.6	0.7	5	compost (20-day lab study)
	NA	24.4	25	NA	0.98	6.1	1.0	5	compost (20-day lab study)
	NA	4.6	10	NA	0.46	2.9	0.5	6	90-day lab study
	NA	9.7	30	NA	0.32	2.0	0.3	6	90-day lab study
	NA	12.4	50	NA	0.25	1.6	0.2	6	90-day lab study
	NA	13.9	100	NA	0.14	0.87	0.1	6	90-day lab study
<i>Average dry/wet weight BAF from field studies<sup>(3)</sup></i>				NA	NA	6.64	1.06		
<i>gamma-BHC</i>	NA	NA	NA	5	1.5-4.2	5	1.5-4.2	4	agricultural soil (0.004 ppm gamma-BHC in soil)
<i>Average dry/wet weight BAF from field studies<sup>(3)</sup></i>				NA	NA	5	2.85		
<i>Heptachlor</i>	NA	NA	NA	10	NA	10	1.60	4	soil type unknown (11-year field study)
<i>Average dry/wet weight BAF from field studies<sup>(3)</sup></i>				NA	NA	10	1.6		
<i>Aldrin</i>	NA	NA	NA	3.3	NA	3.3	0.528	3	from data collected in 7 agricultural fields
<i>Chlordane</i>	NA	NA	NA	5	NA	5.0	0.8	3	from data collected in 7 agricultural fields
<i>Endrin</i>	NA	NA	NA	3.6	NA	3.6	0.576	3	from data collected in 26 agricultural fields
<i>Heptachlor epoxide</i>	NA	NA	NA	3	NA	3.0	0.48	3	from data collected in 9 agricultural fields

Notes:

BAF - bioaccumulation factor = worm concentration/soil concentration

NA - Not applicable

The percent solids of earthworms is assumed to be 0.16 [Sample et al., 1997]

1 - The calculated dry weight BAF was either obtained directly from the study or was calculated by dividing the wet weight BAF by 0.16

2 - The calculated wet weight BAF was either obtained directly from the study or was calculated by multiplying the dry weight BAF by 0.16

Reference

1 - Beyer and Gish, 1980 and Beyer and Kryniitsky, 1989

2 - Davis, 1971

3 - Gish, 1970

4 - Wheatly and Hardman, 1968

5 - Jeffries and Davis, 1968

6 - Venter and Reinecke, 1985

**APPENDIX C – ATTACHMENT 4**  
**HISTORICAL ANALYTICAL DATA TABLES**

SURFACE SOIL SAMPLES  
PHASE I RI  
SITE 2B - AREA A WETLAND  
NSB-NLON, GROTON, CONNECTICUT  
PAGE 1 OF 6

SITE	2B	2B	2B	2B	2B
AOC	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS
PHASE	PH1	PH1	PH1	PH1	PH1
LOCATION	2WMW2S	2WMW39DS	2WMW45DS	2WMW5S	2WMW5S
NSAMPLE	082390-2WMW2(0-2)	2W-SU-39DS-00-99	2W-SU-45DS-00-99	090490-2WMW5(0-2)	090490-2WMW5(0-2)-D
SAMPLE	2WMW2(0-2)	2W-SU-39DS-00-99	2W-SU-45DS-00-99	2WMW5(0-2)	2WTB11(0-2)
MATRIX	SO	SO	SO	SO	SO
TOP DEPTH	0	0	0	0	0
BOTTOM DEPTH	2	2	2	2	2
SAMPLE DATE	08/23/90	05/18/99	05/16/99	09/04/90	09/04/90
Volatile Organics (ug/kg)					
2-BUTANONE	11 U	13 U	23 U	17 UJ	17 U
ACETONE	11 U	25	160	17 UJ	17 U
CARBON DISULFIDE	5 U	13 U	23 U	8 UJ	8 U
METHYLENE CHLORIDE	5 U	3	6	8 UJ	8 U
TETRACHLOROETHENE	5 U	13 U	23 U	3 UJ	8 U
TRICHLOROETHENE	5 U	13 U	23 U	8 UJ	8 U
Semivolatile Organics (ug/kg)					
ACENAPHTHENE	350 U	270	61 U	550 U	560 U
ACENAPHTHYLENE	350 U	88 U	120 U	550 U	59 J
ANTHRACENE	350 U	6.5 U	9.3 U	550 U	560 U
BENZO(A)ANTHRACENE	83 J	260	30	160 J	140 J
BENZO(A)PYRENE	81 J	310	52	550 U	560 U
BENZO(B)FLUORANTHENE	74 J	420	120	180 J	260 J
BENZO(G,H,I)PERYLENE	350 U	260	49	550 U	560 U
BENZO(K)FLUORANTHENE	71 J	180	9.3 U	120 J	180 J
BENZOIC ACID	1700 U			2700 U	2700 U
BIS(2-ETHYLHEXYL)PHTHALATE	350 U	51.4 J	290 U	790	590
CHRYSENE	110 J	290	9.3 U	230 J	330 J
DIBENZO(A,H)ANTHRACENE	350 U	32	15 U	550 U	560 U
FLUORANTHENE	160 J	890	83	180 J	240 J
INDENO(1,2,3-CD)PYRENE	350 U	140	9.3 U	550 U	560 U
PHENANTHRENE	120 J	300	9.3 U	130 J	160 J
PYRENE	150 J	810	120	320 J	330 J
TOTAL PAH	849	4162	454	1320	1699
Pesticides/PCBs (ug/kg)					
4,4'-DDD	17 U	40	40	27 U	27 U
4,4'-DDE	17 U	5.2	10	27 U	27 U
4,4'-DDT	17 U	28	5.8 U	27 U	27 U
ALPHA-CHLORDANE	85 U	3	2.9 U	130 U	140 U
AROCLOR-1260	170 U	110	58 U	270 U	270 U
TOTAL AROCLOR	0 U	110	0 U	0 U	0 U
GAMMA-CHLORDANE	85 U	2.2	2.9 U	130 U	140 U
TOTAL DDT	0	73.2	50	0	0

TABLE 1  
SURFACE SOIL SAMPLES  
PHASE I RI  
SITE 2B - AREA A WETLAND  
NSB-NLON, GROTON, CONNECTICUT  
PAGE 2 OF 6

SITE	2B	2B	2B	2B	2B
AOC	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS
PHASE	PH1	PH1	PH1	PH1	PH1
LOCATION	2WMW2S	2WMW39DS	2WMW45DS	2WMW5S	2WMW5S
NSAMPLE	082390-2WMW2(0-2)	2W-SU-39DS-00-99	2W-SU-45DS-00-99	090490-2WMW5(0-2)	090490-2WMW5(0-2)-D
SAMPLE	2WMW2(0-2)	2W-SU-39DS-00-99	2W-SU-45DS-00-99	2WMW5(0-2)	2WTB11(0-2)
MATRIX	SO	SO	SO	SO	SO
TOP DEPTH	0	0	0	0	0
BOTTOM DEPTH	2	2	2	2	2
SAMPLE DATE	08/23/90	05/18/99	05/16/99	09/04/90	09/04/90
<b>Inorganics (mg/kg)</b>					
ALUMINUM	13000	4860	17400	11200 J	11500 J
ARSENIC	2.5	1.2 B	15.1	7 J	7.6 J
BARIUM	53.3	28.3 B	48.9 B	45.7	41.3
BERYLLIUM	0.55	0.28 B	0.89 B	1.2 J	1.3 J
CADMIUM	6.9	0.16 B	0.55 B	4.7	4.9
CALCIUM	1170 J	1360	2070	1230 J	1310 J
CHROMIUM	20.5 J	12.2	43.2	57.1	65.2
COBALT	8.5	4.8 B	11.1 B	7.3	12.3
COPPER	26.1 J	21.7	16.9	38.5	35.2
IRON	14200	8100	30400	21500	23100
LEAD	16.3 J	22.4	11.8	38.5	26.8
MAGNESIUM	4170	2170	6480	5520	5590
MANGANESE	195 J	99.2	256	160 J	357 J
MERCURY	0.11 U	0.06 U	0.09 U	0.22	0.25
NICKEL	25.3 J	16.9	22.9	14.7	15.9
POTASSIUM	2130 J	1000 B	4160	3180	2420
SELENIUM	0.44 U	1.1 U	2.4	0.7	0.77
SILVER	1.8 U	0.2 U	0.28 U	2.5 U	2.5 U
SODIUM	118 J	224 B	954 B	957 J	930 J
VANADIUM	33.2	35.9	44.1	48.9 J	51.8 J
ZINC	125 J	49.7	58.9	54.6 J	61.3 J

SURFACE SOIL SAMPLES  
PHASE I RI  
SITE 2B - AREA A WETLAND  
NSB-NLON, GROTON, CONNECTICUT  
PAGE 3 OF 6

SITE	2B	2B	2B	2B	2B
AOC	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS
PHASE	PH1	PH1	PH1	PH1	PH1
LOCATION	2WMW5S	2WTB2	2WTB2	2WTB2	2WTB4
NSAMPLE	090490-2WMW5(0-2)-MAX	090690-2WTB2(0-2)	090690-2WTB2(0-2)-D	090690-2WTB2(0-2)-MAX	090690-2WTB4(0-2)
SAMPLE	2WMW5(0-2)MAX	2WTB2(0-2)	090690-2WTB9(4-6)	2WTB2(0-2)MAX	2WTB4(0-2)
MATRIX	SO	SO	SO	SO	SO
TOP DEPTH	0	0	0	0	0
BOTTOM DEPTH	2	2	2	2	2-
SAMPLE DATE	09/04/90	09/06/90	09/06/90	09/06/90	09/06/90
Volatile Organics (ug/kg)					
2-BUTANONE	17 U	20 U	18 U	20 U	16 U
ACETONE	17 U	20 U	18 U	20 U	16 U
CARBON DISULFIDE	8 U	10 U	9 U	10 U	8 U
METHYLENE CHLORIDE	8 U	10 U	9 U	10 U	8 U
TETRACHLOROETHENE	8 U	10 U	9 U	10 U	8 U
TRICHLOROETHENE	8 U	10 U	9 U	10 U	4 J
Semivolatile Organics (ug/kg)					
ACENAPHTHENE	560 U	2600 U	590 U	2600 U	530 U
ACENAPHTHYLENE	59 J	2600 U	590 U	2600 U	77 J
ANTHRACENE	560 U	2600 U	590 U	2600 U	52 J
BENZO(A)ANTHRACENE	160 J	400 J	590 U	400 J	220 J
BENZO(A)PYRENE	560 U	2600 U	590 U	2600 U	230 J
BENZO(B)FLUORANTHENE	260 J	2600 U	590 U	2600 U	220 J
BENZO(G,H,I)PERYLENE	560 U	2600 U	590 U	2600 U	530 U
BENZO(K)FLUORANTHENE	180 J	2600 U	590 U	2600 U	310 J
BENZOIC ACID	2700 U	13000 U	2900 U	13000 U	160 J
BIS(2-ETHYLHEXYL)PHTHALATE	790	2600 U	590 U	2600 U	280 J
CHRYSENE	330 J	2600 U	590 U	2600 U	320 J
DIBENZO(A,H)ANTHRACENE	560 U	2600 U	590 U	2600 U	530 U
FLUORANTHENE	240 J	460 J	590 U	460 J	310 J
INDENO(1,2,3-CD)PYRENE	560 U	2600 U	590 U	2600 U	530 U
PHENANTHRENE	160 J	2600 U	590 U	2600 U	270 J
PYRENE	330 J	380 J	590 U	380 J	460 J
TOTAL PAH	1719	1240	0	1240	2469
Pesticides/PCBs (ug/kg)					
4,4'-DDD	27 U	31 U	29 U	31 U	26 UJ
4,4'-DDE	27 U	31 U	29 U	31 U	32 UJ
4,4'-DDT	27 U	31 U	29 U	31 U	26 UJ
ALPHA-CHLORDANE	140 U	160 U	140 U	160 U	130 UJ
AROCLOR-1260	270 U	370 J	290 UJ	370 J	260 UJ
TOTAL AROCLOR	0 U	370	0 U	370	0 U
GAMMA-CHLORDANE	140 U	160 U	140 U	160 U	130 UJ
TOTAL DDT	0	0	0	0	0

TABLE 1

SURFACE SOIL SAMPLES  
 PHASE I RI  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 4 OF 6

SITE	2B	2B	2B	2B	2B
AOC	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS
PHASE	PH1	PH1	PH1	PH1	PH1
LOCATION	2WMW5S	2WTB2	2WTB2	2WTB2	2WTB4
NSAMPLE	090490-2WMW5(0-2)-MAX	090690-2WTB2(0-2)	090690-2WTB2(0-2)-D	090690-2WTB2(0-2)-MAX	090690-2WTB4(0-2)
SAMPLE	2WMW5(0-2)MAX	2WTB2(0-2)	090690-2WTB9(4-6)	2WTB2(0-2)MAX	2WTB4(0-2)
MATRIX	SO	SO	SO	SO	SO
TOP DEPTH	0	0	0	0	0
BOTTOM DEPTH	2	2	2	2	2
SAMPLE DATE	09/04/90	09/06/90	09/06/90	09/06/90	09/06/90
Inorganics (mg/kg)					
ALUMINIUM	11500 J	14100 J	14500 J	14500 J	15300 J
ARSENIC	7.6 J	6.7 J	8.1 J	8.1 J	8.7
BARIUM	45.7	52.1	47.7	52.1	61.3
BERYLLIUM	1.3 J	1.8 J	1.7 J	1.8 J	0.3 U
CADMIUM	4.9	6.9	7.2	7.2	0.29 U
CALCIUM	1310 J	3300 J	3690 J	3690 J	1250
CHROMIUM	65.2	41.2	39.9	41.2	89
COBALT	12.3	11.1	10.8	11.1	8.5
COPPER	38.5	20.2	17.1	20.2	47.6 J
IRON	23100	27200	27600	27600	30800
LEAD	38.5	11.2 J	11.2	11.2	128 J
MAGNESIUM	5590	7230	7630	7630	6130
MANGANESE	357 J	306	376	376	188 J
MERCURY	0.25	0.18 U	0.17 U	0.18 U	0.69 J
NICKEL	15.9	25.9	26.8	26.8	16.7
POTASSIUM	3180	3530	3800	3800	4320
SELENIUM	0.77	0.71 U	0.7 U	0.71 U	1.2
SILVER	2.5 U	2.8 U	3.5	3.5	2.4 U
SODIUM	957 J	2040 J	1980 J	2040 J	232 J
VANADIUM	51.8 J	57.9 J	56.7 J	57.9 J	50.5 J
ZINC	61.3 J	75.5 J	66.4 J	75.5 J	60.8 J

SURFACE SOIL SAMPLES  
PHASE I RI  
SITE 2B - AREA A WETLAND  
NSB-NLON, GROTON, CONNECTICUT  
PAGE 5 OF 6

SITE	2B	2B	2B	2B	2B
AOC	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS
PHASE	PH1	PH1	PH1	PH1	PH1
LOCATION	2WTB6	2WTB7	2WTB7	2WTB7	2WTB8
NSAMPLE	090690-2WTB6(0-2)	090590-2WTB7(0-2)	090590-2WTB7(0-2)-D	090590-2WTB7(0-2)-MAX	083090-2WTB8(1-3)
SAMPLE	2WTB6(0-2)	2WTB7(0-2)	2WTB7(25-27)	2WTB7(0-2)MAX	2WTB8(1-3)
MATRIX	SO	SO	SO	SO	SO
TOP DEPTH	0	0	0	0	1
BOTTOM DEPTH	2	2	2	2	3
SAMPLE DATE	09/06/90	09/05/90	09/05/90	09/05/90	08/30/90
<b>Volatile Organics (ug/kg)</b>					
2-BUTANONE	87	19 U	5 J	5 J	310
ACETONE	540	19 U	37	37	850
CARBON DISULFIDE	6 J	9 U	8 U	9 U	8 J
METHYLENE CHLORIDE	11 U	9 U	8 U	9 U	4 J
TETRACHLOROETHENE	11 U	9 U	8 U	9 U	7 J
TRICHLOROETHENE	11 U	9 U	8 U	9 U	10 U
<b>Semivolatile Organics (ug/kg)</b>					
ACENAPHTHENE	720 U	620 U	530 U	620 U	670 U
ACENAPHTHYLENE	720 U	620 U	530 U	620 U	120 J
ANTHRACENE	720 U	620 U	530 U	620 U	79 J
BENZO(A)ANTHRACENE	200 J	620 U	530 U	620 U	370 J
BENZO(A)PYRENE	190 J	620 U	530 U	620 U	390 J
BENZO(B)FLUORANTHENE	210 J	620 U	530 U	620 U	550 J
BENZO(G,H,I)PERYLENE	720 U	620 U	530 U	620 U	670 U
BENZO(K)FLUORANTHENE	250 J	620 U	530 U	620 U	390 J
BENZOIC ACID	130 J	3000 U	220 J	220 J	3300 U
BIS(2-ETHYLHEXYL)PHTHALATE	350 J	620 U	220 J	220 J	1300
CHRYSENE	250 J	620 U	530 U	620 U	600 J
DIBENZO(A,H)ANTHRACENE	720 U	620 U	530 U	620 U	670 U
FLUORANTHENE	300 J	72 J	53 J	72 J	600 J
INDENO(1,2,3-CD)PYRENE	720 U	620 U	530 U	620 U	270 J
PHENANTHRENE	160 J	620 U	530 U	620 U	340 J
PYRENE	270 J	98 J	530 U	98 J	570 J
TOTAL PAH	1830	170	53	170	4279
<b>Pesticides/PCBs (ug/kg)</b>					
4,4'-DDD	69 J	30 U	26 U	30 U	33 U
4,4'-DDE	35 UJ	30 U	26 U	30 U	33 U
4,4'-DDT	35 UR	30 U	26 U	30 U	33 U
ALPHA-CHLORDANE	170 UJ	150 U	130 U	150 U	160 U
AROCLOR-1260	350 UJ	300 U	260 U	300 U	330 U
TOTAL AROCLOR	0 U	0 U	0 U	0 U	0 U
GAMMA-CHLORDANE	170 UJ	150 U	130 U	150 U	160 U
TOTAL DDT	69	0	0	0	0

TABLE 1

SURFACE SOIL SAMPLES  
PHASE I RI  
SITE 2B - AREA A WETLAND  
NSB-NLON, GROTON, CONNECTICUT  
PAGE 6 OF 6

SITE	2B	2B	2B	2B	2B
AOC	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS
PHASE	PH1	PH1	PH1	PH1	PH1
LOCATION	2WTB6	2WTB7	2WTB7	2WTB7	2WTB8
NSAMPLE	090690-2WTB6(0-2)	090590-2WTB7(0-2)	090590-2WTB7(0-2)-D	090590-2WTB7(0-2)-MAX	083090-2WTB8(1-3)
SAMPLE	2WTB6(0-2)	2WTB7(0-2)	2WTB7(25-27)	2WTB7(0-2)MAX	2WTB8(1-3)
MATRIX	SO	SO	SO	SO	SO
TOP DEPTH	0	0	0	0	1
BOTTOM DEPTH	2	2	2	2	3
SAMPLE DATE	09/06/90	09/05/90	09/05/90	09/05/90	08/30/90
Inorganics (mg/kg)					
ALUMINUM	16800 J	13900 J	15900 J	15900 J	17900 J
ARSENIC	8.4	7.2 J	6.9 J	7.2 J	6.8 J
BARIUM	60.7	49.3	45.1	49.3	93.8
BERYLLIUM	0.73 J	1.5 J	1.8 J	1.8 J	1.8 J
CADMIUM	1.9 J	4.5	6.7	6.7	6.7 J
CALCIUM	1660	1590 J	1530 J	1590 J	2360
CHROMIUM	101	72.7	41	72.7	102
COBALT	9.4	7.6	12.7	12.7	9.2
COPPER	64.1 J	35.4	39.1	39.1	55
IRON	27100	20200	23100	23100	27900
LEAD	44 J	49.5 J	12.7 J	49.5 J	83.7
MAGNESIUM	6540	6580	6950	6950	7840
MANGANESE	202 J	189	265	265	233
MERCURY	0.23 U	0.38	0.21	0.38	0.67 J
NICKEL	20.2	16.8	26.2	26.2	26.9
POTASSIUM	3680	2680	3520	3520	4040 J
SELENIUM	1.5	0.79 U	0.78 U	0.79 U	0.93 J
SILVER	3.2 U	3 U	3 U	3 U	4.5
SODIUM	1570 J	1310 J	1430 J	1430 J	3150
VANADIUM	52 J	56.9 J	60.6 J	60.6 J	75 J
ZINC	74.9 J	59.5 J	79.8 J	79.8 J	62.9 J

2  
 SEDIMENT SAMPLES  
 PHASE I AND II RI AND FFS  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 1 OF 16

SITE	2B	2B	2B	2B	2B	2B	2B	2B	2B
AOC	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS
PHASE	Phase I	Phase I	Phase I	Phase II					
LOCATION	2WSD1	2WSD1	2WSD1	2WSD10	2WSD11	2WSD12	2WSD13	2WSD14	2WSD14
NSAMPLE	112690-2WSD1(0-0.5)	112690-2WSD1(0-0.5)-D	112690-2WSD1(0-0.5)-MAX	2WSD10(FIELD)	2WSD11(FIELD)	2WSD12(FIELD)	2WSD13(FIELD)	2WSD14	2WSD14(FIELD)
SAMPLE	112690-2WSD1(0-0.5)	112690-2WSD10(0-0.5)	112690-2WSD1(0-0.5)MAX	2WSD10(FIELD)	2WSD11(FIELD)	2WSD12(FIELD)	2WSD13(FIELD)	2WSD14	2WSD14(FIELD)
MATRIX	SD	SD	SD	SD	SD	SD	SD	SD	SD
TOP DEPTH	0	0	0	0	0	0	0	0	0
BOTTOM DEPTH	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
SAMPLE DATE	---	---	---	---	---	---	---	---	---
Volatile Organics (ug/kg)									
2-BUTANONE	140 J	42 J	140 J						
ACETONE	210	300	300						
CARBON DISULFIDE	11	17	17						
CHLOROBENZENE	11 U	11 U	11 U						
METHYLENE CHLORIDE	11 U	11 U	11 U						
TETRACHLOROETHENE	11 U	11 U	11 U						
TOLUENE	11 U	11 U	11 U						
TOTAL XYLENES	11 U	11 U	11 U						
TRICHLOROETHENE	11 U	11 U	11 U						
Semivolatile Organics (ug/kg)									
1,4-DICHLOROBENZENE	3500 U	3700 U	3700 U						
2,4-DIMETHYLPHENOL	3500 U	3700 U	3700 U						
2-METHYLNAPHTHALENE	3500 U	3700 U	3700 U						
4-METHYLPHENOL	3500 U	3700 U	3700 U						
ACENAPHTHENE	3500 U	3700 U	3700 U						
ACENAPHTHYLENE	3500 U	3700 U	3700 U						
ANTHRACENE	3500 U	3700 U	3700 U						
BENZO(A)ANTHRACENE	670 J	3700 U	670 J						
BENZO(A)PYRENE	3500 U	3700 U	3700 U						
BENZO(B)FLUORANTHENE	380 J	3700 U	380 J						
BENZO(G,H,I)PERYLENE	3500 U	3700 U	3700 U						
BENZO(K)FLUORANTHENE	480 J	3700 U	480 J						
BENZOIC ACID	17000 U	18000 U	18000 U						
BIS(2 ETHYLHEXYL)PHTHALATE	3500 U	3700 U	3700 U						
BUTYL BENZYL PHTHALATE	3500 U	3700 U	3700 U						
CARBAZOLE									
CHRYSENE	630 J	3700 U	630 J						
DI-N-BUTYL PHTHALATE	3500 U	3700 U	3700 U						
DIBENZO(A,H)ANTHRACENE	3500 U	3700 U	3700 U						
DIBENZOFURAN	3500 U	3700 U	3700 U						
FLUORANTHENE	970 J	3700 U	970 J						
FLUORENE	3500 U	3700 U	3700 U						
INDENO(1,2,3-CD)PYRENE	3500 U	3700 U	3700 U						
NAPHTHALENE	3500 U	3700 U	3700 U						
PENTACHLOROPHENOL	17000 U	18000 U	18000 U						
PHENANTHRENE	3500 U	3700 U	3700 U						
PYRENE	1300 J	3700 U	1300 J						
TOTAL PAH	4430	0	4430						



SEDIMENT SAMPLES  
 PHASE I AND II RI AND FFS  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 3 OF 16

SITE	2B	2B	2B	2B	2B	2B	2B	2B	2B
AOC	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS					
PHASE	Phase II	Phase I	Phase II	Phase II	Phase II				
LOCATION	2WSD15	2WSD16	2WSD17-	2WSD18	2WSD19	2WSD2	2WSD20	2WSD21	2WSD22
NSAMPLE	2WSD15(FIELD)	2WSD16(FIELD)	2WSD17(FIELD)	2WSD18(FIELD)	2WSD19(FIELD)	112690-2WSD2(0-0.5)	2WSD20(FIELD)	2WSD21(FIELD)	2WSD22(FIELD)
SAMPLE	2WSD15(FIELD)	2WSD16(FIELD)	2WSD17(FIELD)	2WSD18(FIELD)	2WSD19(FIELD)	112690-2WSD2(0-0.5)	2WSD20(FIELD)	2WSD21(FIELD)	2WSD22(FIELD)
MATRIX	SD	SD	SD	SD	SD	SD	SD	SD	SD
TOP DEPTH	0	0	0	0	0	0	0	0	0
BOTTOM DEPTH	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
SAMPLE DATE	---	---	---	---	---	---	---	---	---
Volatile Organics (ug/kg)									
2-BUTANONE						100			
ACETONE						130			
CARBON DISULFIDE						16			
CHLOROBENZENE						10 U			
METHYLENE CHLORIDE						10 U			
TETRACHLOROETHENE						10 U			
TOLUENE						10 U			
TOTAL XYLENES						10 U			
TRICHLOROETHENE						10 U			
Semivolatile Organics (ug/kg)									
1,4-DICHLOROBENZENE						3400 U			
2,4-DIMETHYLPHENOL						3400 U			
2-METHYLNAPHTHALENE						3400 U			
4-METHYLPHENOL						3400 U			
ACENAPHTHENE						3400 U			
ACENAPHTHYLENE						3400 U			
ANTHRACENE						3400 U			
BENZO(A)ANTHRACENE						3400 U			
BENZO(A)PYRENE						3400 U			
BENZO(B)FLUORANTHENE						3400 U			
BENZO(G,H,I)PERYLENE						3400 U			
BENZO(K)FLUORANTHENE						3400 U			
BENZOIC ACID						16000 U			
BIS(2-ETHYLHEXYL)PHTHALATE						3400 U			
BUTYL BENZYL PHTHALATE						3400 U			
CARBAZOLE									
CHRYSENE						3400 U			
DI-N-BUTYL PHTHALATE						6100 U			
DIBENZO(A,H)ANTHRACENE						3400 U			
DIBENZOFURAN						3400 U			
FLUORANTHENE						3400 U			
FLUORENE						3400 U			
INDENO(1,2,3-CD)PYRENE						3400 U			
NAPHTHALENE						3400 U			
PENTACHLOROPHENOL						16000 U			
PHENANTHRENE						3400 U			
PYRENE						3400 U			
TOTAL PAH						0			





TABLE 2

SEDIMENT SAMPLES  
 PHASE I AND II RI AND FFS  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 6 OF 16

SITE	2B	2B	2B	2B	2B	2B	2B	2B	2B
AOC	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS
PHASE	FFS	FFS	FFS	FFS	Phase II	Phase II	Phase II	Phase I	Phase II
LOCATION	2WSD23	2WSD24	2WSD25	2WSD26	2WSD27	2WSD28	2WSD29	2WSD30	2WSD30
NSAMPLE	2WSD23 (0.0-1.0)	2WSD24 (0.0-1.0)	2WSD25 (0.0-1.0)	2WSD26 (0.0-1.0)	2WSD27(FIELD)	2WSD28(FIELD)	2WSD29(FIELD)	112690-2WSD30(0-0.5)	2WSD30(FIELD)
SAMPLE	2WSD23 (0.0-1.0)	2WSD24 (0.0-1.0)	2WSD25 (0.0-1.0)	2WSD26 (0.0-1.0)	2WSD27(FIELD)	2WSD28(FIELD)	2WSD29(FIELD)	112690-2WSD30(0-0.5)	2WSD30(FIELD)
MATRIX	SD	SD	SD	SD	SD	SD	SD	SD	SD
TOP DEPTH	0	0	0	0	0	0	0	0	0
BOTTOM DEPTH	1	1	1	1	0.5	0.5	0.5	0.5	0.5
SAMPLE DATE	---	---	---	---	---	---	---	---	---
<b>Pesticides/PCBs (ug/kg)</b>									
4,4'-DDD	410 J	3100 J	4800 J	23 U	26 61 U	25 93 U	24 87 U	35 UJ	34 62 U
4,4'-DDE	93	240 J	720 J	6 2 J	26 61 U	25 93 U	24 87 U	35 UJ	34 62 U
4,4'-DDT	190	47 U	2900	6 9 UJ	26 61 U	25 93 U	24 87 U	57 J	34 62 U
ALDRIN	7 6 U	24 UJ	25 UJ	3 6 UJ				17 UJ	
ALPHA-CHLORDANE	9 5	24 UJ	25 UJ	3 6 UJ				170 UJ	
AROCLOR-1260	150 U	470 U	480 U	6 9 U				350 UJ	
TOTAL AROCLOR	0 U	0 U	0 U	0 U				0 U	
BETA-BHC	7 6 U	24 UJ	25 UJ	3 6 UJ				17 UJ	
DELTA-BHC	7 6 U	24 UJ	25 UJ	3 6 UJ				17 UJ	
DIELDRIN	15 U	47 UJ	48 UJ	6 9 UJ	26 61 U	25 93 U	24 87 U	35 UJ	34 62 U
ENDOSULFAN I	7 6 U	24 UJ	25 UJ	3 6 UJ				17 UJ	
ENDOSULFAN II	15 U	47 U	48 U	6 9 U				35 UJ	
ENDOSULFAN SULFATE	15 U	47 U	48 U	6 9 U				35 UJ	
ENDRIN	15 U	47 U	48 U	6 9 U				35 UJ	
ENDRIN ALDEHYDE	15 U	47 U	48 U	6 9 U					
ENDRIN KETONE	15 U	47 U	48 U	6 9 U				35 UJ	
GAMMA-BHC (LINDANE)	7 6 U	24 UJ	25 UJ	3 6 UJ				17 UJ	
GAMMA-CHLORDANE	6 J	14 J	25 UJ	3 6 UJ				170 UJ	
HEPTACHLOR	7 6 U	24 UJ	25 UJ	3 6 UJ				17 UJ	
HEPTACHLOR EPOXIDE	7 6 U	24 UJ	25 UJ	3 6 UJ				17 UJ	
METHOXYCHLOR	7 6 U	240 U	250 U	3 6 U				170 UJ	
TOTAL DDT	693	3340	8420	6 2				57	
<b>Inorganics (mg/kg)</b>									
ALUMINIUM								18800	
ANTIMONY								9 8 UR	
ARSENIC								9 5 J	
BARIUM								56 6	
BERYLLIUM								0 88	
BORON								4400 R	
CADMIUM								5 3	
CALCIUM								4220	
CHROMIUM								92 6	
COBALT								11 1	
COPPER								69 5 J	
CYANIDE								2 4 U	
IRON								29700	
LEAD								46 1	
MAGNESIUM								7990	
MANGANESE								341	
MERCURY								0 31 J	
NICKEL								27	
POTASSIUM								4280	
SELENIUM								1 3	
SILVER								2 7 UJ	
SODIUM								6090	
VANADIUM								45 5	
ZINC								127	
<b>Miscellaneous Parameters (mg/kg)</b>									
TOTAL ORGANIC CARBON	65000	91000	28000	26000					





SEDIMENT SAMPLES  
 PHASE I AND II RI AND FFS  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 9 OF 16

SITE	2B AREA A WETLANDS								
PHASE	Phase II	Phase II	Phase I	Phase II	Phase I				
LOCATION	2WSD39	2WSD39	2WSD4	2WSD40	2WSD40	2WSD41	2WSD41	2WSD42	2WSD5
NSAMPLE	2WSD39	2WSD39(FIELD)	112690-2WSD4(0-0.5)	2WSD40	2WSD40(FIELD)	2WSD41	2WSD41(FIELD)	2WSD42(FIELD)	112690-2WSD5(0-0.5)
SAMPLE	2WSD39	2WSD39(FIELD)	112690-2WSD4(0-0.5)	2WSD40	2WSD40(FIELD)	2WSD41	2WSD41(FIELD)	2WSD42(FIELD)	112690-2WSD5(0-0.5)
MATRIX	SD								
TOP DEPTH	0	0	0	0	0	0	0	0	0
BOTTOM DEPTH	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
SAMPLE DATE	---	---	---	---	---	---	---	---	---
Volatile Organics (ug/kg)									
2-BUTANONE			20 U						11 U
ACETONE			20 U						11 U
CARBON DISULFIDE			10 U						5 U
CHLOROBENZENE			10 U						5 U
METHYLENE CHLORIDE			10 J						3 J
TETRACHLOROETHENE			16 J						4 J
TOLUENE			10 U						5 U
TOTAL XYLENES			10 U						5 U
TRICHLOROETHENE			11 J						3 J
Semivolatile Organics (ug/kg)									
1,4-DICHLOROBENZENE			3400 U						1800 U
2,4-DIMETHYLPHENOL			3400 U						1800 U
2-METHYLNAPHTHALENE			3400 U						1800 U
4-METHYLPHENOL			3400 U						1800 U
ACENAPHTHENE			3400 U						1800 U
ACENAPHTHYLENE			3400 U						1800 U
ANTHRACENE			3400 U						1800 U
BENZO(A)ANTHRACENE			3400 U						1800 U
BENZO(A)PYRENE			3400 U						1800 U
BENZO(B)FLUORANTHENE			3400 U						1800 U
BENZO(G,H,I)PERYLENE			3400 U						1800 U
BENZO(K)FLUORANTHENE			3400 U						1800 U
BENZOIC ACID			16000 U						8700 U
BIS(2-ETHYLHEXYL)PHTHALATE			3400 U						1800 U
BUTYL BENZYL PHTHALATE			3400 U						1800 U
CARBAZOLE									
CHRYSENE			3400 U						1800 U
DI-N BUTYL PHTHALATE			3400 U						1800 U
DIBENZO(A,H)ANTHRACENE			3400 U						1800 U
DIBENZOFURAN			3400 U						1800 U
FLUORANTHENE			410 J						1800 U
FLUORENE			3400 U						1800 U
INDENO(1,2,3 CD)PYRENE			3400 U						1800 U
NAPHTHALENE			3400 U						1800 U
PENTACHLOROPHENOL			16000 U						8700 U
PHENANTHRENE			3400 U						1800 U
PYRENE			460 J						1800 U
TOTAL PAH			870						0



PAGE 2  
 SEDIMENT SAMPLES  
 PHASE I AND II RI AND FFS  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 11 OF 16

SITE	2B	2B	2B	2B	2B	2B	2B	2B	2B	2B	2B
AOC	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS	AREA A WETLANDS
PHASE	Phase I	Phase I	Phase I	Phase I	Phase I	Phase I	Phase I	Phase I	Phase I	Phase I	Phase I
LOCATION	2WSD6	2WSD7	2WSD8	2WSD9	T10A	T10B	T1A	T1B	T2A	T2A	T2A
NSAMPLE	112690-2WSD6(0-0.5)	112690-2WSD7(0-0.5)	112690-2WSD8(0-0.5)	112690-2WSD9(0-0.5)	T10-A	T10-B	T1-A	T1-B	T2-A	T2-A-O	T2-A-MAX
SAMPLE	112690-2WSD6(0-0.5)	112690-2WSD7(0-0.5)	112690-2WSD8(0-0.5)	112690-2WSD9(0-0.5)	T10-A	T10-B	T1-A	T1-B	T2-A	T2-A	T2-AMAX
MATRIX	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD	SD
TOP DEPTH	0	0	0	0	0	0	0	0	0	0	0
BOTTOM DEPTH	0.5	0.5	0.5	0.5	1	1	1	1	1	1	1
SAMPLE DATE	---	---	---	---	---	---	---	---	---	---	---
Volatile Organics (ug/kg)											
2-BUTANONE	86 U	110 U	20 U	40 U	14 U	21 U	12 U	11 U	520	420	520
ACETONE	240	720	190	40 U	25 U	67 U	12 U	11 U	49 U	65 U	85 U
CARBON DISULFIDE	9 J	56 U	15	20 U	14 U	12 J	12 U	11 U	16 U	15 U	16 U
CHLOROBENZENE	43 U	56 U	10 U	20 U	14 U	22 U	12 U	11 U	16 U	2 J	2
METHYLENE CHLORIDE	10 J	56 U	2 J	5 J	14 U	32 U	12 U	12 U	16 U	19 U	19 U
TETRACHLOROETHENE	10 J	56 U	10 U	11 J	14 U	22 U	12 U	11 U	16 U	15 UJ	16 U
TOLUENE	43 U	56 U	10 U	20 U	3 J	22 U	12 U	11 U	5 J	2 J	5
TOTAL XYLENES	43 U	56 U	10 U	20 U	14 U	22 UJ	12 U	11 U	16 U	15 UJ	16 U
TRICHLOROETHENE	43 U	56 U	10 U	6 J	14 U	22 U	12 U	11 U	16 U	15 U	16 U
Semivolatile Organics (ug/kg)											
1,4-DICHLOROBENZENE	2800 U	3700 U	3300 U	6600 U	470 U	730 U	410 U	380 U	520 U	480 U	520 U
2,4-DIMETHYLPHENOL	2800 U	3700 U	3300 U	6600 U	470 U	730 U	410 U	380 U	520 U	480 UJ	520 U
2-METHYLNAPHTHALENE	2800 U	3700 U	3300 U	6600 U	40 J	730 U	410 U	380 UJ	520 U	480 UJ	520 U
4-METHYLPHENOL	2800 U	3700 U	3300 U	6600 U	470 U	730 U	410 U	380 U	520 U	480 U	520 U
ACENAPHTHENE	2800 U	3700 U	3300 U	6600 U	110 J	730 U	410 U	380 UJ	30 J	91 J	91
ACENAPHTHYLENE	2800 U	3700 U	3300 U	6600 U	470 U	730 U	410 U	380 UJ	520 U	480 UJ	520 U
ANTHRACENE	2800 U	3700 U	3300 U	2400 J	48 J	730 U	410 U	40 J	71 J	200 J	200
BENZO(A)ANTHRACENE	2800 U	3700 U	3300 U	27000	290 J	90 J	45 J	180 J	260 J	680 J	680
BENZO(A)PYRENE	2800 U	3700 U	3300 U	35000	320 J	110 J	55 J	190 J	290 J	690 J	690
BENZO(B)FLUORANTHENE	2800 U	3700 U	420 J	55000	270 J	110 J	54 J	220 J	310 J	660 J	660
BENZO(G,H,I)PERYLENE	2800 U	3700 U	3300 U	23000	290 J	730 U	50 J	160 J	230 J	630 J	630
BENZO(K)FLUORANTHENE	2800 U	3700 U	670 J	45000	300 J	120 J	62 J	150 J	220 J	520 J	520
BENZOIC ACID	14000 U	780 J	16000 U	32000 J	2500 U	3800 U	2100 U	2000 U	2600 U	2500 U	2600 U
BIS(2-ETHYLHEXYL)PHTHALATE	2800 U	3700 U	3300 U	6600 U	470 U	730 U	410 U	380 U	520 U	480 U	520 U
BUTYL BENZYL PHTHALATE	2800 U	3700 U	3300 U	6600 U	36 J	730 U	410 U	380 U	520 U	480 U	520 U
CARBAZOLE					470 U	730 U	410 U	380 U	32 J	480 U	32
CHRYSENE	2800 U	3700 U	610 J	42000	360 J	110 J	65 J	220 J	340 J	780 J	780
DI-N-BUTYL PHTHALATE	2800 U	3700 U	3300 U	7900 U	470 U	730 U	410 U	380 U	520 U	31 J	31
DIBENZO(A,H)ANTHRACENE	2800 U	3700 U	3300 U	6600 U	470 U	730 U	410 U	380 U	100 J	310 J	310
DIBENZOFURAN	2800 U	3700 U	3300 U	1000 J	62 J	730 U	410 U	380 UJ	520 U	44 J	44
FLUORANTHENE	2800 U	470 J	1300 J	80000	490	100 J	110 J	240 J	490 J	1000 J	1000
FLUORENE	2800 U	3700 U	3300 U	1000 J	83 J	730 U	410 U	380 UJ	37 J	95 J	95
INDENO(1,2,3-CD)PYRENE	2800 U	3700 U	3300 U	23000	260 J	86 J	41 J	140 J	200 J	550 J	550
NAPHTHALENE	2800 U	3700 U	3300 U	6600 U	73 J	730 U	410 U	380 UJ	520 U	480 UJ	520 U
PENTACHLOROPHENOL	14000 U	18000 U	16000 U	32000 U	1100 U	1800 U	1000 U	240 J	1200 U	1200 U	1200 U
PHENANTHRENE	2800 U	3700 U	440 J	36000	280 J	79 J	43 J	190 J	260 J	920 J	920
PYRENE	470 J	610 J	1500 J	42000 J	720	320 J	96 J	470	560 J	2000 J	2000
TOTAL PAH	470	1080	4940	411400	3934	1125	621	2200	3398	9126	9126

TABLE 2

SEDIMENT SAMPLES  
 PHASE I AND II RI AND FFS  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 12 OF 16

SITE	2B AREA A WETLANDS										
PHASE	Phase I										
LOCATION	2WSD6	2WSD7	2WSD8	2WSD9	T10A	T10B	T1A	T1B	T2A	T2A	T2A
NSAMPLE	112690-2WSD6(0-0.5)	112690-2WSD7(0-0.5)	112690-2WSD8(0-0.5)	112690-2WSD9(0-0.5)	T10-A	T10-B	T1-A	T1-B	T2-A	T2-A	T2-A
SAMPLE	112690-2WSD6(0-0.5)	112690-2WSD7(0-0.5)	112690-2WSD8(0-0.5)	112690-2WSD9(0-0.5)	T10-A	T10-B	T1-A	T1-B	T2-A	T2-A	T2-A
MATRIX	SD										
TOP DEPTH	0	0	0	0	0	0	0	0	0	0	0
BOTTOM DEPTH	0.5	0.5	0.5	0.5	1	1	1	1	1	1	1
SAMPLE DATE	---	---	---	---	---	---	---	---	---	---	---
Pesticides/PCBs (ug/kg)											
4,4'-DDD	28 UJ	36 UJ	32 UJ	64 UJ	190 J	35 J	9 J	12 R	650 J	250 J	650
4,4'-DDE	28 UJ	36 UJ	32 UJ	64 UJ	23 J	7.4 J	4.1 U	3.8 U	39 J	13 J	30
4,4'-DDT	28 UJ	36 UJ	32 UJ	64 UJ	85 J	7.3 UR	15 R	45	44	60 J	60
ALDRIN	14 UJ	18 UJ	16 UJ	32 UJ	2.4 UJ	3.8 UJ	2.1 U	2 U	2.7 U	2.5 U	2.7 U
ALPHA-CHLORDANE	140 UJ	180 UJ	160 UJ	320 UJ	5.5 J	3.8 U	4.8 J	2.9 J	2.7 U	2.5 U	2.7 U
AROCLOR-1260	280 UJ	360 UJ	320 UJ	640 UJ	4.7 U	7.3 U	150 J	180	52 U	49 U	52 U
TOTAL AROCLOR	0 U	0 U	0 U	0 U	0 U	0 U	150	180	0 U	0 U	0 U
BETA-BHC	14 UJ	18 UJ	16 UJ	32 UJ	2.4 UJ	3.8 UJ	2.1 U	2 U	2.7 U	2.5 U	2.7 U
DELTA-BHC	14 UJ	18 UJ	16 UJ	32 UJ	2.4 UJ	3.8 UJ	2.1 U	2 U	2.7 U	2.5 U	2.7 U
DIELDRIN	28 UJ	36 UJ	32 UJ	64 UJ	4.7 U	7.3 U	4.1 U	3.8 U	5.2 U	4.9 U	5.2 U
ENDOSULFAN I	14 UJ	18 UJ	16 UJ	32 UJ	4.8 J	3.8 U	2.4 J	2.1 J	2.7 U	2.5 U	2.7 U
ENDOSULFAN II	28 UJ	36 UJ	32 UJ	64 UJ	4.7 U	7.3 U	4.1 U	3.8 U	3.1 J	4.9 UJ	3.1
ENDOSULFAN SULFATE	28 UJ	36 UJ	32 UJ	64 UJ	4.7 U	7.3 U	4.1 U	3.8 U	5.2 U	4.9 U	5.2 U
ENDRIN	28 UJ	36 UJ	32 UJ	64 UJ	4.7 U	7.3 U	4.1 U	3.8 U	5.2 U	4.9 U	5.2 U
ENDRIN ALDEHYDE					4.7 U	7.3 U	4.1 U	3.8 U	5.6 J	16 J	16
ENDRIN KETONE	28 UJ	36 UJ	32 UJ	64 UJ	4.7 U	7.3 U	4.1 U	3.8 U	5.2 U	4.9 U	5.2 U
GAMMA-BHC (LINDANE)	14 UJ	18 UJ	16 UJ	32 UJ	2.4 UJ	3.8 UJ	2.1 U	2 U	2.7 U	2.5 U	2.7 U
GAMMA-CHLORDANE	140 UJ	180 UJ	160 UJ	320 UJ	4.5 J	3.8 U	4.8 J	3.7 J	4.8 J	2.5 UJ	4.8
HEPTACHLOR	14 UJ	18 UJ	16 UJ	32 UJ	2.4 U	3.8 U	2.8 J	2 U	2.7 U	2.5 U	2.7 U
HEPTACHLOR EPOXIDE	14 UJ	18 UJ	16 UJ	32 UJ	2.4 U	3.8 U	3.7 J	4.5	2.7 U	2.5 U	2.7 U
METHOXYCHLOR	140 UJ	180 UJ	160 UJ	320 UJ	2.4 UJ	3.8 UJ	2.1 U	2 U	2.7 U	2.5 U	2.7 U
TOTAL DDT	0	0	0	0	298	42.4	9	45	724	323	740
Inorganics (mg/kg)											
ALUMINIUM	19300	17800	18300	20800	6700	12800	3530	4240	4470	4700	4700
ANTIMONY	10.1 UR	9.4 UR	10.1 UR	9.9 UR	0.56 U	0.81 U	0.48 J	0.48 UJ	0.72 J	0.62 UJ	0.72
ARSENIC	10.5 J	13.9 J	8.1 J	11.8 J	3.4	9.1	1.9	1.6	2.5	2.5	2.5
BARIUM	59.9	58.7	64	72.8	57	42.9	27.9	33.9	46.7	56.5	56.5
BERYLLIUM	0.81	0.51	0.84	0.85	0.24 J	0.62	0.23	0.24	0.48	0.7	0.7
BORON	730 R	450 R	3600 R	490 R	2.1 U	29.5	0.35 U	0.36 U	2.2 U	7.6 U	7.6 U
CADMIUM	3.5	3.5	5	6.1	0.37	0.34 J	0.29	0.12 J	0.29	0.46	0.46
CALCIUM	2090	1830	6800	2160	1650	3330	1130	1240	1510	1510	1510
CHROMIUM	93.5	69.3	95.7	63.7	15.4	39.9	8.1	9.6	15.2	16.9	16.9
COBALT	8.4	7.6	10.2	8.6	3.9	7.7	3.6	4.3	6.8	8.4	8.4
COPPER	51.3 J	34.6 J	71.5	39.6 J	31.4	18.8	79.6	45.5	104	101	104
CYANIDE	2.5 U	2.4 U	2.5 U	3.2	0.7 UJ	1 UJ					
IRON	22900	24100	25500	44000	12400	27500	5980	7740	10300	15600	15600
LEAD	69	37.8	69.2	241 J	71.3	16.1	23.9	28.7	123	142	142
MAGNESIUM	6700	6060	7300	5880	2770	7340	1750	2120	1940	2000	2000
MANGANESE	210	193	278	357	121	323	132	125	78.8	99.7	99.7
MERCURY	0.32 J	0.42 J	0.48 J	0.24 J	0.28 U	0.28 U	0.12 UR	0.36 J	0.17 J	0.24 J	0.24
NICKEL	19.9	17.6	24.2	21.2	12.7	20.8	6.6 J	9.1 J	61.5 J	53 J	61.5
POTASSIUM	3980	3790	4070	3310	1630 J	3770 J	890	1080	1120	1070	1120
SELENIUM	1.1	1.6	1.2	1.3	0.7 U	1.5 J	0.58 UJ	0.6 UJ	0.68 UJ	0.77 UJ	0.77 U
SILVER	2.8 UJ	2.6 UJ	2.8 UJ	2.8 UJ	0.14 U	0.2 U	0.37	0.73	0.17 J	0.22	0.22
SODIUM	1150 J	464 J	6650	321 J	253	3870	114	215	512	556	556
VANADIUM	49.3	44.8	41	55.9	38.7	39.1	17.2 J	13.2 J	161 J	183 J	183
ZINC	57.6 J	51.5 J	124	109	133 J	66.5 J	63.6 J	100 J	225 J	378 J	378
Miscellaneous Parameters (mg/kg)											
TOTAL ORGANIC CARBON					52400	68300	32100	24500		47500	47500

E 2  
 SEDIMENT SAMPLES  
 PHASE I AND II RI AND FFS  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 13 OF 16

SITE	2B						
AOC	AREA A WETLANDS						
PHASE	FFS						
LOCATION	T2B	T3A	T3B	T4A	T4B	T5A	T5B
NSAMPLE	T2-B	T3-A	T3-B	T4-A	T4-B	T5-A	T5-B
SAMPLE	T2-B	T3-A	T3-B	T4-A	T4-B	T5-A	T5-B
MATRIX	SD						
TOP DEPTH	0	0	0	0	0	0	0
BOTTOM DEPTH	1	1	1	1	1	1	1
SAMPLE DATE	---	---	---	---	---	---	---
Volatile Organics (ug/kg)							
2-BUTANONE	32 U	12 U	15 U	12 U	12 U	17 U	12 U
ACETONE	53 U	12 U	18 U	12 U	12 U	30 U	12 U
CARBON DISULFIDE	17 U	12 U	15 U	12 U	12 U	14 U	12 U
CHLOROBENZENE	17 U	12 U	15 U	12 U	12 U	14 U	12 U
METHYLENE CHLORIDE	17 U	12 U	19 U	24 U	35 U	39 U	29 U
TETRACHLOROETHENE	17 U	12 U	15 U	12 U	12 U	14 U	12 U
TOLUENE	17 U	12 U	3 J	12 U	12 U	14 U	12 U
TOTAL XYLENES	17 U	12 U	15 U	12 U	12 U	14 U	12 U
TRICHLOROETHENE	17 U	12 U	15 U	12 U	12 U	14 U	12 U
Semivolatile Organics (ug/kg)							
1,4-DICHLOROBENZENE	550 U	390 U	500 U	380 U	400 U	480 U	380 U
2,4-DIMETHYLPHENOL	550 U	390 U	500 U	390 U	400 U	480 U	380 U
2-METHYLNAPHTHALENE	550 U	55 J	500 U	380 U	400 U	480 U	380 U
4-METHYLPHENOL	550 U	390 U	500 U	380 U	400 U	480 U	380 U
ACENAPHTHENE	550 U	58 J	500 U	380 U	400 U	220 J	380 U
ACENAPHTHYLENE	550 U	390	70 J	390 U	400 U	34 J	380 U
ANTHRACENE	58 J	450	90 J	45 J	400 U	43 J	380 U
BENZO(A)ANTHRACENE	260 J	3000	480 J	120 J	77 J	180 J	25 J
BENZO(A)PYRENE	300 J	2600	480 J	130 J	74 J	220 J	30 J
BENZO(B)FLUORANTHENE	550 U	390 U	420 J	130 J	85 J	210 J	44 J
BENZO(G,H,I)PERYLENE	240 J	1100	320 J	100 J	66 J	230 J	27 J
BENZO(K)FLUORANTHENE	440 J	3700	510	130 J	75 J	270 J	380 U
BENZOIC ACID	2800 U	2000 U	2600 U	1800 U	2000 U	2400 U	2000 U
BIS(2-ETHYLHEXYL)PHTHALATE	550 U	400 U	500 U	380 U	400 U	480 U	380 U
BUTYL BENZYL PHTHALATE	550 U	390 U	51 J	21 J	400 U	480 U	380 U
CARBAZOLE	550 U	57 J	32 J	25 J	400 U	130 J	380 U
CHRYSENE	350 J	3700	590	150 J	90 J	280 J	38 J
DI-N-BUTYL PHTHALATE	550 U	390 U	63 J	26 J	23 J	44 J	26 J
DIBENZO(A,H)ANTHRACENE	87 J	390 U	170 J	380 U	400 U	480 U	380 U
DIBENZOFURAN	550 U	35 J	500 U	380 U	400 U	72 J	380 U
FLUORANTHENE	450 J	4200	890	260 J	180 J	520	56 J
FLUORENE	550 U	160 J	38 J	21 J	400 U	29 J	380 U
INDENO(1,2,3-CD)PYRENE	200 J	1000	300 J	92 J	57 J	180 J	22 J
NAPHTHALENE	550 U	56 J	500 U	360 U	400 U	76 J	380 U
PENTACHLOROPHENOL	1300 U	950 U	1200 U	930 U	960 U	1200 U	930 U
PHENANTHRENE	230 J	1800	320 J	180 J	93 J	240 J	22 J
PYRENE	620	4600	910	220 J	150 J	440 J	46 J
TOTAL PAH	3235	26869	5588	1578	947	3172	310

TABLE 2  
 SEDIMENT SAMPLES  
 PHASE I AND II RI AND FFS  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 14 OF 16

SITE	2B						
AOC	AREA A WETLANDS						
PHASE	FFS						
LOCATION	T2B	T3A	T3B	T4A	T4B	T5A	T5B
NSAMPLE	T2-B	T3-A	T3-B	T4-A	T4-B	T5-A	T5-B
SAMPLE	T2-B	T3-A	T3-B	T4-A	T4-B	T5-A	T5-B
MATRIX	SD						
TOP DEPTH	0	0	0	0	0	0	0
BOTTOM DEPTH	1	1	1	1	1	1	1
SAMPLE DATE	---	---	---	---	---	---	---
<b>Pesticides/PCBs (ug/kg)</b>							
4,4'-DDD	210 J	390	1400	38 U	45 J	1300	38 U
4,4'-DDE	19 J	260	330	38 U	4 U	57 J	38 U
4,4'-DDT	88 J	890	2900	38 U	4 U	380	44 J
ALDRIN	28 U	20 U	26 U	2 U	2 U	32 J	2 U
ALPHA-CHLORDANE	76	44 J	12 J	2 U	2 U	13	2 U
AROCLOR-1260	55 U	390 U	500 U	38 U	40 U	240 U	38 U
TOTAL AROCLOR	0 U	0 U	0 U	0 U	0 U	0 U	0 U
BETA-BHC	28 U	20 U	26 U	2 U	2 U	27 J	2 U
DELTA-BHC	28 U	20 U	26 U	2 U	2 U	42 J	2 U
DIELDRIN	55 U	39 U	50 U	38 U	4 U	84 J	38 U
ENDOSULFAN I	28 U	11 J	26 U	2 U	2 U	12 U	2 U
ENDOSULFAN II	69 J	39 U	50 U	38 U	4 U	24 U	38 U
ENDOSULFAN SULFATE	55 U	14 J	50 U	38 U	4 U	69 J	38 U
ENDRIN	55 U	39 U	50 U	38 U	4 U	16 J	38 U
ENDRIN ALDEHYDE	55 U	39 U	16 J	38 U	4 U	10 J	38 U
ENDRIN KETONE	55 U	39 U	50 U	38 U	4 U	20 J	38 U
GAMMA BHC (LINDANE)	28 U	20 U	26 U	2 U	2 U	35 J	2 U
GAMMA-CHLORDANE	51	20 U	13 J	2 U	2 U	87 J	2 U
HEPTACHLOR	28 U	20 U	26 U	2 U	2 U	45 J	2 U
HEPTACHLOR EPOXIDE	37 J	20 U	26 U	2 U	2 U	22 J	2 U
METHOXYCHLOR	28 U	200 U	260 U	20 U	20 U	38 J	20 U
TOTAL DDT	317	1540	4630	0	45	1737	44
<b>Inorganics (mg/kg)</b>							
ALUMINUM	6020	3820	5040	3570	2980	5080	2690
ANTIMONY	0.52 UJ	0.53 UJ	0.58 UJ	0.47 UJ	0.47 UJ	0.6 UJ	0.47 UJ
ARSENIC	22	3	2.6	1.4	1.4	2.1	1 J
BARIUM	73.8	28.8	38.4	29.2	41.1	59.4	11.9
BERYLLIUM	0.32	0.21 J	0.33	0.16 J	0.14 J	0.26 J	0.14 J
BORON	0.46 UJ	0.4 U	0.51 U	0.35 U	0.36 U	1.3 U	0.35 U
CADMIUM	0.3	0.26	0.43	0.12 U	0.21 J	0.15 U	0.12 U
CALCIUM	1300	900	868	1060	934	1630	1220
CHROMIUM	19.5	8.6	11.1	8	7	15	7.7
COBALT	5	4.4	7.9	3	3.2	4.2	3.3
COPPER	44.9	34.5	52.6	14.7	36.7	28.4	19.5
CYANIDE				1.5	0.59 U	0.9 J	0.9 J
IRON	12500	9390	8020	5800	5630	10600	5660
LEAD	42	32.1	61.5	26.5	32.4	40.6	16.2
MAGNESIUM	2860	1650	2200	1770	1570	2280	1820
MANGANESE	132	310	99.8	90.1	55.3	101	89.3
MERCURY	0.32 J	0.15 J	0.19 J	0.12 UR	0.12 UR	0.15 UR	0.12 UR
NICKEL	17 J	8.6 J	18.9 J	6.7 J	10.3 J	13.8 J	8.5 J
POTASSIUM	1890	813	1100	1120	926	1290	659
SELENIUM	0.77 UJ	0.67 UJ	0.73 UJ	0.59 UJ	0.59 UJ	0.75 UJ	0.58 UJ
SILVER	0.15 U	0.13 U	0.15 U	0.12 U	0.12 U	0.15 U	0.12 U
SODIUM	516	285	1050	250	462	2480	388
VANADIUM	66.4 J	17 J	18.5 J	12.8 J	11.2 J	37.4 J	8.9 J
ZINC	95.4 J	87.4 J	284 J	39.2 J	45.1 J	68.2 J	35 J
<b>Miscellaneous Parameters (mg/kg)</b>							
TOTAL ORGANIC CARBON	63400	31100	33500	9960	8420	16900	9410

SEDIMENT SAMPLES  
 PHASE I AND II RI AND FFS  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 15 OF 16

SITE	2B									
AOC	AREA A WETLANDS									
PHASE	FFS									
LOCATION	T6A	T6A	T6A	T6B	T7A	T7B	T8A	T8B	T9A	T9B
NSAMPLE	T6-A	T6-A-D	T6-A-MAX	T6-B	T7-A	T7-B	T8-A	T8-B	T9-A	T9-B
SAMPLE	T6-A	DUP-5	T6-AMAX	T6-B	T7-A	T7-B	T8-A	T8-B	T9-A	T9-B
MATRIX	SD									
TOP DEPTH	0	0	0	0	0	0	0	0	0	0
BOTTOM DEPTH	1	1	1	1	1	1	1	1	1	1
SAMPLE DATE	---	---	---	---	---	---	---	---	---	---
Volatile Organics (ug/kg)										
2-BUTANONE	19 UJ	19 U	19 U	21 U	37 U	120 UR	27 U	1400	34 U	59
ACETONE	19 U	19 U	19 U	40 U	76 U	260 UR	67 U	110 U	90 U	21 U
CARBON DISULFIDE	19 UJ	19 U	19 U	24 U	19 U	67 UR	21 U	22 U	17 U	3 J
CHLOROBENZENE	19 UJ	19 UJ	19 U	3 J	19 U	14 J	21 U	22 U	17 UJ	21 U
METHYLENE CHLORIDE	62 U	61 U	62 U	74 U	38 U	210 UR	21 U	42 U	17 U	21 U
TETRACHLOROETHENE	19 UJ	19 UJ	19 U	24 UJ	19 U	67 UR	21 U	22 U	17 UJ	21 U
TOLUENE	3 J	3 J	3	24 UJ	5 J	67 UR	21 U	6 J	17 UJ	21 U
TOTAL XYLENES	19 UJ	19 UJ	19 U	3 J	19 U	67 UR	21 U	22 UJ	17 UJ	21 U
TRICHLOROETHENE	19 UJ	19 U	19 U	24 U	19 U	67 UR	21 U	22 U	17 U	21 U
Semivolatile Organics (ug/kg)										
1,4-DICHLOROBENZENE	620 U	630 UJ	630 U	42 J	620 U	2200 UR	690 U	730 U	570 U	700 U
2,4-DIMETHYLPHENOL	620 U	630 UJ	630 U	800 UJ	620 U	2200 UR	690 U	210 J	570 U	700 U
2-METHYLNAPHTHALENE	620 U	630 UJ	630 U	800 UJ	620 U	2200 UR	49 J	730 U	51 J	700 U
4-METHYLPHENOL	620 U	630 UJ	630 U	800 UJ	620 U	2200 UR	690 U	730 U	43 J	700 U
ACENAPHTHENE	620 U	630 UJ	630 U	110 J	77 J	380 J	35 J	730 U	48 J	700 U
ACENAPHTHYLENE	52 J	630 UJ	52	800 UJ	620 U	2200 UR	690 U	730 U	570 U	700 U
ANTHRACENE	56 J	34 J	56	800 U	79 J	2200 UR	71 J	38 J	34 J	52 J
BENZO(A)ANTHRACENE	190 J	280 J	280	140 J	390 J	240 J	370 J	210 J	140 J	230 J
BENZO(A)PYRENE	230 J	310 J	310	160 J	430 J	340 J	340 J	210 J	160 J	260 J
BENZO(B)FLUORANTHENE	220 J	390 J	390	240 J	430 J	370 J	370 J	210 J	130 J	240 J
BENZO(G,H,I)PERYLENE	250 J	420 J	420	290 J	350 J	340 J	300 J	180 J	220 J	190 J
BENZO(K)FLUORANTHENE	330 J	240 J	330	140 J	320 J	270 J	350 J	210 J	140 J	200 J
BENZOIC ACID	3200 U	3200 U	3200 U	4100 U	3200 U	11000 UR	3500 U	3800 U	3000 U	3600 U
BIS(2-ETHYLHEXYL)PHTHALATE	620 U	630 U	630 U	800 U	620 U	4900 UR	690 U	730 U	570 U	700 U
BUTYL BENZYL PHTHALATE	50 J	630 U	50	800 U	620 U	390 J	690 U	730 U	570 UJ	700 U
CARBAZOLE	43 J	630 UJ	43	800 U	620 U	2200 UR	690 U	730 U	570 U	700 U
CHRYSENE	300 J	430 J	430	230 J	410 J	340 J	490 J	280 J	230 J	260 J
DI-N-BUTYL PHTHALATE	56 J	34 J	56	800 U	33 J	2200 UR	690 U	730 U	570 U	700 U
DIBENZO(A,H)ANTHRACENE	86 J	630 U	86	800 U	130 J	2200 UR	690 UJ	730 U	72 J	700 U
DIBENZOFURAN	620 U	630 UJ	630 U	68 J	55 J	280 J	40 J	730 U	35 J	700 U
FLUORANTHENE	500 J	330 J	500	200 J	650	560 J	520 J	310 J	160 J	220 J
FLUORENE	620 U	630 UJ	630 U	69 J	90 J	340 J	79 J	730 U	47 J	700 U
INDENO(1,2,3-CD)PYRENE	210 J	330 J	330	220 J	360 J	350 J	260 J	150 J	140 J	170 J
NAPHTHALENE	620 U	630 UJ	630 U	800 UJ	620 U	2200 UR	57 J	730 U	77 J	700 U
PENTACHLOROPHENOL	1500 U	1500 UJ	1500 U	2000 U	1500 U	5300 UR	1700 U	1800 U	1400 U	1700 U
PHENANTHRENE	200 J	220 J	220	140 J	350 J	300 J	260 J	180 J	160 J	140 J
PYRENE	470 J	900 J	900	370 J	530 J	450 J	1300	630 J	430 J	690 J
TOTAL PAH	3094	3884	4304	2309	4596	4280	4851	2608	2239	2652

TABLE 2  
 SEDIMENT SAMPLES  
 PHASE I AND II RI AND FFS  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 16 OF 16

SITE	2B									
AOC	AREA A WETLANDS									
PHASE	FFS									
LOCATION	T6A	T6A	T6A	T6B	T7A	T7B	T8A	T8B	T9A	T9B
NSAMPLE	T6-A	T6-A-D	T6-A-MAX	T6-B	T7-A	T7-B	T8-A	T8-B	T9-A	T9-B
SAMPLE	T6-A	DUP-5	T6-AMAX	T6-B	T7-A	T7-B	T8-A	T8-B	T9-A	T9-B
MATRIX	SD									
TOP DEPTH	0	0	0	0	0	0	0	0	0	0
BOTTOM DEPTH	1	1	1	1	1	1	1	1	1	1
SAMPLE DATE	---	---	---	---	---	---	---	---	---	---
Pesticides/PCBs (ug/kg)										
4,4'-DDD	46 R	49 R	49 R	410	62 U	22 UR	86 J	73 UJ	630 J	7 UJ
4,4'-DDE	10 J	15 J	15	38	62 U	140 J	19 J	73 UJ	49 J	7 UJ
4,4'-DDT	55 R	39 R	55 R	8 U	62 U	23 R	69 UR	73 UJ	57 UR	7 UJ
ALDRIN	32 U	33 U	33 U	41 U	32 U	11 UR	35 UJ	38 UJ	29 UJ	36 UJ
ALPHA-CHLORDANE	29	29	29	15 J	32 U	22 J	35 U	38 U	6 J	36 U
AROCLOR-1260	1400	1500	1500	530	82 J	550 J	69 U	73 U	57 U	70 U
TOTAL AROCLOR	1400	1500	1500	530	82	2100	0 U	0 U	0 U	0 U
BETA-BHC	32 U	33 U	33 U	41 U	32 U	11 UR	35 UJ	38 UJ	29 UJ	36 UJ
DELTA-BHC	32 U	33 U	33 U	41 U	32 U	11 UR	35 U	38 U	29 U	36 U
DIELDRIN	26	23	26	8 U	62 U	22 UR	69 U	73 U	57 U	7 U
ENDOSULFAN I	32 U	33 U	33 U	41 U	32 U	11 UR	35 U	38 U	29 U	36 U
ENDOSULFAN II	48 R	50 R	50 R	17 R	62 U	22 UR	69 U	73 U	57 U	7 U
ENDOSULFAN SULFATE	97 R	10 R	10 R	8 U	62 U	22 UR	69 U	73 U	57 U	7 U
ENDRIN	82 J	78 J	82	8 U	62 U	22 UR	69 U	73 U	57 U	7 U
ENDRIN ALDEHYDE	22 R	23 R	23 R	8 U	62 U	22 UR	69 U	73 U	57 U	7 U
ENDRIN KETONE	54 R	12 R	12 R	8 U	62 U	22 UR	69 U	73 U	57 U	7 U
GAMMA-BHC (LINDANE)	32 U	33 U	33 U	41 U	32 U	11 UR	35 UJ	38 UJ	29 UJ	36 UJ
GAMMA-CHLORDANE	21 J	23 J	23	11 J	32 U	11 UR	35 U	38 U	29 U	36 U
HEPTACHLOR	32 U	33 U	33 U	41 U	32 U	11 UR	35 U	38 U	29 U	36 U
HEPTACHLOR EPOXIDE	32 U	33 U	33 U	41 U	32 U	11 UR	35 U	38 U	29 U	36 U
METHOXYCHLOR	31 R	39 R	39 R	41 U	32 U	110 UR	35 UJ	38 UJ	29 UJ	36 UJ
TOTAL DDT	10	15	15	448	0	140	105	0	679	0
Inorganics (mg/kg)										
ALUMINUM	15800	14800	15800	9820	7150	27100 J	14200	19900	18100	15100
ANTIMONY	0.85 UJ	0.77 UJ	0.85 U	1.1 J	1.2 J	3.1 UR	0.84 U	0.95 U	0.72 U	0.85 U
ARSENIC	72	81	81	48	25	81 J	11	141	72	108
BARIUM	113	120	120	941	124	318 J	534	779	868	477
BERYLLIUM	13	12	13	13	0.28 J	4.1 J	0.66	0.98	0.81	0.73
BORON	0.64 UJ	0.58 UJ	0.64 U	0.77 UJ	0.9 U	2.3 UR	28.7	39.3	14.7	39.6
CADMIUM	0.72	0.63	0.72	0.68	0.23 J	1.6 J	0.41 J	0.47 J	0.57	0.43 J
CALCIUM	3290	2960	3290	2230	3350	5330 J	2790	3610	2640	4110
CHROMIUM	44.5	49.8	49.8	40.2	13.7	44.3 J	80.3	96.8	48.5	59.9
COBALT	10	9.2	10	4.7	8.1	13.6 J	8.1	9.6	8.1	9
COPPER	133	114	133	82	33.8	173 J	48.4	64.2	48.1	37.2
CYANIDE	2 J	2.1	2.1	1.8 J	1.1 U	6.1 J	1.1 UJ	1.2 UJ	0.9 UJ	1.1 UJ
IRON	47500	53100	53100	35700	19200	198000 J	24500	37100	25500	30300
LEAD	141	109	141	78.4	17.4	110 J	61.2	66.6	40.6	39.3
MAGNESIUM	5550	4800	5550	2660	4170	3900 J	7230	9150	6100	8450
MANGANESE	285	271	285	134	154	269 J	240	338	313	355
MERCURY	0.21 UR	0.43 J	0.43	1.2 J	0.44 J	0.76 UR	0.4 U	0.51 U	0.27 U	0.21 UJ
NICKEL	29 J	26.2 J	29	15.8 J	12.2 J	47.7 J	21.7	27.8	20.7	23.8
POTASSIUM	2880	2650	2880	1490	3000	2030 J	3880 J	5170 J	3300 J	4410 J
SELENIUM	1.1 UJ	2.2 J	2.2	1.3 UJ	1.1 UJ	6.8 J	2.2 J	3 J	1.8 J	1.9 J
SILVER	0.96	0.81	0.96	0.85	0.22 U	0.86 J	0.33 J	0.47 J	0.18 J	0.21 U
SODIUM	1130	966	1130	725	926	1410 J	2980	3320	1930	4980
VANADIUM	176 J	152 J	176	120 J	38.2 J	203 J	45.3	59.5	49	43.8
ZINC	213 J	195 J	213	172 J	76.3 J	702 J	73 J	95.5 J	132 J	82.8 J
Miscellaneous Parameters (mg/kg)										
TOTAL ORGANIC CARBON	11200	32600		25000	54800	63000	84100	79600	34700	61600

TABLE 3  
 SURFACE WATER SAMPLES  
 PHASES I AND II RIS  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 1 OF 2

SITE	2B						
AOC	AREA A WETLANDS						
PHASE	PH1	PH2-1	PH2-1	PH2-1	PH2-1	PH1	PH1
LOCATION	2WSW1	2WSW1	2WSW10	2WSW11	2WSW12	2WSW2	2WSW2
NSAMPLE	121090-2WSW1	2WSW1	2WSW10	2WSW11	2WSW12	121090-2WSW2	121090-2WSW2-AVG
SAMPLE	121090-2WSW1	2WSW1	2WSW10	2WSW11	2WSW12	121090-2WSW2	121090-2WSW2AVG
MATRIX	SW						
TOP DEPTH	---	---	---	---	---	---	---
BOTTOM DEPTH	---	---	---	---	---	---	---
SAMPLE DATE	12/10/90	11/30/93	12/05/93	11/23/93	11/23/93	12/10/90	12/10/90
<b>Volatile Organics (ug/L)</b>							
TETRACHLOROETHENE	5 U	10 U	10 U	10 U	2 J	5 U	5 U
<b>Semivolatile Organics (ug/L)</b>							
DIETHYL PHTHALATE	10 U					10 U	35 J
<b>Radiochemistry (pci/L)</b>							
GROSS ALPHA	0.4					0.7	1.6
GROSS BETA	1.6					6.9	8.95
<b>Total Inorganics (ug/L)</b>							
ALUMINUM	137	20900	1200	99.8	66.9 J	153	125.6
ARSENIC	3 U	2.9	2 U	2.8	2.8	3 U	3 U
BARIIUM	22.3	76.1 U	10.4 UJ	45.8 U	97.6	23.8	25
BORON	17 R	270	50 U	197	154	2.1 R	2.2 R
CADMIUM	137	2.8 U	2 UJ	2 UJ	2 UJ	126 J	66.55 J
CALCIUM	7890	29300	1070	27000	46500	10000	10950
CHROMIUM	6.8	6.5 U	3.6 UJ	3 U	3 U	5 U	5 U
COBALT	5 U	63.9	5 UJ	5 U	5 U	5 U	5 U
COPPER	13 J	29.3 J	6.1 J	2 UJ	2 UJ	13.2 J	11.45 J
IRON	293 J	9030	1070 J	7100	11300	4010 J	3415 J
LEAD	7.8	2 J	4.5 J	1 R	1 R	2	1.5
MAGNESIUM	2070	18500	443	8620	10300	4350	4635
MANGANESE	52 J	1860	43.7 J	624	596	136	131
MERCURY	0.2 U	0.2 U	0.2 R	0.2 U	0.2 U	0.2 U	0.2 U
NICKEL	7 U	84.7	11 UJ	11 U	11 U	7.3	5.4
POTASSIUM	4020 J	7010	1170	8400	10300	7990 J	8475 J
SODIUM	22100	20800	1640 U	87600	111000	56700	62400
VANADIUM	20 U	3 U	4.3 J	3 U	3 U	20 U	20 U
ZINC	22.8 J	318	60.3	14.4 U	16.9 U	19.6 J	14.95 J
<b>Filtered Inorganics (ug/L)</b>							
ALUMINUM		157		94.7	95.4		
BARIIUM		16.6 U		30.1 U	83.3		
BORON		50 U		75.9 J	119		
CALCIUM		7420		25700	45300		
COPPER		2 UJ		2 UJ	2 UJ		
IRON		309		2860	19400		
LEAD		1.8 J		1 U	1 U		
MAGNESIUM		2270		8190	9630		
MANGANESE		96.1		325	571		
MERCURY		0.2 U		0.2 U	0.22 J		
POTASSIUM		2450		7990	9800		
SODIUM		32300		82100	103000		
VANADIUM		3 U		3 U	3 U		
ZINC		31.4		17.9 U	3 U		
<b>Miscellaneous Parameters (mg/L)</b>							
HARDNESS		148	16	128	160		

TABLE 3

SURFACE WATER SAMPLES  
PHASES I AND II RIS  
SITE 2B - AREA A WETLAND  
NSB-NLON, GROTON, CONNECTICUT  
PAGE 2 OF 2

SITE	2B	2B	2B	2B	2B	2B
AOC	AREA A WETLANDS					
PHASE	PH1	PH2-1	PH2-1	PH2-1	PH2-1	PH2-1
LOCATION	2WSW2	2WSW2	2WSW6	2WSW7	2WSW8	2WSW9
NSAMPLE	121090-2WSW2-D	2WSW2	2WSW6	2WSW7	2WSW8	2WSW9
SAMPLE	121090-2WSW3	2WSW2	2WSW6	2WSW7	2WSW8	2WSW9
MATRIX	SW	SW	SW	SW	SW	SW
TOP DEPTH	---	---	---	---	---	---
BOTTOM DEPTH	---	---	---	---	---	---
SAMPLE DATE	12/10/90	11/23/93	11/30/93	12/05/93	12/02/93	12/02/93
Volatile Organics (ug/L)						
TETRACHLOROETHENE	5 U	10 U	10 U	10 U	10 U	10 U
Semivolatile Organics (ug/L)						
DIETHYL PHTHALATE	2 J	10 U				
Radiochemistry (pci/L)						
GROSS ALPHA	25					
GROSS BETA	11					
Total Inorganics (ug/L)						
ALUMINUM	982	586	20800	117 U	939	717 J
ARSENIC	3 U	2 U	2 U	2 U	2 U	2 U
BARIUM	262	115	73.5 U	8 U	20 U	19 U
BORON	22 R	369	121	50 U	50 U	50 U
CADMIUM	7.1 J	2 UJ	4.4 U	2 UJ	2 UJ	2 UJ
CALCIUM	11900	36700	29800	272 J	9350	8900
CHROMIUM	5 U	3 U	4.5 U	3 UJ	3 U	3 U
COBALT	5 U	5 U	66.8	5 UJ	5 U	5 U
COPPER	97 J	3.1 U	16.8 U	7.7 J	2 UJ	2 UJ
IRON	2820 J	3320	10800	9.9 UJ	11.9 U	5.7 U
LEAD	2 U	2.1 J	5 J	1 UJ	1 R	1 R
MAGNESIUM	4920	11600	18200	94 U	2310	2260
MANGANESE	126	591	1870	6 UJ	6.4 U	6.1 U
MERCURY	0.2 U	0.2 U	0.2 U	0.2 R	0.2 U	0.21 J
NICKEL	7 U	11.1 J	82.1	11 UJ	11 U	11 U
POTASSIUM	8960 J	15300	6810	447 J	1860	2040
SODIUM	68100	143000	19700	1150 U	24200	23200
VANADIUM	20 U	3 U	3 U	3 UJ	3 U	3 U
ZINC	10.3 J	47.6	334	9.7 UJ	3 U	3.3 U
Filtered Inorganics (ug/L)						
ALUMINUM		86.3	259	136 U		
BARIUM		91.2	23.1 U	8 U		
BORON		111	222	50 U		
CALCIUM		35600	7640	256 J		
COPPER		5.3 U	2 UJ	4.5 J		
IRON		699	548	110		
LEAD		1 U	1 R	6.1 J		
MAGNESIUM		10800	2410	103 J		
MANGANESE		551	99.8	9.6 J		
MERCURY		0.2 U	0.2 U	0.2 U		
POTASSIUM		14700	2490	542 J		
SODIUM		133000	34900	720 U		
VANADIUM		3 U	3 U	3.3 J		
ZINC		29.6	27.7	17.9 UJ		
Miscellaneous Parameters (mg/L)						
HARDNESS		148	32	1 U	32	32

TABLE 4  
 TISSUE SAMPLES  
 PHASE I RI  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 1 OF 3

SITE	2/3	2/3	2/3	2/3	2/3	2/3	2/3	2/3	2/3	2/3
AOC	AREA A									
PHASE	PH1									
LOCATION	90 MBO 01	90 MBO 02	90 MBO 03	90 MBO 04	90 MBO 05	90 MBO 06	90 MBO 07	90 MBO 08	90 MBO 09	90 MBO 10
NSAMPLE	90 MBO 01	90 MBO 02	90 MBO 03	90 MBO 04	90 MBO 05	90 MBO 06	90 MBO 07	90 MBO 08	90 MBO 09	90 MBO 10
SAMPLE	90 MBO 01	90 MBO 02	90 MBO 03	90 MBO 04	90 MBO 05	90 MBO 06	90 MBO 07	90 MBO 08	90 MBO 09	90 MBO 10
MATRIX	BIRD									
TOP DEPTH	---	---	---	---	---	---	---	---	---	---
BOTTOM DEPTH	---	---	---	---	---	---	---	---	---	---
SAMPLE DATE	08/16/90	08/16/90	08/16/90	08/16/90	08/16/90	08/16/90	08/16/90	08/16/90	08/16/90	08/30/90
<b>Inorganics (mg/kg)</b>										
ALUMINUM	34	69	87	98	11	77	44	59 U	6 U	65
ANTIMONY	48 U	5 U	62	5 U	48 U	49 U	21	4.9 U	5 U	49 U
BARIUM	29 U	4	3 U	3 U	29 U	2.9 U	3 U	2.9 U	3 U	2.9 U
BERYLLIUM	0.19 U	0.2 U	0.2 U	0.2 U	0.19 U	0.2 U	0.4	0.2 U	0.2 U	0.2 U
BORON	630 J	700 J	740 J	760 J	730 J	700 J	820 J	700 J	720 J	710 J
CADMIUM	0.38 U	0.4 U	0.4 U	0.4 U	0.38 U	0.39 U	2.2	0.039 U	0.4 U	0.39 U
CALCIUM	1680	13400	9100	8000	6100	2900	2600	6000	4400	1920
CHROMIUM	0.96 U	14	1 U	0.99 U	0.95 U	0.98 U	5.9	0.98 U	1 U	1
COBALT	0.96 U	1 U	1 U	0.99 U	0.95 U	0.98 U	4.5	-0.98 U	1 U	0.98 U
COPPER	7.9	4.8	7.3	7.6	11	6.2	11	3.1	4.3	4.2
CYANIDE	2.5 U									
IRON	170 J	65 J	90 J	66 J	78 J	430 J	130 J	76 J	150 J	83 J
LEAD	0.38 U	2.3	1	1.1	1.9	1	0.4 U	0.98	0.4 U	0.4 U
MAGNESIUM	210	430	330	340	320	270	310	320	280	230
MANGANESE	13	15	18	15	24	1.7	2.5	0.9	1.6	2.2
MERCURY	0.1 UJ	0.091 UJ	0.095 UJ	0.091 UJ	0.074 UJ	0.057 UJ	0.083 UJ	0.087 UJ	0.083 UJ	0.087 UJ
NICKEL	15	14 U	14 U	14 U	13 U	1.6	8	1.4 U	1.4 U	1.4 U
POTASSIUM	2900	3100	3200	3100	3200	3400	4100	3100	3300	3300
SELENIUM	1.1	1	0.94	0.77	0.8	0.67	0.52	0.39	0.46	0.37
SILVER	1.3 UR	1.4 UR	1.4 UR	1.4 UR	1.3 UR	1.4 UR	8.4 R	1.4 UR	1.4 UR	1.4 R
SODIUM	1300	1300	1200	1200	1200	1200	1400	1100	1100	1100
VANADIUM	3.8 U	4 U	4 U	4 U	3.8 U	3.9 U	6.3	3.9 U	4 U	3.9 U
ZINC	17 J	30 J	24 J	24 J	22 J	22 J	16 J	21 J	20 J	14 J
<b>Miscellaneous Parameters (%)</b>										
LIPIDS	17	15	13	16	14	13	15	16	15	17

TABLE 4  
 TISSUE SAMPLES  
 PHASE I RI  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 2 OF 3

SITE	2/3	2/3	2/3	2/3	2/3	2/3	2/3	2/3	2/3
AOC	AREA A	AREA A							
PHASE	PH1	PH1							
LOCATION	90 MBO 11	90 MBO 12	90 MBO 13	90 MBO 14	90 MBO 15	90 MBO 16	90 MBO 17	90 MBO 18	90 MBO 18
NSAMPLE	90 MBO 11	90 MBO 12	90 MBO 13	90 MBO 14	90 MBO 15	90 MBO 16	90 MBO 17	90 MBO 18 FROG CONTROL-LI	90 MBO 18 FROG CONTROL-TI
SAMPLE	90 MBO 11	90 MBO 12	90 MBO 13	90 MBO 14	90 MBO 15	90 MBO 16	90 MBO 17	90 MBO 18 FROG CONTROL-LI	90 MBO 18 FROG CONTROL-TI
MATRIX	BIRD	FROG	FROG						
TOP DEPTH	---	---	---	---	---	---	---	---	---
BOTTOM DEPTH	---	---	---	---	---	---	---	---	---
SAMPLE DATE	08/29/90	08/29/90	08/30/90	08/30/90	09/28/90	09/27/90	09/29/90	08/10/90	08/10/90
Inorganics (mg/kg)									
ALUMINUM	8.7	11	41	5.9 U	11	6.5	5.9 U		13
ANTIMONY	4.7 U	5 U	23	4.9 U	5.8	4.8 U	4.9 U		5 U
BARIUM	2.8 U	3 U	4	2.9 U	3.1	2.9 U	2.9 U		3 U
BERYLLIUM	0.19 U	0.2 U	0.35	0.2 U	0.19 U	0.19 U	0.2 U		0.2 U
BORON	670 J	670 J	750 J	700 J	670 J	590 J	710 J		450 J
CADMIUM	0.8 U	0.8 U	2	0.8 U	0.8 U	0.8 U	0.8 U		0.4 U
CALCIUM	7100	7000	5800	8400	13600	4900	3900		240 J
CHROMIUM	0.94 U	0.99 U	4.9	0.98 U	0.94 U	0.96 U	0.98 U		0.99 U
COBALT	0.94 U	0.99 U	3.8	0.98 U	0.94 U	0.96 U	0.98 U		0.99 U
COPPER	3.7	4.7	9.2	0.98 U	3.2	5.5	4.8		1.1
CYANIDE	2.5 U		56						
IRON	120 J	110 J	170 J	67 J	91 J	95 J	130 J		13
LEAD	0.47	1.6	0.5	0.53	0.77	0.42	0.41		0.4 U
MAGNESIUM	350	320	370	310	430	260	280		240
MANGANESE	1.5	1.1	2.6	2.5	2.2	1.5	1.1		0.68
MERCURY	0.063 UJ	0.08 UJ	0.1 UJ	0.083 J	0.091 UJ	0.087 UJ	0.091 UJ		0.091 U
NICKEL	1.3 U	1.4 U	7	1.4 U	1.3 U	1.3 U	1.4 U		1.4 U
POTASSIUM	3300	3000	4200	2700	3000	3000	3100		3000
SELENIUM	1	0.67 U	0.67 U	0.35	0.38	0.19	0.27		0.65
SILVER	1.3 U	1.4 U	7.5	1.4 U	1.3 U	1.3 U	1.4 U		1.4 U
SODIUM	1200	1200	1500	1100	1200	1100	1100		860
VANADIUM	3.8 U	4 U	5.7	3.9 U	3.8 U	3.8 U	3.9 U		4 U
ZINC	21 J	19 J	21 J	22 J	26 J	17 J	17 J		7.4
Miscellaneous Parameters (%)									
LIPIDS	18	16	12.7	17.5	18	18	17	7.5	0

TABLE 4  
 TISSUE SAMPLES  
 PHASE I RI  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 3 OF 3

SITE	2B							
AOC	AREA A WETLANDS							
PHASE	PH1							
LOCATION	POND 1A	POND 1A	POND 1B	POND 1B	POND 1C	POND 1C	POND 1D	POND 1D
NSAMPLE	POND 1A LIVER	POND 1A MUSCLE	POND 1B LIVER	POND 1B MUSCLE	POND 1C LIVER	POND 1C MUSCLE	POND 1D LIVER	POND 1D MUSCLE/BONE
SAMPLE	POND 1A LIVER	POND 1A MUSCLE	POND 1B LIVER	POND 1B MUSCLE	POND 1C LIVER	POND 1C MUSCLE	POND 1D LIVER	POND 1D MUSCLE/BONE
MATRIX	FROG							
TOP DEPTH	--	--	--	--	--	--	--	--
BOTTOM DEPTH	--	--	--	--	--	--	--	--
SAMPLE DATE	09/14/90	09/14/90	09/14/90	09/14/90	09/14/90	09/14/90	09/14/90	09/14/90
Inorganics (mg/kg)								
ALUMINUM		470		16 J		26 J		75
ANTIMONY		49 U		49 U		5 U		49 U
BARIUM		2.9 U		2.9 U		3 U		2.9 U
BERYLLIUM		0.2 U		0.19 U		0.2 U		0.2 U
BORON		500 J		510 J		470 J		420
CADMIUM		0.39 U		0.39 U		0.4 U		0.39 U
CALCIUM		3000		170 J		270 J		240 J
CHROMIUM		1.2		0.97 U		1 U		0.98 U
COBALT		0.98 U		0.97 U		1 U		0.98 U
COPPER		2.1		0.97 U		1 U		1
CYANIDE		4.7 U		17		5.8		4.1
IRON		44 J		15 J		15 J		22
LEAD		0.39 U		0.66		0.4 U		0.78
MAGNESIUM		270		120		220		220
MANGANESE		3.5 J		2 J		3.8 J		0.58
MERCURY		0.1 U		0.1 U		0.1 U		0.091 U
NICKEL		1.4 U		1.4 U		1.4 U		1.4 U
POTASSIUM		3000 J		3000 J		3300 J		3100
SELENIUM		0.65		0.58		0.4		0.88
SILVER		1.8 R		1.4 UR		1.4 UR		1.4 U
SODIUM		1000 J		850 J		680 J		930
VANADIUM		3.9 U		3.9 U		4 U		3.9 U
ZINC		11 J		7.2 J		6.4 J		7.8
Miscellaneous Parameters (%)								
LIPIDS	24.4	4	18.5	1	0	1	112	7

TABLE 5

OCCURRENCE AND DISTRIBUTION DATA - FROG TISSUE  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT

Chemical	Minimum Concentration <sup>(1)</sup>	Maximum Concentration <sup>(1)</sup>	Sample of Maximum Concentration	Average of All Samples <sup>(2)</sup>	Average of Positive Detections <sup>(3)</sup>	Frequency of Detection
<b>Inorganics (mg/kg)</b>						
ALUMINUM	7.5	470	POND 1A MUSCLE	106.5	106.5	5/5
BORON	420	510 J	POND 1B MUSCLE	470	470	5/5
CALCIUM	170 J	3000	POND 1A MUSCLE	784	784	5/5
CHROMIUM	1.2	1.2	POND 1A MUSCLE	0.63	1.2	1/5
COPPER	1	2.1	POND 1A MUSCLE	1.0	1.4	3/5
CYANIDE	4.1	56	90 MBO 18 FROG CONTROL-TI	17.1	20.7	4/5
IRON	13	44 J	POND 1A MUSCLE	22	22	5/5
LEAD	0.66	0.78	POND 1D MUSCLE/BONE	0.41	0.72	2/5
MAGNESIUM	120	270	POND 1A MUSCLE	214	214	5/5
MANGANESE	0.58	3.8 J	POND 1C MUSCLE	2.1	2.1	5/5
POTASSIUM	3000 J	3300 J	POND 1C MUSCLE	3080	3080	5/5
SELENIUM	0.4	0.88	POND 1D MUSCLE/BONE	0.63	0.63	5/5
SODIUM	680 J	1000 J	POND 1A MUSCLE	864	864	5/5
ZINC	6.4 J	11 J	POND 1A MUSCLE	8.0	8.0	5/5
<b>Miscellaneous Parameters (%)</b>						
LIPIDS	0	7.5	90 MBO 18 FROG CONTROL-LI	3.8	3.8	2/2
LIPIDS-LIVER	0	112	POND 1D LIVER	38.7	38.7	4/4
LIPIDS-MUSCLE	1	7	POND 1D MUSCLE/BONE	3	3	4/4

**Footnotes**

- 1 - Sample and duplicate are considered as two separate samples when determining the minimum and maximum concentrations detected concentrations and as one sample when determining the frequency of detection.
- 2 - Average of all analytical results are calculated using half of the detection limit for nondetects.
- 3 - Average of positive analytical results only.

TABLE 6

OCCURRENCE AND DISTRIBUTION DATA - BIRD TISSUE  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT

Chemical	Minimum Concentration <sup>(1)</sup>	Maximum Concentration <sup>(1)</sup>	Sample of Maximum Concentration	Average of All Samples <sup>(2)</sup>	Average of Positive Detections <sup>(3)</sup>	Frequency of Detection
<b>Inorganics (mg/kg)</b>						
ALUMINUM	6.5	44	90 MBO 07	12.9	15.9	13/17
ANTIMONY	5.8	23	90 MBO 13	5.2	14.0	4/17
BARIUM	3.1	4	90 MBO 02, 90 MBO 13	1.9	3.7	3/17
BERYLLIUM	0.35	0.4	90 MBO 07	0.13	0.38	2/17
BORON	590 J	820 J	90 MBO 07	704	704	17/17
CADMIUM	2	2.2	90 MBO 07	0.5	2.1	2/17
CALCIUM	1680	13600	90 MBO 15	6282	6282	17/17
CHROMIUM	1	5.9	90 MBO 07	1.1	3.3	4/17
COBALT	3.8	4.5	90 MBO 07	0.9	4.2	2/17
COPPER	3.1	11	90 MBO 05, 90 MBO 07	5.8	6.2	16/17
IRON	65 J	430 J	90 MBO 06	125	125	17/17
LEAD	0.41	2.3	90 MBO 02	0.81	1.00	13/17
MAGNESIUM	210	430	90 MBO 02, 90 MBO 15	315	315	17/17
MANGANESE	0.9	2.6	90 MBO 13	1.8	1.8	17/17
MERCURY	0.083 J	0.083 J	90 MBO 14	0.045	0.083	1/17
NICKEL	1.5	8	90 MBO 07	1.6	4.5	4/17
POTASSIUM	2700	4200	90 MBO 13	3235	3235	17/17
SELENIUM	0.19	1.1	90 MBO 01	0.58	0.61	15/17
SILVER	7.5	7.5	90 MBO 13	1.7	7.5	1/7
SODIUM	1100	1500	90 MBO 13	1206	1206	17/17
VANADIUM	5.7	6.3	90 MBO 07	2.4	6.0	2/17
ZINC	14 J	30 J	90 MBO 02	21	21	17/17
<b>Miscellaneous Parameters (%)</b>						
LIPIDS	12.7	18	90 MBO 11, 90 MBO 15, 90 MBO 16	15.8	15.8	17/17

**Footnotes**

- 1 - Sample and duplicate are considered as two separate samples when determining the minimum and maximum concentrations detected concentrations and as one sample when determining the frequency of detection.
- 2 - Average of all analytical results are calculated using half of the detection limit for nondetects
- 3 - Average of positive analytical results only.

**APPENDIX C – ATTACHMENT 5**  
**FOOD CHAIN MODEL CALCULATION SHEETS**

**CHEMICAL CONCENTRATIONS IN SURFACE SOIL, SURFACE WATER, AND TISSUE FOR TERRESTRIAL FOOD CHAIN MODELS  
SITE 2B - AREA A WETLAND  
NSB-NLON, GROTON, CONNECTICUT**

Chemical	Surface Soil Concentrations (mg/kg)		Surface Water Concentrations (mg/L)		Earthworm Bioaccumulation Factors <sup>(2)</sup>		Earthworm Concentrations (mg/kg)		Plant Bioaccumulation Factors <sup>(2)</sup>		Plant Concentrations (mg/kg)	
	Maximum Detection	Average <sup>(1)</sup>	Maximum Detection	Average <sup>(1)</sup>	Conservative	Average	Conservative	Average	Conservative	Average	Conservative	Average
<b>Semivolatile Organics</b>												
ACENAPHTHENE	2.70E-01	2.70E-01			0.669	0.256	1.81E-01	6.91E-02	Regression or BAF from Eco SSL		1.18E-02	1.18E-02
ACENAPHTHYLENE	1.20E-01	8.53E-02			0.669	0.256	8.03E-02	2.18E-02	Regression or BAF from Eco SSL		5.95E-02	4.55E-02
ANTHRACENE	7.90E-02	6.55E-02			0.669	0.256	5.29E-02	1.68E-02	Regression or BAF from Eco SSL		5.16E-02	4.46E-02
BENZO(A)ANTHRACENE	4.00E-01	2.22E-01			0.669	0.256	2.68E-01	5.69E-02	Regression or BAF from Eco SSL		3.87E-02	2.73E-02
BENZO(A)PYRENE	3.90E-01	2.91E-01			0.669	0.256	2.61E-01	7.44E-02	Regression or BAF from Eco SSL		5.08E-02	3.81E-02
BENZO(B)FLUORANTHENE	5.50E-01	3.22E-01			0.669	0.256	3.68E-01	8.25E-02	Regression or BAF from Eco SSL		1.71E-01	9.99E-02
BENZO(G,H,I)PERYLENE	2.60E-01	1.55E-01			0.669	0.256	1.74E-01	3.96E-02	Regression or BAF from Eco SSL		8.01E-02	4.33E-02
BENZO(K)FLUORANTHENE	3.90E-01	2.71E-01			0.669	0.256	2.61E-01	6.94E-02	Regression or BAF from Eco SSL		5.14E-02	3.76E-02
CHRYSENE	6.00E-01	3.27E-01			0.669	0.256	4.01E-01	8.36E-02	Regression or BAF from Eco SSL		4.92E-02	3.43E-02
DIBENZO(A,H)ANTHRACENE	3.20E-02	3.20E-02			0.669	0.256	2.14E-02	8.19E-03	Regression or BAF from Eco SSL		4.16E-03	4.16E-03
FLUORANTHENE	8.90E-01	3.42E-01			0.669	0.256	5.95E-01	8.75E-02	Regression or BAF from Eco SSL		4.45E-01	1.71E-01
INDENO(1,2,3-CD)PYRENE	2.70E-01	2.05E-01			0.669	0.256	1.81E-01	5.25E-02	Regression or BAF from Eco SSL		2.97E-02	2.26E-02
PHENANTHRENE	3.40E-01	2.69E-01			0.669	0.256	2.27E-01	6.90E-02	Regression or BAF from Eco SSL		4.34E-01	3.75E-01
PYRENE	8.10E-01	3.54E-01			0.669	0.256	5.42E-01	9.05E-02	Regression or BAF from Eco SSL		5.83E-01	2.55E-01
<b>Pesticides/PCBs</b>												
4,4'-DDD	6.90E-02	2.55E-02			Regression or BAF from Eco SSL		4.95E-01	2.47E-01	Regression or BAF from Eco SSL		1.09E-02	5.13E-03
4,4'-DDE	1.00E-02	7.60E-03			Regression or BAF from Eco SSL		2.07E-01	1.62E-01	Regression or BAF from Eco SSL		2.54E-03	2.06E-03
4,4'-DDT	2.80E-02	1.39E-02			Regression or BAF from Eco SSL		3.75E-01	2.04E-01	Regression or BAF from Eco SSL		5.50E-03	3.25E-03
ALPHA-CHLORDANE	3.00E-03	3.00E-03			5	5	1.50E-02	1.50E-02	0.025	0.025	7.50E-05	7.50E-05
AROCLOR-1260	3.70E-01	1.36E-01			15.909	6.667	5.89E+00	9.09E-01	0.0029	0.0029	1.07E-03	3.95E-04
GAMMA-CHLORDANE	2.20E-03	2.20E-03			5	5	1.10E-02	1.10E-02	0.025	0.025	5.50E-05	5.50E-05
<b>Inorganics</b>												
CADMIUM	7.20E+00	3.76E+00	1.26E-01	4.01E-02	Regression or BAF from Eco SSL		3.98E+01	2.37E+01	Regression or BAF from Eco SSL		1.83E+00	1.28E+00
CHROMIUM	1.02E+02	5.85E+01	6.80E-03	6.80E-03	Regression or BAF from Eco SSL		3.12E+01	1.79E+01	Regression or BAF from Eco SSL		4.18E+00	2.40E+00
COPPER	6.41E+01	3.60E+01	2.93E-02	1.35E-02	Regression or BAF from Eco SSL		3.30E+01	1.85E+01	Regression or BAF from Eco SSL		1.00E+01	8.01E+00
LEAD	1.28E+02	4.24E+01	7.80E-03	3.82E-03	Regression or BAF from Eco SSL		4.03E+01	1.65E+01	Regression or BAF from Eco SSL		4.03E+00	2.17E+00
MERCURY	6.90E-01	2.47E-01	2.10E-04	2.10E-04	Regression - Sample et al., (1998)		9.54E-01	6.75E-01	5	0.652	3.45E+00	1.61E-01
SELENIUM	2.40E+00	9.20E-01			Regression or BAF from Eco SSL		1.76E+00	8.73E-01	Regression or BAF from Eco SSL		1.34E+00	4.63E-01
SILVER	4.50E+00	1.52E+00			Regression or BAF from Eco SSL		9.20E+00	3.10E+00	Regression or BAF from Eco SSL		6.30E-02	2.12E-02
VANADIUM	7.50E+01	5.08E+01	4.30E-03	4.30E-03	Regression or BAF from Eco SSL		3.15E+00	2.13E+00	Regression or BAF from Eco SSL		3.64E-01	2.46E-01
ZINC	1.25E+02	7.01E+01	3.34E-01	1.33E-01	Regression or BAF from Eco SSL		4.17E+02	3.45E+02	Regression or BAF from Eco SSL		7.01E+01	5.09E+01

(1) - Average concentration is average of all samples using one-half of the detection limit for non-detects, unless the average concentration is greater than the maximum concentration. In that case, the average of positive detections is used.

(2) - Bioaccumulation factors are presented in Attachment 2.

CHEMICAL CONCENTRATIONS IN SEDIMENT, SURFACE WATER, AND TISSUE  
SITE 2B - AREA A WETLAND  
NSB-NLON, GROTON, CONNECTICUT

Chemical	Sediment Concentrations (mg/kg)		Surface Water Concentrations (mg/L)		Invertebrate Bioaccumulation Factors <sup>(2)</sup>		Invertebrate Concentrations (mg/kg)	
	Maximum Detection	Average <sup>(1)</sup>	Maximum Detection	Average <sup>(1)</sup>	Conservative	Average	Conservative	Average
<b>Semivolatile Organics</b>								
1,4-DICHLOROBENZENE	4.20E-02	4.20E-02			1	1	9.44E-02	9.44E-02
2-METHYLNAPHTHALENE	5.50E-02	4.88E-02			1	1	1.24E-01	1.10E-01
ACENAPHTHENE	3.80E-01	1.22E-01			0.29	0.29	2.48E-01	7.96E-02
ACENAPHTHYLENE	3.90E-01	1.37E-01			0.29	0.29	2.54E-01	8.90E-02
ANTHRACENE	2.40E+00	6.35E-01			0.29	0.29	1.56E+00	4.14E-01
BENZO(A)ANTHRACENE	2.70E+01	1.58E+00			0.29	0.29	1.76E+01	1.03E+00
BENZO(A)PYRENE	3.50E+01	1.90E+00			0.29	0.29	2.28E+01	1.24E+00
BENZO(B)FLUORANTHENE	5.50E+01	2.41E+00			0.29	0.29	3.59E+01	1.57E+00
BENZO(G,H,I)PERYLENE	2.30E+01	1.43E+00			0.29	0.29	1.50E+01	9.30E-01
BENZO(K)FLUORANTHENE	4.50E+01	2.20E+00			0.29	0.29	2.93E+01	1.43E+00
CHRYSENE	4.20E+01	2.13E+00			0.29	0.29	2.74E+01	1.39E+00
DIBENZO(A,H)ANTHRACENE	3.10E-01	1.25E-01			0.29	0.29	2.02E-01	8.15E-02
DIBENZOFURAN	1.00E+00	5.99E-01			1	1	2.25E+00	1.35E+00
FLUORANTHENE	8.00E+01	3.41E+00			0.29	0.29	5.22E+01	2.22E+00
FLUORENE	1.00E+00	5.96E-01			0.29	0.29	6.52E-01	3.88E-01
INDENO(1,2,3-CD)PYRENE	2.30E+01	1.40E+00			0.29	0.29	1.50E+01	9.10E-01
NAPHTHALENE	7.70E-02	6.78E-02			0.29	0.29	5.02E-02	4.42E-02
PENTACHLOROPHENOL	2.40E-01	2.40E-01			0	0	0.00E+00	0.00E+00
PHENANTHRENE	3.60E+01	1.84E+00			0.29	0.29	2.35E+01	1.20E+00
PYRENE	4.20E+01	2.23E+00			0.29	0.29	2.74E+01	1.45E+00
<b>Pesticides/PCBs</b>								
4,4'-DDD	4.80E+00	2.39E-01			0.28	0.28	3.02E+00	1.51E-01
4,4'-DDE	7.20E-01	4.25E-02			7.7	7.7	1.25E+01	7.36E-01
4,4'-DDT	2.90E+00	1.46E-01			1.67	1.67	1.09E+01	5.48E-01
ALDRIN	3.20E-03	3.20E-03			1.8	1.8	1.29E-02	1.29E-02
ALPHA-CHLORDANE	2.90E-02	2.45E-02			4.77	4.77	3.11E-01	2.62E-01
AROCOLOR-1260	1.50E+00	1.81E-01			64.122	36.215	2.16E+02	1.47E+01
BETA-BHC	2.70E-03	2.70E-03			1.8	1.8	1.09E-02	1.09E-02
DELTA-BHC	4.20E-03	4.20E-03			1.8	1.8	1.70E-02	1.70E-02
DIELDRIN	2.60E-02	1.17E-02			1.8	1.8	1.05E-01	4.74E-02
ENDOSULFAN I	1.10E-02	4.63E-03			1.8	1.8	4.45E-02	1.87E-02
ENDOSULFAN II	3.10E-02	9.58E-03			1.8	1.8	1.25E-01	3.88E-02
ENDOSULFAN SULFATE	1.40E-02	8.65E-03			1.8	1.8	5.67E-02	3.50E-02
ENDRIN	1.60E-02	9.02E-03			1.8	1.8	6.47E-02	3.65E-02
ENDRIN ALDEHYDE	1.60E-02	6.22E-03			1.8	1.8	6.47E-02	2.52E-02
ENDRIN KETONE	2.00E-02	9.15E-03			1.8	1.8	8.09E-02	3.70E-02
GAMMA-BHC (LINDANE)	3.50E-03	3.50E-03			1.8	1.8	1.42E-02	1.42E-02
GAMMA-CHLORDANE	2.30E-02	8.79E-03			2.22	2.22	1.15E-01	4.39E-02
HEPTACHLOR	4.50E-03	4.49E-03			1.8	1.8	1.82E-02	1.82E-02
HEPTACHLOR EPOXIDE	4.50E-03	3.53E-03			1.8	1.8	1.82E-02	1.43E-02
METHOXYCHLOR	3.80E-02	3.80E-02			1.8	1.8	1.54E-01	1.54E-01
<b>Inorganics</b>								
ARSENIC	1.41E+01	6.55E+00	2.90E-03	2.83E-03	0.69	0.143	9.73E+00	9.36E-01
CADMIUM	6.10E+00	1.66E+00	1.26E-01	4.01E-02	7.99	0.6	4.87E+01	9.96E-01
CHROMIUM	9.68E+01	4.39E+01	6.80E-03	6.80E-03	0.468	0.1	4.53E+01	4.39E+00
COPPER	1.73E+02	5.36E+01	2.93E-02	1.35E-02	5.25	1.556	9.08E+02	8.34E+01
LEAD	2.41E+02	5.89E+01	7.80E-03	3.82E-03	0.607	0.071	1.46E+02	4.18E+00
MERCURY	1.20E+00	2.95E-01	2.10E-04	2.10E-04	2.868	1.136	3.44E+00	3.36E-01
NICKEL	6.15E+01	2.02E+01	8.47E-02	4.58E-02	2.32	0.486	1.43E+02	9.83E+00
SELENIUM	6.80E+00	1.18E+00			1	1	6.80E+00	1.18E+00
SILVER	9.60E-01	6.15E-01			1	1	9.60E-01	6.15E-01
ZINC	7.02E+02	1.27E+02	3.34E-01	1.33E-01	7.527	1.936	5.28E+03	2.45E+02

(1) - Average concentration is average of all samples using one-half of the detection limit for non-detects, unless the average concentration is greater than the maximum concentration. In that case, the average of positive detections is used.

(2) - Bioaccumulation factors are presented in Attachment 2.

Percent TOC 4.20E+00  
Percent Lipids 9.44E+00

(Percent lipids were calculated by averaging dry-weight % lipid values for Freshwater crustacea, molluscs, and worms in Attachment 2)

MEADOW VOLE - CONSERVATIVE INPUTS  
 TERRESTRIAL WILDLIFE MODEL ECOLOGICAL EFFECTS QUOTIENT CALCULATION  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT

Chemical	Max Soil Conc. (mg/kg)	Max SW Conc. (mg/L)	Vegetation Conc. (mg/kg)	Dose (mg/kg/d) from:			Total Dose (mg/kg/d)	NOAEL (mg/kg/d)	LOAEL (mg/kg/d)	Hazard Quotients	
				Soil	Surface Water	Veget.				NOAEL	LOAEL
<b>Semivolatile Organics</b>											
ACENAPHTHENE	2.70E-01	0.00E+00	1.18E-02	1.91E-03	0.00E+00	2.60E-03	4.51E-03	1.75E+01	3.50E+01	2.6E-04	1.3E-04
ACENAPHTHYLENE	1.20E-01	0.00E+00	5.95E-02	8.48E-04	0.00E+00	1.32E-02	1.40E-02	7.00E+01	7.00E+02	2.0E-04	2.0E-05
ANTHRACENE	7.90E-02	0.00E+00	5.16E-02	5.59E-04	0.00E+00	1.14E-02	1.20E-02	1.00E+02	1.00E+03	1.2E-04	1.2E-05
BENZO(A)ANTHRACENE	4.00E-01	0.00E+00	3.87E-02	2.83E-03	0.00E+00	8.55E-03	1.14E-02	1.70E-01	1.70E+00	6.7E-02	6.7E-03
BENZO(A)PYRENE	3.90E-01	0.00E+00	5.08E-02	2.76E-03	0.00E+00	1.12E-02	1.40E-02	1.00E+00	1.00E+01	1.4E-02	1.4E-03
BENZO(B)FLUORANTHENE	5.50E-01	0.00E+00	1.71E-01	3.89E-03	0.00E+00	3.77E-02	4.16E-02	4.00E+00	4.00E+01	1.0E-02	1.0E-03
BENZO(G,H,I)PERYLENE	2.60E-01	0.00E+00	8.01E-02	1.84E-03	0.00E+00	1.77E-02	1.95E-02	7.20E+00	7.20E+01	2.7E-03	2.7E-04
BENZO(K)FLUORANTHENE	3.90E-01	0.00E+00	5.14E-02	2.76E-03	0.00E+00	1.14E-02	1.41E-02	7.20E+00	7.20E+01	2.0E-03	2.0E-04
CHRYSENE	6.00E-01	0.00E+00	4.92E-02	4.24E-03	0.00E+00	1.09E-02	1.51E-02	1.70E-01	1.70E+00	8.9E-02	8.9E-03
DIBENZO(A,H)ANTHRACENE	3.20E-02	0.00E+00	4.16E-03	2.26E-04	0.00E+00	9.19E-04	1.15E-03	1.33E+00	1.33E+01	8.6E-04	8.6E-05
FLUORANTHENE	8.90E-01	0.00E+00	4.45E-01	6.29E-03	0.00E+00	9.83E-02	1.05E-01	1.25E+01	2.50E+01	8.4E-03	4.2E-03
INDENO(1,2,3-CD)PYRENE	2.70E-01	0.00E+00	2.97E-02	1.91E-03	0.00E+00	6.56E-03	8.47E-03	7.20E+00	7.20E+01	1.2E-03	1.2E-04
PHENANTHRENE	3.40E-01	0.00E+00	4.34E-01	2.40E-03	0.00E+00	9.58E-02	9.82E-02	1.00E+00	1.00E+01	9.8E-02	9.8E-03
PYRENE	8.10E-01	0.00E+00	5.83E-01	5.73E-03	0.00E+00	1.29E-01	1.35E-01	7.50E+00	1.25E+01	1.8E-02	1.1E-02
<b>Pesticides/PCBs</b>											
4,4'-DDD	6.90E-02	0.00E+00	1.09E-02	4.88E-04	0.00E+00	2.40E-03	2.89E-03	1.47E-01	2.74E-01	2.0E-02	1.1E-02
4,4'-DDE	1.00E-02	0.00E+00	2.54E-03	7.07E-05	0.00E+00	5.61E-04	6.31E-04	1.47E-01	2.74E-01	4.3E-03	2.3E-03
4,4'-DDT	2.80E-02	0.00E+00	5.50E-03	1.98E-04	0.00E+00	1.22E-03	1.41E-03	1.47E-01	2.74E-01	9.6E-03	5.2E-03
ALPHA-CHLORDANE	3.00E-03	0.00E+00	7.50E-05	2.12E-05	0.00E+00	1.66E-05	3.78E-05	4.58E+00	9.16E+00	8.2E-06	4.1E-06
AROCLOP-1260	3.70E-01	0.00E+00	1.07E-03	2.62E-03	0.00E+00	2.37E-04	2.85E-03	6.80E-02	6.80E-01	4.2E-02	4.2E-03
GAMMA-CHLORDANE	2.20E-03	0.00E+00	5.50E-05	1.56E-05	0.00E+00	1.22E-05	2.77E-05	4.58E+00	9.16E+00	6.0E-06	3.0E-06
<b>Inorganics</b>											
ARSENIC	1.51E+01	2.90E-03	5.67E-01	1.07E-01	1.28E-03	1.25E-01	2.33E-01	2.47E+00	4.55E+00	9.4E-02	5.1E-02
CADMIUM	7.20E+00	1.26E-01	1.83E+00	5.09E-02	5.57E-02	4.04E-01	5.10E-01	7.70E-01	6.90E+00	6.6E-01	7.4E-02
CHROMIUM	1.02E+02	6.80E-03	4.18E+00	7.21E-01	3.01E-03	9.24E-01	1.65E+00	2.40E+00	5.82E+01	6.9E-01	2.8E-02
COPPER	6.41E+01	2.93E-02	1.00E+01	4.53E-01	1.29E-02	2.22E+00	2.69E+00	5.82E+00	8.14E+01	4.6E-01	3.3E-02
LEAD	1.28E+02	7.80E-03	4.03E+00	9.05E-01	3.45E-03	8.91E-01	1.80E+00	4.70E+00	1.86E+02	3.8E-01	9.7E-03
MERCURY	6.90E-01	2.10E-04	3.45E+00	4.88E-03	9.28E-05	7.62E-01	7.67E-01	3.20E-02	1.60E-01	2.4E+01	4.6E+00
NICKEL	2.69E+01	8.47E-02	1.27E+00	1.90E-01	3.74E-02	2.81E-01	5.08E-01	1.70E+00	1.48E+01	3.0E-01	3.4E-02
SELENIUM	2.40E+00	0.00E+00	1.34E+00	1.70E-02	0.00E+00	2.95E-01	3.12E-01	2.00E-01	3.30E-01	1.6E+00	9.5E-01
SILVER	4.50E+00	0.00E+00	6.30E-02	3.18E-02	0.00E+00	1.39E-02	4.57E-02	6.02E+00	1.19E+02	7.6E-03	3.9E-04
ZINC	1.25E+02	3.34E-01	7.01E+01	8.84E-01	1.48E-01	1.55E+01	1.65E+01	1.60E+02	3.20E+02	1.0E-01	5.2E-02

Cells are shaded if the value is greater than 1.0

Body Weight = (BW) 1.70E-02 kg  
 Food Ingestion Rate = (If) 3.76E-03 kg/day  
 Water Ingestion Rate = (Iw) 7.51E-03 L/day  
 Soil Ingestion Rate = (Is) 1.20E-04 kg/day  
 Home Range = (HR) Assume 100% on site  
 Contaminated Area = (CA) Assume equal to home range

Dose (soil) = (Cs \* Is)(H)/BW  
 Dose (vegetation) = (Cv \* If)(H)/BW  
 Dose (water) = (Cw \* Iw)(H)/BW  
 Cv = Contaminant concentration in vegetation  
 Cs = Contaminant concentration in soil  
 Cw = Contaminant concentration in water  
 Total Dose = Dose (soil) + Dose (vegetation) + Dose (water)  
 H=HR/CA (Assume = to 1)

Conc = Concentration  
 LOAEL = Lowest Observed Adverse Effects Concentration  
 NOAEL = No Observed Adverse Effects Concentration  
 SW = Surface Water

MEADOW VOLE - AVERAGE INPUTS  
 TERRESTRIAL WILDLIFE MODEL ECOLOGICAL EFFECTS QUOTIENT CALCULATION  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT

Chemical	Avg Soil Conc. (mg/kg)	Avg SW Conc. (mg/L)	Vegetation Conc. (mg/kg)	Dose (mg/kg/d) from:			Total Dose (mg/kg/d)	NOAEL (mg/kg/d)	LOAEL (mg/kg/d)	Hazard Quotients	
				Soil	Surface Water	Veget.				NOAEL	LOAEL
<b>Semivolatile Organics</b>											
ACENAPHTHENE	2.70E-01	0.00E+00	1.18E-02	3.16E-04	0.00E+00	1.15E-03	1.46E-03	1.75E+01	3.50E+01	8.4E-05	4.2E-05
ACENAPHTHYLENE	8.53E-02	0.00E+00	4.55E-02	9.98E-05	0.00E+00	4.43E-03	4.53E-03	7.00E+01	7.00E+02	6.5E-05	6.5E-06
ANTHRACENE	6.55E-02	0.00E+00	4.46E-02	7.66E-05	0.00E+00	4.34E-03	4.42E-03	1.00E+02	1.00E+03	4.4E-05	4.4E-06
BENZO(A)ANTHRACENE	2.22E-01	0.00E+00	2.73E-02	2.60E-04	0.00E+00	2.66E-03	2.92E-03	1.70E-01	1.70E+00	1.7E-02	1.7E-03
BENZO(A)PYRENE	2.91E-01	0.00E+00	3.81E-02	3.40E-04	0.00E+00	3.72E-03	4.06E-03	1.00E+00	1.00E+01	4.1E-03	4.1E-04
BENZO(B)FLUORANTHENE	3.22E-01	0.00E+00	9.99E-02	3.77E-04	0.00E+00	9.73E-03	1.01E-02	4.00E+00	4.00E+01	2.5E-03	2.5E-04
BENZO(G,H,I)PERYLENE	1.55E-01	0.00E+00	4.33E-02	1.81E-04	0.00E+00	4.22E-03	4.40E-03	7.20E+00	7.20E+01	6.1E-04	6.1E-05
BENZO(K)FLUORANTHENE	2.71E-01	0.00E+00	3.76E-02	3.17E-04	0.00E+00	3.67E-03	3.99E-03	7.20E+00	7.20E+01	5.5E-04	5.5E-05
CHRYSENE	3.27E-01	0.00E+00	3.43E-02	3.82E-04	0.00E+00	3.34E-03	3.72E-03	1.70E-01	1.70E+00	2.2E-02	2.2E-03
DIBENZO(A,H)ANTHRACENE	3.20E-02	0.00E+00	4.16E-03	3.74E-05	0.00E+00	4.05E-04	4.43E-04	1.33E+00	1.33E+01	3.3E-04	3.3E-05
FLUORANTHENE	3.42E-01	0.00E+00	1.71E-01	4.00E-04	0.00E+00	1.66E-02	1.70E-02	1.25E+01	2.50E+01	1.4E-03	6.8E-04
INDENO(1,2,3-CD)PYRENE	2.05E-01	0.00E+00	2.26E-02	2.40E-04	0.00E+00	2.20E-03	2.44E-03	7.20E+00	7.20E+01	3.4E-04	3.4E-05
PHENANTHRENE	2.69E-01	0.00E+00	3.75E-01	3.15E-04	0.00E+00	3.66E-02	3.69E-02	1.00E+00	1.00E+01	3.7E-02	3.7E-03
PYRENE	3.54E-01	0.00E+00	2.55E-01	4.14E-04	0.00E+00	2.48E-02	2.52E-02	7.50E+00	1.25E+01	3.4E-03	2.0E-03
<b>Pesticides/PCBs</b>											
4,4'-DDD	2.55E-02	0.00E+00	5.13E-03	2.98E-05	0.00E+00	5.00E-04	5.30E-04	1.47E-01	2.74E-01	3.6E-03	1.9E-03
4,4'-DDE	7.60E-03	0.00E+00	2.06E-03	8.89E-06	0.00E+00	2.01E-04	2.10E-04	1.47E-01	2.74E-01	1.4E-03	7.7E-04
4,4'-DDT	1.39E-02	0.00E+00	3.25E-03	1.63E-05	0.00E+00	3.17E-04	3.33E-04	1.47E-01	2.74E-01	2.3E-03	1.2E-03
ALPHA-CHLORDANE	3.00E-03	0.00E+00	7.50E-05	3.51E-06	0.00E+00	7.31E-06	1.08E-05	4.58E+00	9.16E+00	2.4E-06	1.2E-06
AROCLOR-1260	1.36E-01	0.00E+00	3.95E-04	1.59E-04	0.00E+00	3.85E-05	1.98E-04	6.80E-02	6.80E-01	2.9E-03	2.9E-04
GAMMA-CHLORDANE	2.20E-03	0.00E+00	5.50E-05	2.57E-06	0.00E+00	5.36E-06	7.93E-06	4.58E+00	9.16E+00	1.7E-06	8.7E-07
<b>Inorganics</b>											
ARSENIC	7.16E+00	2.83E-03	2.69E-01	8.37E-03	4.95E-04	2.62E-02	3.50E-02	2.47E+00	4.55E+00	1.4E-02	7.7E-03
CADMIUM	3.76E+00	4.01E-02	1.28E+00	4.39E-03	7.02E-03	1.25E-01	1.36E-01	7.70E-01	6.90E+00	1.8E-01	2.0E-02
CHROMIUM	5.85E+01	6.80E-03	2.40E+00	6.84E-02	1.19E-03	2.34E-01	3.03E-01	2.40E+00	5.82E+00	1.3E-01	5.2E-03
COPPER	3.60E+01	1.35E-02	8.01E+00	4.21E-02	2.36E-03	7.80E-01	8.24E-01	5.82E+00	8.14E+01	1.4E-01	1.0E-02
LEAD	4.24E+01	3.82E-03	2.17E+00	4.95E-02	6.67E-04	2.11E-01	2.61E-01	4.70E+00	1.86E+02	5.6E-02	1.4E-03
MERCURY	2.47E-01	2.10E-04	1.61E-01	2.89E-04	3.67E-05	1.57E-02	1.60E-02	3.20E-02	1.60E-01	5.0E-01	1.0E-01
NICKEL	2.13E+01	4.58E-02	1.07E+00	2.49E-02	8.01E-03	1.04E-01	1.37E-01	1.70E+00	1.48E+01	8.1E-02	9.3E-03
SELENIUM	9.20E-01	0.00E+00	4.63E-01	1.08E-03	0.00E+00	4.52E-02	4.62E-02	2.00E-01	3.30E-01	2.3E-01	1.4E-01
SILVER	1.52E+00	0.00E+00	2.12E-02	1.77E-03	0.00E+00	2.07E-03	3.84E-03	6.02E+00	1.19E+02	6.4E-04	3.2E-05
ZINC	7.01E+01	1.33E-01	5.09E+01	8.19E-02	2.32E-02	4.96E+00	5.06E+00	1.60E+02	3.20E+02	3.2E-02	1.6E-02

Body Weight = (BW) 3.58E-02 kg  
 Food Ingestion Rate = (If) 3.49E-03 kg/day  
 Water Ingestion Rate = (Iw) 6.26E-03 L/day  
 Soil Ingestion Rate = (Is) 4.19E-05 kg/day  
 Home Range = (HR) 6.59E-02 acres  
 Contaminated Area = (CA) Assume equal to home range

Dose (soil) = (Cs \* Is)(H)/BW  
 Dose (vegetation) = (Cv \* If)(H)/BW  
 Dose (water) = (Cw \* Iw)(H)/BW  
 Cv = Contaminant concentration in vegetation  
 Cs = Contaminant concentration in soil  
 Cw = Contaminant concentration in water  
 Total Dose = Dose (soil) + Dose (vegetation) + Dose (water)  
 H=HR/CA (Assume = to 1)

Conc = Concentration  
 LOAEL = Lowest Observed Adverse Effects Concentration  
 NOAEL = No Observed Adverse Effects Concentration  
 SW = Surface Water

BOBWHITE QUAIL - CONSERVATIVE INPUTS  
 TERRESTRIAL WILDLIFE MODEL ECOLOGICAL EFFECTS QUOTIENT CALCULATION  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT

Chemical	Max Soil Conc. (mg/kg)	Max SW Conc. (mg/L)	Vegetation Conc. (mg/kg)	Dose (mg/kg/d) from:			Total Dose (mg/kg/d)	NOAEL (mg/kg/d)	LOAEL (mg/kg/d)	Hazard Quotients	
				Soil	Surface Water	Veget.				NOAEL	LOAEL
<b>Semivolatile Organics</b>											
ACENAPHTHENE	2.70E-01	0.00E+00	1.18E-02	1.19E-03	0.00E+00	3.73E-04	1.56E-03	2.00E+00	2.00E+01	7.8E-04	7.8E-05
ACENAPHTHYLENE	1.20E-01	0.00E+00	5.95E-02	5.29E-04	0.00E+00	1.89E-03	2.42E-03	2.00E+00	2.00E+01	1.2E-03	1.2E-04
ANTHRACENE	7.90E-02	0.00E+00	5.16E-02	3.48E-04	0.00E+00	1.63E-03	1.98E-03	2.00E+00	2.00E+01	9.9E-04	9.9E-05
BENZO(A)ANTHRACENE	4.00E-01	0.00E+00	3.87E-02	1.76E-03	0.00E+00	1.23E-03	2.99E-03	2.00E+00	2.00E+01	1.5E-03	1.5E-04
BENZO(A)PYRENE	3.90E-01	0.00E+00	5.08E-02	1.72E-03	0.00E+00	1.61E-03	3.33E-03	2.00E+00	2.00E+01	1.7E-03	1.7E-04
BENZO(B)FLUORANTHENE	5.50E-01	0.00E+00	1.71E-01	2.42E-03	0.00E+00	5.40E-03	7.83E-03	2.00E+00	2.00E+01	3.9E-03	3.9E-04
BENZO(G,H,I)PERYLENE	2.60E-01	0.00E+00	8.01E-02	1.15E-03	0.00E+00	2.54E-03	3.68E-03	2.00E+00	2.00E+01	1.8E-03	1.8E-04
BENZO(K)FLUORANTHENE	3.90E-01	0.00E+00	5.14E-02	1.72E-03	0.00E+00	1.63E-03	3.35E-03	2.00E+00	2.00E+01	1.7E-03	1.7E-04
CHRYSENE	6.00E-01	0.00E+00	4.92E-02	2.64E-03	0.00E+00	1.56E-03	4.20E-03	2.00E+00	2.00E+01	2.1E-03	2.1E-04
DIBENZO(A,H)ANTHRACENE	3.20E-02	0.00E+00	4.16E-03	1.41E-04	0.00E+00	1.32E-04	2.73E-04	2.00E+00	2.00E+01	1.4E-04	1.4E-05
FLUORANTHENE	8.90E-01	0.00E+00	4.45E-01	3.92E-03	0.00E+00	1.41E-02	1.80E-02	2.00E+00	2.00E+01	9.0E-03	9.0E-04
INDENO(1,2,3-CD)PYRENE	2.70E-01	0.00E+00	2.97E-02	1.19E-03	0.00E+00	9.41E-04	2.13E-03	2.00E+00	2.00E+01	1.1E-03	1.1E-04
PHENANTHRENE	3.40E-01	0.00E+00	4.34E-01	1.50E-03	0.00E+00	1.37E-02	1.52E-02	2.00E+00	2.00E+01	7.6E-03	7.6E-04
PYRENE	8.10E-01	0.00E+00	5.83E-01	3.57E-03	0.00E+00	1.85E-02	2.20E-02	2.00E+00	2.00E+01	1.1E-02	1.1E-03
<b>Pesticides/PCBs</b>											
4,4'-DDD	6.90E-02	0.00E+00	1.09E-02	3.04E-04	0.00E+00	3.44E-04	6.48E-04	2.27E-01	2.81E-01	2.9E-03	2.3E-03
4,4'-DDE	1.00E-02	0.00E+00	2.54E-03	4.40E-05	0.00E+00	8.04E-05	1.24E-04	2.27E-01	2.81E-01	5.5E-04	4.4E-04
4,4'-DDT	2.80E-02	0.00E+00	5.50E-03	1.23E-04	0.00E+00	1.74E-04	2.98E-04	2.27E-01	2.81E-01	1.3E-03	1.1E-03
ALPHA-CHLORDANE	3.00E-03	0.00E+00	7.50E-05	1.32E-05	0.00E+00	2.38E-06	1.56E-05	2.14E+00	1.07E+01	7.3E-06	1.5E-06
AROCLOR-1260	3.70E-01	0.00E+00	1.07E-03	1.63E-03	0.00E+00	3.40E-05	1.66E-03	1.80E-01	1.80E+00	9.2E-03	9.2E-04
GAMMA-CHLORDANE	2.20E-03	0.00E+00	5.50E-05	9.69E-06	0.00E+00	1.74E-06	1.14E-05	2.14E+00	1.07E+01	5.3E-06	1.1E-06
<b>Inorganics</b>											
ARSENIC	1.51E+01	2.90E-03	5.67E-01	6.65E-02	4.29E-04	1.80E-02	8.49E-02	2.24E+00	4.51E+00	3.8E-02	1.9E-02
CADMIUM	7.20E+00	1.26E-01	1.83E+00	3.17E-02	1.86E-02	5.79E-02	1.08E-01	1.47E+00	6.35E+00	7.4E-02	1.7E-02
CHROMIUM	1.02E+02	6.80E-03	4.18E+00	4.49E-01	1.01E-03	1.33E-01	5.83E-01	2.66E+00	1.56E+01	2.2E-01	3.7E-02
COPPER	6.41E+01	2.93E-02	1.00E+01	2.82E-01	4.33E-03	3.18E-01	6.05E-01	4.05E+00	3.48E+01	1.5E-01	1.7E-02
LEAD	1.28E+02	7.80E-03	4.03E+00	5.64E-01	1.15E-03	1.28E-01	6.93E-01	1.63E+00	4.46E+01	4.2E-01	1.6E-02
MERCURY	6.90E-01	2.10E-04	3.45E+00	3.04E-03	3.10E-05	1.09E-01	1.12E-01	6.40E-03	6.40E-02	1.8E+01	1.8E+00
NICKEL	2.69E+01	8.47E-02	1.27E+00	1.18E-01	1.25E-02	4.03E-02	1.71E-01	6.71E+00	1.86E+01	2.6E-02	9.2E-03
SELENIUM	2.40E+00	0.00E+00	1.34E+00	1.06E-02	0.00E+00	4.23E-02	5.29E-02	4.00E-01	8.00E-01	1.3E-01	6.6E-02
SILVER	4.50E+00	0.00E+00	6.30E-02	1.98E-02	0.00E+00	2.00E-03	2.18E-02	2.02E+00	6.05E+01	1.1E-02	3.6E-04
ZINC	1.25E+02	3.34E-01	7.01E+01	5.51E-01	4.94E-02	2.22E+00	2.82E+00	1.45E+01	1.31E+02	1.9E-01	2.2E-02

Cells are shaded if the value is greater than 1.0

Body Weight = (BW)	1.54E-01	kg	Dose (soil) = (Cs * Is)(H)/BW	Conc = Concentration
Food Ingestion Rate = (If)	4.88E-03	kg/day	Dose (vegetation) = (Cv * If)(H)/BW	LOAEL = Lowest Observed Adverse Effects Concentration
Water Ingestion Rate = (Iw)	2.28E-02	L/day	Dose (water) = (Cw * Iw)(H)/BW	NOAEL = No Observed Adverse Effects Concentration
Soil Ingestion Rate = (Is)	6.78E-04	kg/day	Cv = Contaminant concentration in vegetation	SW = Surface Water
Home Range = (HR)	Assume 100% on site		Cs = Contaminant concentration in soil	
Contaminated Area = (CA)	Assume equal to home range		Cw = Contaminant concentration in water	
			Total Dose = Dose (soil) + Dose (vegetation) + Dose (water)	
			H=HR/CA (Assume = to 1)	

BOBWHITE QUAIL - AVERAGE INPUTS  
 TERRESTRIAL WILDLIFE MODEL ECOLOGICAL EFFECTS QUOTIENT CALCULATION  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT

Chemical	Avg Soil Conc. (mg/kg)	Avg SW Conc. (mg/L)	Vegetation Conc. (mg/kg)	Dose (mg/kg/d) from:			Total Dose (mg/kg/d)	NOAEL (mg/kg/d)	LOAEL (mg/kg/d)	Hazard Quotients	
				Soil	Surface Water	Veget.				NOAEL	LOAEL
<b>Semivolatile Organics</b>											
ACENAPHTHENE	2.70E-01	0.00E+00	1.18E-02	3.84E-04	0.00E+00	2.74E-04	6.58E-04	2.00E+00	2.00E+01	3.3E-04	3.3E-05
ACENAPHTHYLENE	8.53E-02	0.00E+00	4.55E-02	1.21E-04	0.00E+00	1.06E-03	1.18E-03	2.00E+00	2.00E+01	5.9E-04	5.9E-05
ANTHRACENE	6.55E-02	0.00E+00	4.46E-02	9.31E-05	0.00E+00	1.04E-03	1.13E-03	2.00E+00	2.00E+01	5.7E-04	5.7E-05
BENZO(A)ANTHRACENE	2.22E-01	0.00E+00	2.73E-02	3.16E-04	0.00E+00	6.36E-04	9.52E-04	2.00E+00	2.00E+01	4.8E-04	4.8E-05
BENZO(A)PYRENE	2.91E-01	0.00E+00	3.81E-02	4.13E-04	0.00E+00	8.89E-04	1.30E-03	2.00E+00	2.00E+01	6.5E-04	6.5E-05
BENZO(B)FLUORANTHENE	3.22E-01	0.00E+00	9.99E-02	4.58E-04	0.00E+00	2.33E-03	2.78E-03	2.00E+00	2.00E+01	1.4E-03	1.4E-04
BENZO(G,H,I)PERYLENE	1.55E-01	0.00E+00	4.33E-02	2.20E-04	0.00E+00	1.01E-03	1.23E-03	2.00E+00	2.00E+01	6.1E-04	6.1E-05
BENZO(K)FLUORANTHENE	2.71E-01	0.00E+00	3.76E-02	3.85E-04	0.00E+00	8.77E-04	1.26E-03	2.00E+00	2.00E+01	6.3E-04	6.3E-05
CHRYSENE	3.27E-01	0.00E+00	3.43E-02	4.64E-04	0.00E+00	7.99E-04	1.26E-03	2.00E+00	2.00E+01	6.3E-04	6.3E-05
DIBENZO(A,H)ANTHRACENE	3.20E-02	0.00E+00	4.16E-03	4.55E-05	0.00E+00	9.69E-05	1.42E-04	2.00E+00	2.00E+01	7.1E-05	7.1E-06
FLUORANTHENE	3.42E-01	0.00E+00	1.71E-01	4.86E-04	0.00E+00	3.98E-03	4.47E-03	2.00E+00	2.00E+01	2.2E-03	2.2E-04
INDENO(1,2,3-CD)PYRENE	2.05E-01	0.00E+00	2.26E-02	2.91E-04	0.00E+00	5.25E-04	8.17E-04	2.00E+00	2.00E+01	4.1E-04	4.1E-05
PHENANTHRENE	2.69E-01	0.00E+00	3.75E-01	3.83E-04	0.00E+00	8.74E-03	9.13E-03	2.00E+00	2.00E+01	4.6E-03	4.6E-04
PYRENE	3.54E-01	0.00E+00	2.55E-01	5.03E-04	0.00E+00	5.93E-03	6.44E-03	2.00E+00	2.00E+01	3.2E-03	3.2E-04
<b>Pesticides/PCBs</b>											
4,4'-DDD	2.55E-02	0.00E+00	5.13E-03	3.62E-05	0.00E+00	1.20E-04	1.56E-04	2.27E-01	2.81E-01	6.9E-04	5.5E-04
4,4'-DDE	7.60E-03	0.00E+00	2.06E-03	1.08E-05	0.00E+00	4.81E-05	5.89E-05	2.27E-01	2.81E-01	2.6E-04	2.1E-04
4,4'-DDT	1.39E-02	0.00E+00	3.25E-03	1.98E-05	0.00E+00	7.58E-05	9.56E-05	2.27E-01	2.81E-01	4.2E-04	3.4E-04
ALPHA-CHLORDANE	3.00E-03	0.00E+00	7.50E-05	4.26E-06	0.00E+00	1.75E-06	6.01E-06	2.14E+00	1.07E+01	2.8E-06	5.6E-07
AROCLOL-1260	1.36E-01	0.00E+00	3.95E-04	1.94E-04	0.00E+00	9.21E-06	2.03E-04	1.80E-01	1.80E+00	1.1E-03	1.1E-04
GAMMA-CHLORDANE	2.20E-03	0.00E+00	5.50E-05	3.13E-06	0.00E+00	1.28E-06	4.41E-06	2.14E+00	1.07E+01	2.1E-06	4.1E-07
<b>Inorganics</b>											
ARSENIC	7.16E+00	2.83E-03	2.69E-01	1.02E-02	3.12E-04	6.26E-03	1.68E-02	2.24E+00	4.51E+00	7.5E-03	3.7E-03
CADMIUM	3.76E+00	4.01E-02	1.28E+00	5.34E-03	4.41E-03	2.98E-02	3.96E-02	1.47E+00	6.35E+00	2.7E-02	6.2E-03
CHROMIUM	5.85E+01	6.80E-03	2.40E+00	8.31E-02	7.48E-04	5.59E-02	1.40E-01	2.66E+00	1.56E+01	5.3E-02	8.9E-03
COPPER	3.60E+01	1.35E-02	8.01E+00	5.12E-02	1.49E-03	1.87E-01	2.39E-01	4.05E+00	3.48E+01	5.9E-02	6.9E-03
LEAD	4.24E+01	3.82E-03	2.17E+00	6.02E-02	4.20E-04	5.05E-02	1.11E-01	1.63E+00	4.46E+01	6.8E-02	2.5E-03
MERCURY	2.47E-01	2.10E-04	1.61E-01	3.51E-04	2.31E-05	3.75E-03	4.13E-03	6.40E-03	6.40E-02	6.4E-01	6.4E-02
NICKEL	2.13E+01	4.58E-02	1.07E+00	3.03E-02	5.04E-03	2.49E-02	6.03E-02	6.71E+00	1.86E+01	9.0E-03	3.2E-03
SELENIUM	9.20E-01	0.00E+00	4.63E-01	1.31E-03	0.00E+00	1.08E-02	1.21E-02	4.00E-01	8.00E-01	3.0E-02	1.5E-02
SILVER	1.52E+00	0.00E+00	2.12E-02	2.15E-03	0.00E+00	4.94E-04	2.65E-03	2.02E+00	6.05E+01	1.3E-03	4.4E-05
ZINC	7.01E+01	1.33E-01	5.09E+01	9.96E-02	1.46E-02	1.19E+00	1.30E+00	1.45E+01	1.31E+02	9.0E-02	9.9E-03

Body Weight = (BW) 1.75E-01 kg  
 Food Ingestion Rate = (If) 4.08E-03 kg/day  
 Water Ingestion Rate = (Iw) 1.93E-02 L/day  
 Soil Ingestion Rate = (Is) 2.49E-04 kg/day  
 Home Range = (HR) 1.88E+01 acres  
 Contaminated Area = (CA) Assume equal to home range

Dose (soil) = (Cs \* Is)(H)/BW  
 Dose (vegetation) = (Cv \* If)(H)/BW  
 Dose (water) = (Cw \* Iw)(H)/BW  
 Cv = Contaminant concentration in vegetation  
 Cs = Contaminant concentration in soil  
 Cw = Contaminant concentration in water  
 Total Dose = Dose (soil) + Dose (vegetation) + Dose (water)  
 H=HR/CA (Assume = to 1)

Conc = Concentration  
 LOAEL = Lowest Observed Adverse Effects Concentration  
 NOAEL = No Observed Adverse Effects Concentration  
 SW = Surface Water

SHORT-TAILED SHREW - CONSERVATIVE INPUTS  
 TERRESTRIAL WILDLIFE MODEL ECOLOGICAL EFFECTS QUOTIENT CALCULATION  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT

Chemical	Max Soil Conc. (mg/kg)	Max SW Conc. (mg/L)	Invertebrate Conc. (mg/kg)	Dose (mg/kg/d) from:			Total Dose (mg/kg/d)	NOAEL (mg/kg/d)	LOAEL (mg/kg/d)	Hazard Quotients	
				Soil	Surface Water	Invert.				NOAEL	LOAEL
<b>Semivolatile Organics</b>											
ACENAPHTHENE	2.70E-01	0.00E+00	1.81E-01	8.64E-04	0.00E+00	1.93E-02	2.01E-02	1.75E+01	3.50E+01	1.2E-03	5.8E-04
ACENAPHTHYLENE	1.20E-01	0.00E+00	8.03E-02	3.84E-04	0.00E+00	8.57E-03	8.95E-03	7.00E+01	7.00E+02	1.3E-04	1.3E-05
ANTHRACENE	7.90E-02	0.00E+00	5.29E-02	2.53E-04	0.00E+00	5.64E-03	5.89E-03	1.00E+02	1.00E+03	5.9E-05	5.9E-06
BENZO(A)ANTHRACENE	4.00E-01	0.00E+00	2.68E-01	1.28E-03	0.00E+00	2.86E-02	2.98E-02	1.70E-01	1.70E+00	1.8E-01	1.8E-02
BENZO(A)PYRENE	3.90E-01	0.00E+00	2.61E-01	1.25E-03	0.00E+00	2.78E-02	2.91E-02	1.00E+00	1.00E+01	2.9E-02	2.9E-03
BENZO(B)FLUORANTHENE	5.50E-01	0.00E+00	3.68E-01	1.76E-03	0.00E+00	3.93E-02	4.10E-02	4.00E+00	4.00E+01	1.0E-02	1.0E-03
BENZO(G,H,I)PERYLENE	2.60E-01	0.00E+00	1.74E-01	8.32E-04	0.00E+00	1.86E-02	1.94E-02	7.20E+00	7.20E+01	2.7E-03	2.7E-04
BENZO(K)FLUORANTHENE	3.90E-01	0.00E+00	2.61E-01	1.25E-03	0.00E+00	2.78E-02	2.91E-02	7.20E+00	7.20E+01	4.0E-03	4.0E-04
CHRYSENE	6.00E-01	0.00E+00	4.01E-01	1.92E-03	0.00E+00	4.28E-02	4.47E-02	1.70E-01	1.70E+00	2.6E-01	2.6E-02
DIBENZO(A,H)ANTHRACENE	3.20E-02	0.00E+00	2.14E-02	1.02E-04	0.00E+00	2.28E-03	2.39E-03	1.33E+00	1.33E+01	1.8E-03	1.8E-04
FLUORANTHENE	8.90E-01	0.00E+00	5.95E-01	2.85E-03	0.00E+00	6.35E-02	6.64E-02	1.25E+01	2.50E+01	5.3E-03	2.7E-03
INDENO(1,2,3-CD)PYRENE	2.70E-01	0.00E+00	1.81E-01	8.64E-04	0.00E+00	1.93E-02	2.01E-02	7.20E+00	7.20E+01	2.8E-03	2.8E-04
PHENANTHRENE	3.40E-01	0.00E+00	2.27E-01	1.09E-03	0.00E+00	2.43E-02	2.54E-02	1.00E+00	1.00E+01	2.5E-02	2.5E-03
PYRENE	8.10E-01	0.00E+00	5.42E-01	2.59E-03	0.00E+00	5.78E-02	6.04E-02	7.50E+00	1.25E+01	8.1E-03	4.8E-03
<b>Pesticides/PCBs</b>											
4,4'-DDD	6.90E-02	0.00E+00	4.95E-01	2.21E-04	0.00E+00	5.28E-02	5.30E-02	1.47E-01	2.74E-01	3.6E-01	1.9E-01
4,4'-DDE	1.00E-02	0.00E+00	2.07E-01	3.20E-05	0.00E+00	2.20E-02	2.21E-02	1.47E-01	2.74E-01	1.5E-01	8.1E-02
4,4'-DDT	2.80E-02	0.00E+00	3.75E-01	8.96E-05	0.00E+00	4.00E-02	4.00E-02	1.47E-01	2.74E-01	2.7E-01	1.5E-01
ALPHA-CHLORDANE	3.00E-03	0.00E+00	1.50E-02	9.60E-06	0.00E+00	1.60E-03	1.61E-03	4.58E+00	9.16E+00	3.5E-04	1.8E-04
AROCOR-1260	3.70E-01	0.00E+00	5.89E+00	1.18E-03	0.00E+00	6.28E-01	6.29E-01	6.80E-02	6.80E-01	9.3E+00	9.3E-01
GAMMA-CHLORDANE	2.20E-03	0.00E+00	1.10E-02	7.04E-06	0.00E+00	1.17E-03	1.18E-03	4.58E+00	9.16E+00	2.6E-04	1.3E-04
<b>Inorganics</b>											
ARSENIC	1.51E+01	2.90E-03	1.64E+00	4.83E-02	8.27E-04	1.75E-01	2.24E-01	2.47E+00	4.55E+00	9.1E-02	4.9E-02
CADMIUM	7.20E+00	1.26E-01	3.98E+01	2.30E-02	3.60E-02	4.24E+00	4.30E+00	7.70E-01	6.90E+00	5.6E+00	6.2E-01
CHROMIUM	1.02E+02	6.80E-03	3.12E+01	3.26E-01	1.94E-03	3.33E+00	3.66E+00	2.40E+00	5.82E+01	1.5E+00	6.3E-02
COPPER	6.41E+01	2.93E-02	3.30E+01	2.05E-01	8.36E-03	3.52E+00	3.74E+00	5.82E+00	8.14E+01	6.4E-01	4.6E-02
LEAD	1.28E+02	7.80E-03	4.03E+01	4.10E-01	2.23E-03	4.30E+00	4.72E+00	4.70E+00	1.86E+02	1.0E+00	2.5E-02
MERCURY	6.90E-01	2.10E-04	9.54E-01	2.21E-03	5.99E-05	1.02E-01	1.04E-01	3.20E-02	1.60E-01	3.3E+00	6.5E-01
NICKEL	2.69E+01	8.47E-02	2.85E+01	8.61E-02	2.42E-02	3.04E+00	3.15E+00	1.70E+00	1.48E+01	1.9E+00	2.1E-01
SELENIUM	2.40E+00	0.00E+00	1.76E+00	7.68E-03	0.00E+00	1.88E-01	1.96E-01	2.00E-01	3.30E-01	9.8E-01	5.9E-01
SILVER	4.50E+00	0.00E+00	9.20E+00	1.44E-02	0.00E+00	9.82E-01	9.96E-01	6.02E+00	1.19E+02	1.7E-01	8.4E-03
ZINC	1.25E+02	3.34E-01	4.17E+02	4.00E-01	9.53E-02	4.45E+01	4.50E+01	1.60E+02	3.20E+02	2.8E-01	1.4E-01

Cells are shaded if the value is greater than 1.0

Body Weight = (BW) 1.50E-02 kg  
 Food Ingestion Rate = (If) 1.60E-03 kg/day  
 Water Ingestion Rate = (Iw) 4.28E-03 L/day  
 Soil Ingestion Rate = (Is) 4.80E-05 kg/day  
 Home Range = (HR) Assume 100% on site  
 Contaminated Area = (CA) Assume equal to home range

Dose (soil) = (Cs \* Is)(H)/BW  
 Dose (invertebrate) = (Ci \* If)(H)/BW  
 Dose (water) = (Cw \* Iw)(H)/BW  
 Ci = Contaminant concentration in invertebrate  
 Cs = Contaminant concentration in soil  
 Cw = Contaminant concentration in water  
 Total Dose = Dose (soil) + Dose (invertebrate) + Dose (water)  
 H=HR/CA (Assume = to 1)

Conc = Concentration  
 LOAEL = Lowest Observed Adverse Effects Concentration  
 NOAEL = No Observed Adverse Effects Concentration  
 SW = Surface Water

SHORT-TAILED SHREW - AVERAGE INPUTS  
 TERRESTRIAL WILDLIFE MODEL ECOLOGICAL EFFECTS QUOTIENT CALCULATION  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT

Chemical	Avg Soil Conc. (mg/kg)	Avg SW Conc. (mg/L)	Invertebrate Conc. (mg/kg)	Dose (mg/kg/d) from:			Total Dose (mg/kg/d)	NOAEL (mg/kg/d)	LOAEL (mg/kg/d)	Hazard Quotients	
				Soil	Surface Water	Invert.				NOAEL	LOAEL
<b>Semivolatile Organics</b>											
ACENAPHTHENE	2.70E-01	0.00E+00	6.91E-02	2.16E-04	0.00E+00	6.15E-03	6.37E-03	1.75E+01	3.50E+01	3.6E-04	1.8E-04
ACENAPHTHYLENE	8.53E-02	0.00E+00	2.18E-02	6.83E-05	0.00E+00	1.94E-03	2.01E-03	7.00E+01	7.00E+02	2.9E-05	2.9E-06
ANTHRACENE	6.55E-02	0.00E+00	1.68E-02	5.25E-05	0.00E+00	1.49E-03	1.54E-03	1.00E+02	1.00E+03	1.5E-05	1.5E-06
BENZO(A)ANTHRACENE	2.22E-01	0.00E+00	5.69E-02	1.78E-04	0.00E+00	5.06E-03	5.24E-03	1.70E-01	1.70E+00	3.1E-02	3.1E-03
BENZO(A)PYRENE	2.91E-01	0.00E+00	7.44E-02	2.33E-04	0.00E+00	6.62E-03	6.85E-03	1.00E+00	1.00E+01	6.9E-03	6.9E-04
BENZO(B)FLUORANTHENE	3.22E-01	0.00E+00	8.25E-02	2.58E-04	0.00E+00	7.34E-03	7.60E-03	4.00E+00	4.00E+01	1.9E-03	1.9E-04
BENZO(G,H,I)PERYLENE	1.55E-01	0.00E+00	3.96E-02	1.24E-04	0.00E+00	3.52E-03	3.64E-03	7.20E+00	7.20E+01	5.1E-04	5.1E-05
BENZO(K)FLUORANTHENE	2.71E-01	0.00E+00	6.94E-02	2.17E-04	0.00E+00	6.18E-03	6.39E-03	7.20E+00	7.20E+01	8.9E-04	8.9E-05
CHRYSENE	3.27E-01	0.00E+00	8.36E-02	2.62E-04	0.00E+00	7.44E-03	7.70E-03	1.70E-01	1.70E+00	4.5E-02	4.5E-03
DIBENZO(A,H)ANTHRACENE	3.20E-02	0.00E+00	8.19E-03	2.56E-05	0.00E+00	7.29E-04	7.55E-04	1.33E+00	1.33E+01	5.7E-04	5.7E-05
FLUORANTHENE	3.42E-01	0.00E+00	8.75E-02	2.74E-04	0.00E+00	7.78E-03	8.06E-03	1.25E+01	2.50E+01	6.4E-04	3.2E-04
INDENO(1,2,3-CD)PYRENE	2.05E-01	0.00E+00	5.25E-02	1.64E-04	0.00E+00	4.67E-03	4.83E-03	7.20E+00	7.20E+01	6.7E-04	6.7E-05
PHENANTHRENE	2.69E-01	0.00E+00	6.90E-02	2.16E-04	0.00E+00	6.14E-03	6.35E-03	1.00E+00	1.00E+01	6.4E-03	6.4E-04
PYRENE	3.54E-01	0.00E+00	9.05E-02	2.83E-04	0.00E+00	8.06E-03	8.34E-03	7.50E+00	1.25E+01	1.1E-03	6.7E-04
<b>Pesticides/PCBs</b>											
4,4'-DDD	2.55E-02	0.00E+00	2.47E-01	2.04E-05	0.00E+00	2.20E-02	2.20E-02	1.47E-01	2.74E-01	1.5E-01	8.0E-02
4,4'-DDE	7.60E-03	0.00E+00	1.62E-01	6.09E-06	0.00E+00	1.44E-02	1.44E-02	1.47E-01	2.74E-01	9.8E-02	5.3E-02
4,4'-DDT	1.39E-02	0.00E+00	2.04E-01	1.12E-05	0.00E+00	1.82E-02	1.82E-02	1.47E-01	2.74E-01	1.2E-01	6.6E-02
ALPHA-CHLORDANE	3.00E-03	0.00E+00	1.50E-02	2.40E-06	0.00E+00	1.33E-03	1.34E-03	4.58E+00	9.16E+00	2.9E-04	1.5E-04
AROCLOR-1260	1.36E-01	0.00E+00	9.09E-01	1.09E-04	0.00E+00	8.08E-02	8.10E-02	6.80E-02	6.80E-01	1.2E+00	1.2E-01
GAMMA-CHLORDANE	2.20E-03	0.00E+00	1.10E-02	1.76E-06	0.00E+00	9.79E-04	9.81E-04	4.58E+00	9.16E+00	2.1E-04	1.1E-04
<b>Inorganics</b>											
ARSENIC	7.16E+00	2.83E-03	9.69E-01	5.73E-03	6.34E-04	8.63E-02	9.26E-02	2.47E+00	4.55E+00	3.7E-02	2.0E-02
CADMIUM	3.76E+00	4.01E-02	2.37E+01	3.01E-03	8.97E-03	2.11E+00	2.12E+00	7.70E-01	6.90E+00	2.8E+00	3.1E-01
CHROMIUM	5.85E+01	6.80E-03	1.79E+01	4.68E-02	1.52E-03	1.59E+00	1.64E+00	2.40E+00	5.82E+01	6.8E-01	2.8E-02
COPPER	3.60E+01	1.35E-02	1.85E+01	2.88E-02	3.02E-03	1.65E+00	1.68E+00	5.82E+00	8.14E+01	2.9E-01	2.1E-02
LEAD	4.24E+01	3.82E-03	1.65E+01	3.39E-02	8.53E-04	1.47E+00	1.51E+00	4.70E+00	1.86E+02	3.2E-01	8.1E-03
MERCURY	2.47E-01	2.10E-04	6.75E-01	1.98E-04	4.70E-05	6.01E-02	6.03E-02	3.20E-02	1.60E-01	1.9E+00	3.8E-01
NICKEL	2.13E+01	4.58E-02	2.26E+01	1.71E-02	1.02E-02	2.01E+00	2.04E+00	1.70E+00	1.48E+01	1.2E+00	1.4E-01
SELENIUM	9.20E-01	0.00E+00	8.73E-01	7.37E-04	0.00E+00	7.77E-02	7.84E-02	2.00E-01	3.30E-01	3.9E-01	2.4E-01
SILVER	1.52E+00	0.00E+00	3.10E+00	1.21E-03	0.00E+00	2.76E-01	2.77E-01	6.02E+00	1.19E+02	4.6E-02	2.3E-03
ZINC	7.01E+01	1.33E-01	3.45E+02	5.61E-02	2.97E-02	3.07E+01	3.08E+01	1.60E+02	3.20E+02	1.9E-01	9.6E-02

Cells are shaded if the value is greater than 1.0

Body Weight = (BW) 1.61E-02 kg  
 Food Ingestion Rate = (If) 1.43E-03 kg/day  
 Water Ingestion Rate = (Iw) 3.60E-03 L/day  
 Soil Ingestion Rate = (Is) 1.29E-05 kg/day  
 Home Range = (HR) 9.70E-01 acres  
 Contaminated Area = (CA) Assume equal to home range

Dose (soil) = (Cs \* Is)(H)/BW  
 Dose (invertebrate) = (Ci \* If)(H)/BW  
 Dose (water) = (Cw \* Iw)(H)/BW  
 Ci = Contaminant concentration in invertebrate  
 Cs = Contaminant concentration in soil  
 Cw = Contaminant concentration in water  
 Total Dose = Dose (soil) + Dose (invertebrate) + Dose (water)  
 H=HR/CA (Assume = to 1)

Conc = Concentration  
 LOAEL = Lowest Observed Adverse Effects Concentration  
 NOAEL = No Observed Adverse Effects Concentration  
 SW = Surface Water

AMERICAN ROBIN - CONSERVATIVE INPUTS  
 TERRESTRIAL WILDLIFE MODEL ECOLOGICAL EFFECTS QUOTIENT CALCULATION  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT

Chemical	Max Soil Conc. (mg/kg)	Max SW Conc. (mg/L)	Invertebrate Conc. (mg/kg)	Dose (mg/kg/d) from:			Total Dose (mg/kg/d)	NOAEL (mg/kg/d)	LOAEL (mg/kg/d)	Hazard Quotients	
				Soil	Surface Water	Invert.				NOAEL	LOAEL
<b>Semivolatile Organics</b>											
ACENAPHTHENE	2.70E-01	0.00E+00	1.81E-01	7.15E-03	0.00E+00	2.92E-02	3.63E-02	2.00E+00	2.00E+01	1.8E-02	1.8E-03
ACENAPHTHYLENE	1.20E-01	0.00E+00	8.03E-02	3.18E-03	0.00E+00	1.30E-02	1.61E-02	2.00E+00	2.00E+01	8.1E-03	8.1E-04
ANTHRACENE	7.90E-02	0.00E+00	5.29E-02	2.09E-03	0.00E+00	8.53E-03	1.06E-02	2.00E+00	2.00E+01	5.3E-03	5.3E-04
BENZO(A)ANTHRACENE	4.00E-01	0.00E+00	2.68E-01	1.06E-02	0.00E+00	4.32E-02	5.38E-02	2.00E+00	2.00E+01	2.7E-02	2.7E-03
BENZO(A)PYRENE	3.90E-01	0.00E+00	2.61E-01	1.03E-02	0.00E+00	4.21E-02	5.24E-02	2.00E+00	2.00E+01	2.6E-02	2.6E-03
BENZO(B)FLUORANTHENE	5.50E-01	0.00E+00	3.68E-01	1.46E-02	0.00E+00	5.94E-02	7.39E-02	2.00E+00	2.00E+01	3.7E-02	3.7E-03
BENZO(G,H,I)PERYLENE	2.60E-01	0.00E+00	1.74E-01	6.88E-03	0.00E+00	2.81E-02	3.50E-02	2.00E+00	2.00E+01	1.7E-02	1.7E-03
BENZO(K)FLUORANTHENE	3.90E-01	0.00E+00	2.61E-01	1.03E-02	0.00E+00	4.21E-02	5.24E-02	2.00E+00	2.00E+01	2.6E-02	2.6E-03
CHRYSENE	6.00E-01	0.00E+00	4.01E-01	1.59E-02	0.00E+00	6.48E-02	8.07E-02	2.00E+00	2.00E+01	4.0E-02	4.0E-03
DIBENZO(A,H)ANTHRACENE	3.20E-02	0.00E+00	2.14E-02	8.47E-04	0.00E+00	3.45E-03	4.30E-03	2.00E+00	2.00E+01	2.2E-03	2.2E-04
FLUORANTHENE	8.90E-01	0.00E+00	5.95E-01	2.36E-02	0.00E+00	9.61E-02	1.20E-01	2.00E+00	2.00E+01	6.0E-02	6.0E-03
INDENO(1,2,3-CD)PYRENE	2.70E-01	0.00E+00	1.81E-01	7.15E-03	0.00E+00	2.92E-02	3.63E-02	2.00E+00	2.00E+01	1.8E-02	1.8E-03
PHENANTHRENE	3.40E-01	0.00E+00	2.27E-01	9.00E-03	0.00E+00	3.67E-02	4.57E-02	2.00E+00	2.00E+01	2.3E-02	2.3E-03
PYRENE	8.10E-01	0.00E+00	5.42E-01	2.14E-02	0.00E+00	8.75E-02	1.09E-01	2.00E+00	2.00E+01	5.4E-02	5.4E-03
<b>Pesticides/PCBs</b>											
4,4'-DDD	6.90E-02	0.00E+00	4.95E-01	1.83E-03	0.00E+00	7.99E-02	8.17E-02	2.27E-01	2.81E-01	3.6E-01	2.9E-01
4,4'-DDE	1.00E-02	0.00E+00	2.07E-01	2.65E-04	0.00E+00	3.33E-02	3.36E-02	2.27E-01	2.81E-01	1.5E-01	1.2E-01
4,4'-DDT	2.80E-02	0.00E+00	3.75E-01	7.41E-04	0.00E+00	6.04E-02	6.12E-02	2.27E-01	2.81E-01	2.7E-01	2.2E-01
ALPHA-CHLORDANE	3.00E-03	0.00E+00	1.50E-02	7.94E-05	0.00E+00	2.42E-03	2.50E-03	2.14E+00	1.07E+01	1.2E-03	2.3E-04
AROCLOR-1260	3.70E-01	0.00E+00	5.89E+00	9.79E-03	0.00E+00	9.50E-01	9.60E-01	1.80E-01	1.80E+00	<b>5.3E+00</b>	5.3E-01
GAMMA-CHLORDANE	2.20E-03	0.00E+00	1.10E-02	5.82E-05	0.00E+00	1.78E-03	1.83E-03	2.14E+00	1.07E+01	8.6E-04	1.7E-04
<b>Inorganics</b>											
ARSENIC	1.51E+01	2.90E-03	1.64E+00	4.00E-01	4.53E-04	2.65E-01	6.65E-01	2.24E+00	4.51E+00	3.0E-01	1.5E-01
CADMIUM	7.20E+00	1.26E-01	3.98E+01	1.91E-01	1.97E-02	6.42E+00	6.63E+00	1.47E+00	6.35E+00	<b>4.5E+00</b>	<b>1.0E+00</b>
CHROMIUM	1.02E+02	6.80E-03	3.12E+01	2.70E+00	1.06E-03	5.04E+00	7.74E+00	2.66E+00	1.56E+01	<b>2.9E+00</b>	5.0E-01
COPPER	6.41E+01	2.93E-02	3.30E+01	1.70E+00	4.57E-03	5.33E+00	7.03E+00	4.05E+00	3.48E+01	<b>1.7E+00</b>	2.0E-01
LEAD	1.28E+02	7.80E-03	4.03E+01	3.39E+00	1.22E-03	6.51E+00	9.90E+00	1.63E+00	4.46E+01	<b>6.1E+00</b>	2.2E-01
MERCURY	6.90E-01	2.10E-04	9.54E-01	1.83E-02	3.28E-05	1.54E-01	1.72E-01	6.40E-03	6.40E-02	<b>2.7E+01</b>	<b>2.7E+00</b>
NICKEL	2.69E+01	8.47E-02	2.85E+01	7.12E-01	1.32E-02	4.60E+00	5.32E+00	6.71E+00	1.86E+01	7.9E-01	2.9E-01
SELENIUM	2.40E+00	0.00E+00	1.76E+00	6.35E-02	0.00E+00	2.84E-01	3.48E-01	4.00E-01	8.00E-01	8.7E-01	4.3E-01
SILVER	4.50E+00	0.00E+00	9.20E+00	1.19E-01	0.00E+00	1.49E+00	1.60E+00	2.02E+00	6.05E+01	7.9E-01	2.7E-02
ZINC	1.25E+02	3.34E-01	4.17E+02	3.31E+00	5.21E-02	6.73E+01	7.06E+01	1.45E+01	1.31E+02	<b>4.9E+00</b>	5.4E-01

Cells are shaded if the value is greater than 1.0

Body Weight = (BW) 7.73E-02 kg  
 Food Ingestion Rate = (If) 1.25E-02 kg/day  
 Water Ingestion Rate = (Iw) 1.21E-02 L/day  
 Soil Ingestion Rate = (Is) 2.05E-03 kg/day  
 Home Range = (HR) Assume 100% on site  
 Contaminated Area = (CA) Assume equal to home range

Dose (soil) = (Cs \* Is)(H)/BW  
 Dose (invertebrate) = (Ci \* If)(H)/BW  
 Dose (water) = (Cw \* Iw)(H)/BW  
 Ci = Contaminant concentration in invertebrate  
 Cs = Contaminant concentration in soil  
 Cw = Contaminant concentration in water  
 Total Dose = Dose (soil) + Dose (invertebrate) + Dose (water)  
 H=HR/CA (Assume = to 1)

Conc = Concentration  
 LOAEL = Lowest Observed Adverse Effects Concentration  
 NOAEL = No Observed Adverse Effects Concentration  
 SW = Surface Water

AMERICAN ROBIN - AVERAGE INPUTS  
 TERRESTRIAL WILDLIFE MODEL ECOLOGICAL EFFECTS QUOTIENT CALCULATION  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT

Chemical	Avg Soil Conc. (mg/kg)	Avg SW Conc. (mg/L)	Invertebrate Conc. (mg/kg)	Dose (mg/kg/d) from:			Total Dose (mg/kg/d)	NOAEL (mg/kg/d)	LOAEL (mg/kg/d)	Hazard Quotients	
				Soil	Surface Water	Invert.				NOAEL	LOAEL
<b>Semivolatile Organics</b>											
ACENAPHTHENE	2.70E-01	0.00E+00	6.91E-02	2.55E-03	0.00E+00	1.02E-02	1.28E-02	2.00E+00	2.00E+01	6.4E-03	6.4E-04
ACENAPHTHYLENE	8.53E-02	0.00E+00	2.18E-02	8.07E-04	0.00E+00	3.23E-03	4.03E-03	2.00E+00	2.00E+01	2.0E-03	2.0E-04
ANTHRACENE	6.55E-02	0.00E+00	1.68E-02	6.19E-04	0.00E+00	2.48E-03	3.10E-03	2.00E+00	2.00E+01	1.5E-03	1.5E-04
BENZO(A)ANTHRACENE	2.22E-01	0.00E+00	5.69E-02	2.10E-03	0.00E+00	8.41E-03	1.05E-02	2.00E+00	2.00E+01	5.3E-03	5.3E-04
BENZO(A)PYRENE	2.91E-01	0.00E+00	7.44E-02	2.75E-03	0.00E+00	1.10E-02	1.37E-02	2.00E+00	2.00E+01	6.9E-03	6.9E-04
BENZO(B)FLUORANTHENE	3.22E-01	0.00E+00	8.25E-02	3.05E-03	0.00E+00	1.22E-02	1.52E-02	2.00E+00	2.00E+01	7.6E-03	7.6E-04
BENZO(G,H,I)PERYLENE	1.55E-01	0.00E+00	3.96E-02	1.46E-03	0.00E+00	5.84E-03	7.30E-03	2.00E+00	2.00E+01	3.7E-03	3.7E-04
BENZO(K)FLUORANTHENE	2.71E-01	0.00E+00	6.94E-02	2.56E-03	0.00E+00	1.03E-02	1.28E-02	2.00E+00	2.00E+01	6.4E-03	6.4E-04
CHRYSENE	3.27E-01	0.00E+00	8.36E-02	3.09E-03	0.00E+00	1.24E-02	1.54E-02	2.00E+00	2.00E+01	7.7E-03	7.7E-04
DIBENZO(A,H)ANTHRACENE	3.20E-02	0.00E+00	8.19E-03	3.03E-04	0.00E+00	1.21E-03	1.51E-03	2.00E+00	2.00E+01	7.6E-04	7.6E-05
FLUORANTHENE	3.42E-01	0.00E+00	8.75E-02	3.23E-03	0.00E+00	1.29E-02	1.62E-02	2.00E+00	2.00E+01	8.1E-03	8.1E-04
INDENO(1,2,3-CD)PYRENE	2.05E-01	0.00E+00	5.25E-02	1.94E-03	0.00E+00	7.75E-03	9.69E-03	2.00E+00	2.00E+01	4.8E-03	4.8E-04
PHENANTHRENE	2.69E-01	0.00E+00	6.90E-02	2.55E-03	0.00E+00	1.02E-02	1.27E-02	2.00E+00	2.00E+01	6.4E-03	6.4E-04
PYRENE	3.54E-01	0.00E+00	9.05E-02	3.34E-03	0.00E+00	1.34E-02	1.67E-02	2.00E+00	2.00E+01	8.4E-03	8.4E-04
<b>Pesticides/PCBs</b>											
4,4'-DDD	2.55E-02	0.00E+00	2.47E-01	2.41E-04	0.00E+00	3.65E-02	3.67E-02	2.27E-01	2.81E-01	1.6E-01	1.3E-01
4,4'-DDE	7.60E-03	0.00E+00	1.62E-01	7.19E-05	0.00E+00	2.40E-02	2.40E-02	2.27E-01	2.81E-01	1.1E-01	8.6E-02
4,4'-DDT	1.39E-02	0.00E+00	2.04E-01	1.32E-04	0.00E+00	3.02E-02	3.03E-02	2.27E-01	2.81E-01	1.3E-01	1.1E-01
ALPHA-CHLORDANE	3.00E-03	0.00E+00	1.50E-02	2.84E-05	0.00E+00	2.22E-03	2.24E-03	2.14E+00	1.07E+01	1.0E-03	2.1E-04
AROCLOR-1260	1.36E-01	0.00E+00	9.09E-01	1.29E-03	0.00E+00	1.34E-01	1.36E-01	1.80E-01	1.80E+00	7.5E-01	7.5E-02
GAMMA-CHLORDANE	2.20E-03	0.00E+00	1.10E-02	2.08E-05	0.00E+00	1.62E-03	1.65E-03	2.14E+00	1.07E+01	7.7E-04	1.5E-04
<b>Inorganics</b>											
ARSENIC	7.16E+00	2.83E-03	9.69E-01	6.77E-02	4.00E-04	1.43E-01	2.11E-01	2.24E+00	4.51E+00	9.4E-02	4.7E-02
CADMIUM	3.76E+00	4.01E-02	2.37E+01	3.55E-02	5.66E-03	3.50E+00	3.54E+00	1.47E+00	6.35E+00	2.4E+00	5.6E-01
CHROMIUM	5.85E+01	6.80E-03	1.79E+01	5.53E-01	9.59E-04	2.64E+00	3.20E+00	2.66E+00	1.56E+01	1.2E+00	2.0E-01
COPPER	3.60E+01	1.35E-02	1.85E+01	3.41E-01	1.91E-03	2.74E+00	3.08E+00	4.05E+00	3.48E+01	7.6E-01	8.9E-02
LEAD	4.24E+01	3.82E-03	1.65E+01	4.00E-01	5.38E-04	2.44E+00	2.84E+00	1.63E+00	4.46E+01	1.7E+00	6.4E-02
MERCURY	2.47E-01	2.10E-04	6.75E-01	2.33E-03	2.96E-05	9.97E-02	1.02E-01	6.40E-03	6.40E-02	1.6E+01	1.6E+00
NICKEL	2.13E+01	4.58E-02	2.26E+01	2.02E-01	6.46E-03	3.34E+00	3.55E+00	6.71E+00	1.86E+01	5.3E-01	1.9E-01
SELENIUM	9.20E-01	0.00E+00	8.73E-01	8.70E-03	0.00E+00	1.29E-01	1.38E-01	4.00E-01	8.00E-01	3.4E-01	1.7E-01
SILVER	1.52E+00	0.00E+00	3.10E+00	1.43E-02	0.00E+00	4.58E-01	4.72E-01	2.02E+00	6.05E+01	2.3E-01	7.8E-03
ZINC	7.01E+01	1.33E-01	3.45E+02	6.63E-01	1.88E-02	5.09E+01	5.16E+01	1.45E+01	1.31E+02	3.6E+00	3.9E-01

Cells are shaded if the value is greater than 1.0

Body Weight = (BW)	8.04E-02	kg	Dose (soil) = (Cs * Is)(H)/BW	Conc = Concentration
Food Ingestion Rate = (If)	1.19E-02	kg/day	Dose (invertebrate) = (Ci * If)(H)/BW	LOAEL = Lowest Observed Adverse Effects Concentration
Water Ingestion Rate = (Iw)	1.13E-02	L/day	Dose (water) = (Cw * Iw)(H)/BW	NOAEL = No Observed Adverse Effects Concentration
Soil Ingestion Rate = (Is)	7.60E-04	kg/day	Ci = Contaminant concentration in invertebrate	SW = Surface Water
Home Range = (HR)	0.00E+00	acres	Cs = Contaminant concentration in soil	
Contaminated Area = (CA)	Assume equal to home range		Cw = Contaminant concentration in water	
			Total Dose = Dose (soil) + Dose (invertebrate) + Dose (water)	
			H=HR/CA (Assume = to 1)	

MALLARD DUCK - CONSERVATIVE INPUTS  
 TERRESTRIAL WILDLIFE MODEL ECOLOGICAL EFFECTS QUOTIENT CALCULATION  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT

Chemical	Max Sed Conc. (mg/kg)	Max SW Conc (mg/L)	Invertebrate Conc. (mg/kg)	Dose (mg/kg/d) from			Total Dose (mg/kg/d)	NOAEL (mg/kg/d)	LOAEL (mg/kg/d)	Hazard Quotients	
				Sediment	Surface Water	Invert				NOAEL	LOAEL
<b>Semivolatile Organics</b>											
1,4-DICHLOROBENZENE	4.20E-02	0.00E+00	9.44E-02	1.09E-04	0.00E+00	7.42E-03	7.53E-03	NV	NV	#VALUE!	#VALUE!
2-METHYLNAPHTHALENE	5.50E-02	0.00E+00	1.24E-01	1.43E-04	0.00E+00	9.72E-03	9.86E-03	2.00E+00	2.00E+01	4.9E-03	4.9E-04
ACENAPHTHENE	3.80E-01	0.00E+00	2.48E-01	9.86E-04	0.00E+00	1.95E-02	2.05E-02	2.00E+00	2.00E+01	1.0E-02	1.0E-03
ACENAPHTHYLENE	3.90E-01	0.00E+00	2.54E-01	1.01E-03	0.00E+00	2.00E-02	2.10E-02	2.00E+00	2.00E+01	1.1E-02	1.1E-03
ANTHRACENE	2.40E+00	0.00E+00	1.56E+00	6.23E-03	0.00E+00	1.23E-01	1.29E-01	2.00E+00	2.00E+01	6.5E-02	6.5E-03
BENZO(A)ANTHRACENE	2.70E+01	0.00E+00	1.76E-01	7.00E-02	0.00E+00	1.38E+00	1.45E+00	2.00E+00	2.00E+01	7.3E-01	7.3E-02
BENZO(A)PYRENE	3.50E+01	0.00E+00	2.28E+01	9.08E-02	0.00E+00	1.79E+00	1.88E+00	2.00E+00	2.00E+01	9.4E-01	9.4E-02
BENZO(B)FLUORANTHENE	5.50E+01	0.00E+00	3.59E+01	1.43E-01	0.00E+00	2.82E+00	2.96E+00	2.00E+00	2.00E+01	1.5E+00	1.5E-01
BENZO(G,H)PERYLENE	2.30E+01	0.00E+00	1.50E-01	5.97E-02	0.00E+00	1.18E+00	1.24E+00	2.00E+00	2.00E+01	6.2E-01	6.2E-02
BENZO(K)FLUORANTHENE	4.50E+01	0.00E+00	2.93E+01	1.17E-01	0.00E+00	2.31E+00	2.42E+00	2.00E+00	2.00E+01	1.2E+00	1.2E-01
CHRYSENE	4.20E+01	0.00E+00	2.74E-01	1.09E-01	0.00E+00	2.15E+00	2.26E+00	2.00E+00	2.00E+01	1.1E+00	1.1E-01
DIBENZO(A,H)ANTHRACENE	3.10E-01	0.00E+00	2.02E-01	8.04E-04	0.00E+00	1.59E-02	1.67E-02	2.00E+00	2.00E+01	8.3E-03	8.3E-04
FLUORANTHENE	8.00E+01	0.00E+00	5.22E+01	2.08E-01	0.00E+00	4.10E+00	4.31E+00	2.00E+00	2.00E+01	2.2E+00	2.2E-01
FLUORENE	1.00E+00	0.00E+00	6.52E-01	2.59E-03	0.00E+00	5.13E-02	5.39E-02	2.00E+00	2.00E+01	2.7E-02	2.7E-03
INDENO(1,2,3-CD)PYRENE	2.30E+01	0.00E+00	1.50E+01	5.97E-02	0.00E+00	1.18E+00	1.24E+00	2.00E+00	2.00E+01	6.2E-01	6.2E-02
NAPHTHALENE	7.70E-02	0.00E+00	5.02E-02	2.00E-04	0.00E+00	3.99E-03	4.15E-03	2.00E+00	2.00E+01	2.1E-03	2.1E-04
PENTACHLOROPHENOL	2.40E-01	0.00E+00	0.00E+00	6.23E-04	0.00E+00	0.00E+00	6.23E-04	6.73E-04	5.20E+01	9.3E-05	1.2E-05
PHENANTHRENE	3.60E+01	0.00E+00	2.35E+01	9.34E-02	0.00E+00	1.85E+00	1.94E+00	2.00E+00	2.00E+01	9.7E-01	9.7E-02
PYRENE	4.20E+01	0.00E+00	2.74E+01	1.09E-01	0.00E+00	2.15E+00	2.26E+00	2.00E+00	2.00E+01	1.1E+00	1.1E-01
<b>Pesticides/PCBs</b>											
4,4'-DDD	4.80E+00	0.00E+00	3.02E+00	1.25E-02	0.00E+00	2.38E-01	2.50E-01	2.27E-01	2.81E-01	1.1E+00	8.9E-01
4,4'-DDE	7.20E-01	0.00E+00	1.25E+01	1.87E-03	0.00E+00	9.80E-01	9.82E-01	2.27E-01	2.81E-01	4.3E+00	3.5E+00
4,4'-DDT	2.90E+00	0.00E+00	1.09E+01	7.52E-03	0.00E+00	8.56E-01	8.64E-01	2.27E-01	2.81E-01	3.8E+00	3.1E+00
ALDRIN	3.20E-03	0.00E+00	1.29E-02	8.30E-06	0.00E+00	1.02E-03	1.03E-03	NV	NV	#VALUE!	#VALUE!
ALPHA-CHLORDANE	2.90E-02	0.00E+00	3.11E-01	7.52E-05	0.00E+00	2.45E-02	2.45E-02	2.14E+00	1.07E+01	1.1E-02	2.3E-03
AROCCLOR-1260	1.50E+00	0.00E+00	2.16E+02	3.89E-03	0.00E+00	1.70E+01	1.70E+01	1.80E+01	1.80E+00	9.4E+01	9.4E+00
BETA-BHC	2.70E-03	0.00E+00	1.09E-02	7.00E-06	0.00E+00	8.59E-04	8.66E-04	5.60E-01	2.25E+00	1.5E-03	3.8E-04
DELTA-BHC	4.20E-03	0.00E+00	1.70E-02	1.09E-05	0.00E+00	1.34E-03	1.35E-03	5.60E-01	2.25E+00	2.4E-03	6.0E-04
DIELDRIN	2.60E-02	0.00E+00	1.05E-01	6.75E-05	0.00E+00	8.27E-03	8.34E-03	7.09E-02	8.00E-01	1.2E-01	1.0E-02
ENDOSULFAN I	1.10E-02	0.00E+00	4.45E-02	2.85E-05	0.00E+00	3.50E-03	3.53E-03	1.00E+01	1.00E+02	3.5E-04	3.5E-05
ENDOSULFAN II	3.10E-02	0.00E+00	1.25E-01	8.04E-05	0.00E+00	9.86E-03	9.94E-03	1.00E+01	1.00E+02	9.9E-04	9.9E-05
ENDOSULFAN SULFATE	1.40E-02	0.00E+00	5.67E-02	3.63E-05	0.00E+00	4.45E-03	4.49E-03	1.00E+01	1.00E+02	4.5E-04	4.5E-05
ENDRIN	1.60E-02	0.00E+00	6.47E-02	4.15E-05	0.00E+00	5.09E-03	5.13E-03	1.04E-02	1.04E-01	5.0E-01	5.0E-02
ENDRIN ALDEHYDE	1.60E-02	0.00E+00	6.47E-02	4.15E-05	0.00E+00	5.09E-03	5.13E-03	1.00E-02	1.00E-01	5.1E-01	5.1E-02
ENDRIN KETONE	2.00E-02	0.00E+00	8.09E-02	5.19E-05	0.00E+00	6.36E-03	6.42E-03	1.00E-02	1.00E-01	6.4E-01	6.4E-02
GAMMA-BHC (LINDANE)	3.50E-03	0.00E+00	1.42E-02	9.08E-06	0.00E+00	1.11E-03	1.12E-03	2.00E+00	2.00E+01	5.6E-04	5.6E-05
GAMMA-CHLORDANE	2.30E-02	0.00E+00	1.15E-01	5.97E-05	0.00E+00	9.03E-03	9.08E-03	2.14E+00	1.07E+01	4.2E-03	8.5E-04
HEPTACHLOR	4.50E-03	0.00E+00	1.82E-02	1.17E-05	0.00E+00	1.43E-03	1.44E-03	NV	NV	#VALUE!	#VALUE!
HEPTACHLOR EPOXIDE	4.50E-03	0.00E+00	1.82E-02	1.17E-05	0.00E+00	1.43E-03	1.44E-03	NV	NV	#VALUE!	#VALUE!
METHOXYCHLOR	3.80E-02	0.00E+00	1.54E-01	9.86E-05	0.00E+00	1.21E-02	1.22E-02	NV	NV	#VALUE!	#VALUE!
<b>Inorganics</b>											
ARSENIC	1.41E+01	2.90E-03	9.73E+00	3.66E-02	1.88E-03	7.65E-01	8.03E-01	2.24E+00	4.51E+00	3.6E-01	1.8E-01
CADMIUM	6.10E+00	1.26E-01	4.87E+01	1.58E-02	8.17E-02	3.83E+00	3.93E+00	1.47E+00	6.35E+00	2.7E+00	6.2E-01
CHROMIUM	9.68E+01	6.80E-03	4.53E+01	2.51E-01	4.41E-03	3.56E+00	3.82E+00	2.66E+00	1.56E+01	1.4E+00	2.4E-01
COPPER	1.73E+02	2.93E-02	9.08E+02	4.49E-01	1.90E-02	7.14E+01	7.19E+01	4.05E+00	3.48E+01	1.8E+01	2.1E+00
LEAD	2.41E+02	7.80E-03	1.46E+02	6.25E-01	5.06E-03	1.15E+01	1.21E+01	1.63E+00	4.46E+01	7.4E+00	2.7E-01
MERCURY	1.20E+00	2.10E-04	3.44E+00	3.11E-03	1.36E-04	2.71E-01	2.74E-01	6.40E-03	6.40E-02	4.3E+01	4.3E+00
NICKEL	6.15E+01	8.47E-02	1.43E+02	1.60E-01	5.49E-02	1.12E+01	1.14E+01	6.71E+00	1.86E+01	1.7E+00	6.2E-01
SELENIUM	6.80E+00	0.00E+00	6.80E+00	1.76E-02	0.00E+00	5.35E-01	5.52E-01	4.00E-01	8.00E-01	1.4E+00	6.9E-01
SILVER	9.60E-01	0.00E+00	9.60E-01	2.49E-03	0.00E+00	7.55E-02	7.80E-02	2.02E+00	6.05E+01	3.9E-02	1.3E-03
ZINC	7.02E+02	3.34E-01	5.28E+03	1.82E+00	2.16E-01	4.15E+02	4.17E+02	1.45E+01	1.31E+02	2.9E+01	9.2E+00

Cells are shaded if the value is greater than 1.0

Body Weight = (BW) 1.04E+00 kg  
 Food Ingestion Rate = (If) 8.20E-02 kg/day  
 Water Ingestion Rate = (Iw) 6.76E-01 L/day  
 Sediment Ingestion Rate = (Is) 2.71E-03 kg/day  
 Home Range = (HR) Assume 100% on site  
 Contaminated Area = (CA) Assume equal to home range

Dose (sediment) = (Cs \* Is)(H)/BW  
 Dose (invertebrate) = (Ci \* If)(H)/BW  
 Dose (water) = (Cw \* Iw)(H)/BW  
 Ci = Contaminant concentration in invertebrate  
 Cs = Contaminant concentration in sediment  
 Cw = Contaminant concentration in water  
 Total Dose = Dose (sediment) + Dose (invertebrate) + Dose (water)  
 H=HR/CA (Assume = to 1)

Conc = Concentration  
 LOAEL = Lowest Observed Adverse Effects Concentration  
 NOAEL = No Observed Adverse Effects Concentration  
 Sed = Sediment  
 SW = Surface Water

MALLARD DUCK - AVERAGE INPUTS  
 TERRESTRIAL WILDLIFE MODEL ECOLOGICAL EFFECTS QUOTIENT CALCULATION  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT

Chemical	Avg Sed. Conc. (mg/kg)	Avg SW Conc. (mg/L)	Invertebrate Conc (mg/kg)	Dose (mg/kg/d) from.			Total Dose (mg/kg/d)	NOAEL (mg/kg/d)	LOAEL (mg/kg/d)	Hazard Quotients	
				Sediment	Surface Water	Invert				NOAEL	LOAEL
<b>Semivolatile Organics</b>											
1,4-DICHLOROBENZENE	4.20E-02	0.00E+00	9.44E-02	9.30E-05	0.00E+00	6.34E-03	6.43E-03	NV	NV	#VALUE!	#VALUE!
2-METHYLNAPHTHALENE	4.88E-02	0.00E+00	1.10E-01	1.08E-04	0.00E+00	7.36E-03	7.46E-03	2.00E+00	2.00E+01	3.7E-03	3.7E-04
ACENAPHTHENE	1.22E-01	0.00E+00	7.96E-02	2.70E-04	0.00E+00	5.34E-03	5.61E-03	2.00E+00	2.00E+01	2.8E-03	2.8E-04
ACENAPHTHYLENE	1.37E-01	0.00E+00	8.90E-02	3.02E-04	0.00E+00	5.97E-03	6.28E-03	2.00E+00	2.00E+01	3.1E-03	3.1E-04
ANTHRACENE	6.35E-01	0.00E+00	4.14E-01	1.41E-03	0.00E+00	2.78E-02	2.92E-02	2.00E+00	2.00E+01	1.5E-02	1.5E-03
BENZO(A)ANTHRACENE	1.58E+00	0.00E+00	1.03E+00	3.50E-03	0.00E+00	6.92E-02	7.27E-02	2.00E+00	2.00E+01	3.6E-02	3.6E-03
BENZO(A)PYRENE	1.90E+00	0.00E+00	1.24E+00	4.20E-03	0.00E+00	8.29E-02	8.71E-02	2.00E+00	2.00E+01	4.4E-02	4.4E-03
BENZO(B)FLUORANTHENE	2.41E+00	0.00E+00	1.57E+00	5.34E-03	0.00E+00	1.06E-01	1.11E-01	2.00E+00	2.00E+01	5.5E-02	5.5E-03
BENZO(G,H,I)PERYLENE	1.43E+00	0.00E+00	9.30E-01	3.16E-03	0.00E+00	6.24E-02	6.56E-02	2.00E+00	2.00E+01	3.3E-02	3.3E-03
BENZO(K)FLUORANTHENE	2.20E+00	0.00E+00	1.43E+00	4.87E-03	0.00E+00	9.62E-02	1.01E-01	2.00E+00	2.00E+01	5.1E-02	5.1E-03
CHRYSENE	2.13E+00	0.00E+00	1.39E+00	4.71E-03	0.00E+00	9.31E-02	9.78E-02	2.00E+00	2.00E+01	4.9E-02	4.9E-03
DIBENZO(A,H)ANTHRACENE	1.25E-01	0.00E+00	8.15E-02	2.77E-04	0.00E+00	5.47E-03	5.75E-03	2.00E+00	2.00E+01	2.9E-03	2.9E-04
FLUORANTHENE	3.41E+00	0.00E+00	2.22E+00	7.55E-03	0.00E+00	1.49E-01	1.57E-01	2.00E+00	2.00E+01	7.8E-02	7.8E-03
FLUORENE	5.96E-01	0.00E+00	3.88E-01	1.32E-03	0.00E+00	2.61E-02	2.74E-02	2.00E+00	2.00E+01	1.4E-02	1.4E-03
INDENO(1,2,3-CD)PYRENE	1.40E+00	0.00E+00	9.10E-01	3.09E-03	0.00E+00	6.11E-02	6.42E-02	2.00E+00	2.00E+01	3.2E-02	3.2E-03
NAPHTHALENE	6.78E-02	0.00E+00	4.42E-02	1.50E-04	0.00E+00	2.97E-03	3.12E-03	2.00E+00	2.00E+01	1.6E-03	1.6E-04
PENTACHLOROPHENOL	2.40E-01	0.00E+00	0.00E+00	5.32E-04	0.00E+00	0.00E+00	5.32E-04	6.73E-04	5.20E-01	7.9E-05	1.0E-05
PHENANTHRENE	1.84E+00	0.00E+00	1.20E+00	4.08E-03	0.00E+00	8.06E-02	8.47E-02	2.00E+00	2.00E+01	4.2E-02	4.2E-03
PYRENE	2.23E+00	0.00E+00	1.45E+00	4.93E-03	0.00E+00	9.74E-02	1.02E-01	2.00E+00	2.00E+01	5.1E-02	5.1E-03
<b>Pesticides/PCBs</b>											
4,4'-DDD	2.39E-01	0.00E+00	1.51E-01	5.30E-04	0.00E+00	1.01E-02	1.06E-02	2.27E-01	2.81E-01	4.7E-02	3.8E-02
4,4'-DDE	4.25E-02	0.00E+00	7.36E-01	9.41E-05	0.00E+00	4.94E-02	4.95E-02	2.27E-01	2.81E-01	2.2E-01	1.8E-01
4,4'-DDT	1.46E-01	0.00E+00	5.48E-01	3.23E-04	0.00E+00	3.68E-02	3.71E-02	2.27E-01	2.81E-01	1.6E-01	1.3E-01
ALDRIN	3.20E-03	0.00E+00	1.29E-02	7.09E-06	0.00E+00	8.69E-04	8.76E-04	NV	NV	#VALUE!	#VALUE!
ALPHA-CHLORDANE	2.45E-02	0.00E+00	2.62E-01	5.42E-05	0.00E+00	1.77E-02	1.77E-02	2.14E+00	1.07E-01	8.3E-03	1.07E-03
AROCLOR-1260	1.81E-01	0.00E+00	1.47E-01	4.00E-04	0.00E+00	9.85E-01	9.87E-01	1.80E-01	1.80E+00	5.5E+00	5.5E-01
BETA-BHC	2.70E-03	0.00E+00	1.09E-02	5.98E-06	0.00E+00	7.33E-04	7.39E-04	5.60E-01	2.25E+00	1.3E-03	3.3E-04
DELTA-BHC	4.20E-03	0.00E+00	1.70E-02	9.30E-06	0.00E+00	1.14E-03	1.15E-03	5.60E-01	2.25E+00	2.1E-03	5.1E-04
DIELDRIN	1.17E-02	0.00E+00	4.74E-02	2.59E-05	0.00E+00	3.18E-03	3.20E-03	7.09E-02	8.00E-01	4.5E-02	4.0E-03
ENDOSULFAN I	4.63E-03	0.00E+00	1.87E-02	1.03E-05	0.00E+00	1.26E-03	1.27E-03	1.00E+01	1.00E+02	1.3E-04	1.3E-05
ENDOSULFAN II	9.58E-03	0.00E+00	3.88E-02	2.12E-05	0.00E+00	2.60E-03	2.62E-03	1.00E+01	1.00E+02	2.6E-04	2.6E-05
ENDOSULFAN SULFATE	8.65E-03	0.00E+00	3.50E-02	1.92E-05	0.00E+00	2.35E-03	2.37E-03	1.00E+01	1.00E+02	2.4E-04	2.4E-05
ENDRIN	9.02E-03	0.00E+00	3.65E-02	2.00E-05	0.00E+00	2.45E-03	2.47E-03	1.04E-02	1.04E-01	2.4E-01	2.4E-02
ENDRIN ALDEHYDE	6.22E-03	0.00E+00	2.52E-02	1.38E-05	0.00E+00	1.69E-03	1.70E-03	1.00E-02	1.00E-01	1.7E-01	1.7E-02
ENDRIN KETONE	9.15E-03	0.00E+00	3.70E-02	2.03E-05	0.00E+00	2.49E-03	2.51E-03	1.00E-02	1.00E-01	2.5E-01	2.5E-02
GAMMA-BHC (LINDANE)	3.50E-03	0.00E+00	1.42E-02	7.75E-06	0.00E+00	9.51E-04	9.58E-04	2.00E+00	2.00E+01	4.8E-04	4.8E-05
GAMMA-CHLORDANE	8.79E-03	0.00E+00	4.39E-02	1.95E-05	0.00E+00	2.95E-03	2.97E-03	2.14E+00	1.07E+01	1.4E-03	2.8E-04
HEPTACHLOR	4.49E-03	0.00E+00	1.82E-02	9.95E-06	0.00E+00	1.22E-03	1.23E-03	NV	NV	#VALUE!	#VALUE!
HEPTACHLOR EPOXIDE	3.53E-03	0.00E+00	1.43E-02	7.81E-06	0.00E+00	9.57E-04	9.65E-04	NV	NV	#VALUE!	#VALUE!
METHOXYCHLOR	3.80E-02	0.00E+00	1.54E-01	8.42E-05	0.00E+00	1.03E-02	1.04E-02	NV	NV	#VALUE!	#VALUE!
<b>Inorganics</b>											
ARSENIC	6.55E+00	2.83E-03	9.36E-01	1.45E-02	1.60E-04	6.28E-02	7.75E-02	2.24E+00	4.51E+00	3.5E-02	1.7E-02
CADMIUM	1.66E+00	4.01E-02	9.96E-01	3.68E-03	2.26E-03	6.69E-02	7.28E-02	1.47E+00	6.35E+00	5.0E-02	1.1E-02
CHROMIUM	4.39E+01	6.80E-03	4.39E+00	9.71E-02	3.84E-04	2.94E-01	3.92E-01	2.66E+00	1.58E+01	1.5E-01	2.5E-02
COPPER	5.36E+01	1.35E-02	8.34E+01	1.19E-01	7.62E-04	5.60E+00	5.72E+00	4.05E+00	3.48E+01	1.4E+00	1.6E-01
LEAD	5.89E+01	3.82E-03	4.18E+00	1.30E-01	2.15E-04	2.81E-01	4.11E-01	1.63E+00	4.46E+01	2.5E-01	9.2E-03
MERCURY	2.95E-01	2.10E-04	3.36E-01	6.54E-04	1.19E-05	2.25E-02	2.32E-02	6.40E-03	6.40E-02	3.6E+00	3.6E-01
NICKEL	2.02E+01	4.59E-02	9.83E+00	4.48E-02	2.59E-03	6.60E-01	7.07E-01	6.71E+00	1.86E+01	1.1E-01	3.8E-02
SELENIUM	1.18E+00	0.00E+00	1.18E+00	2.51E-03	0.00E+00	7.91E-02	8.17E-02	4.00E-01	8.00E-01	2.0E-01	1.0E-01
SILVER	6.15E-01	0.00E+00	6.15E-01	1.36E-03	0.00E+00	4.13E-02	4.26E-02	2.02E+00	6.05E+01	2.1E-02	7.0E-04
ZINC	1.27E+02	1.33E-01	2.45E+02	2.81E-01	7.50E-03	1.65E+01	1.67E+01	1.45E+01	1.31E+02	1.2E+00	1.3E-01

Cells are shaded if the value is greater than 1 0

Body Weight = (BW) 1.17E+00 kg  
 Food Ingestion Rate = (If) 7.83E-02 kg/day  
 Water Ingestion Rate = (Iw) 6.58E-02 L/day  
 Sediment Ingestion Rate = (Is) 2.58E-03 kg/day  
 Home Range = (HR) 1.00E+01 acres  
 Contaminated Area = (CA) Assume equal to home range

Dose (sediment) = (Cs \* Is)(H)/BW  
 Dose (invertebrate) = (Ci \* If)(H)/BW  
 Dose (water) = (Cw \* Iw)(H)/BW  
 Ci = Contaminant concentration in invertebrate  
 Cs = Contaminant concentration in sediment  
 Cw = Contaminant concentration in water  
 Total Dose = Dose (sediment) + Dose (invertebrate) + Dose (water)  
 H=HR/CA (Assume = to 1)

Conc = Concentration  
 LOAEL = Lowest Observed Adverse Effects Concentration  
 NOAEL = No Observed Adverse Effects Concentration  
 Sed = Sediment  
 SW = Surface Water  
 NV = No Value  
 #VALUE = Value could not be calculated

RACCOON - CONSERVATIVE INPUTS  
 TERRESTRIAL WILDLIFE MODEL ECOLOGICAL EFFECTS QUOTIENT CALCULATION  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT

Chemical	Max Sed Conc. (mg/kg)	Max SW Conc (mg/L)	Invertebrate Conc. (mg/kg)	Dose (mg/kg/d) from			Total Dose (mg/kg/d)	NOAEL (mg/kg/d)	LOAEL (mg/kg/d)	Hazard Quotients	
				Sediment	Surface Water	Invert				NOAEL	LOAEL
<b>Semivolatile Organics</b>											
1,4-DICHLORO BENZENE	4.20E-02	0.00E+00	9.44E-02	2.55E-04	0.00E+00	6.10E-03	6.35E-03	3.00E+01	3.00E+02	2.1E-04	2.1E-05
2-METHYLNAPHTHALENE	5.50E-02	0.00E+00	1.24E-01	3.34E-04	0.00E+00	7.99E-03	8.32E-03	4.10E+01	4.10E+02	2.0E-04	2.0E-05
ACENAPHTHENE	3.80E-01	0.00E+00	2.48E-01	2.31E-03	0.00E+00	1.60E-02	1.83E-02	1.75E+01	3.50E+01	1.0E-03	5.2E-04
ACENAPHTHYLENE	3.90E-01	0.00E+00	2.54E-01	2.37E-03	0.00E+00	1.64E-02	1.88E-02	7.00E+01	7.00E+02	2.7E-04	2.7E-05
ANTHRACENE	2.40E+00	0.00E+00	1.56E+00	1.46E-02	0.00E+00	1.01E-01	1.16E-01	1.00E+02	1.00E+03	1.2E-03	1.2E-04
BENZO(A)ANTHRACENE	2.70E+01	0.00E+00	1.76E+01	1.64E-01	0.00E+00	1.14E+00	1.30E+00	1.70E-01	1.70E+00	7.7E+00	7.7E-01
BENZO(A)PYRENE	3.50E+01	0.00E+00	2.28E+01	2.12E-01	0.00E+00	1.47E+00	1.69E+00	1.00E+00	1.00E+01	1.7E+00	1.7E-01
BENZO(B)FLUORANTHENE	5.50E+01	0.00E+00	3.59E+01	3.34E-01	0.00E+00	2.32E+00	2.65E+00	4.00E+00	4.00E+01	6.6E-01	6.6E-02
BENZO(G,H,I)PERYLENE	2.30E+01	0.00E+00	1.50E+01	1.40E-01	0.00E+00	9.68E-01	1.11E+00	7.20E+00	7.20E+01	1.5E-01	1.5E-02
BENZO(K)FLUORANTHENE	4.50E+01	0.00E+00	2.93E+01	2.73E-01	0.00E+00	1.89E+00	2.17E+00	7.20E+00	7.20E+01	3.0E-01	3.0E-02
CHRYSENE	4.20E+01	0.00E+00	2.74E+01	2.55E-01	0.00E+00	1.77E+00	2.02E+00	1.70E-01	1.70E+00	1.2E+01	1.2E+00
DIBENZO(A,H)ANTHRACENE	3.10E-01	0.00E+00	2.02E-01	1.88E-03	0.00E+00	1.31E-02	1.49E-02	1.33E+00	1.33E+01	1.1E-02	1.1E-03
FLUORANTHENE	8.00E+01	0.00E+00	5.22E+01	4.86E-01	0.00E+00	3.37E+00	3.85E+00	1.25E+01	2.50E+01	3.1E-01	1.5E-01
FLUORENE	1.00E+00	0.00E+00	6.52E-01	6.07E-03	0.00E+00	4.21E-02	4.82E-02	1.25E+01	2.50E+01	3.9E-03	1.9E-03
INDENO(1,2,3-CD)PYRENE	2.30E+01	0.00E+00	1.50E+01	1.40E-01	0.00E+00	9.68E-01	1.11E+00	7.20E+00	7.20E+01	1.5E-01	1.5E-02
NAPHTHALENE	7.70E-02	0.00E+00	5.02E-02	4.67E-04	0.00E+00	3.24E-03	3.71E-03	7.10E+00	1.42E+01	5.2E-04	2.6E-04
PENTACHLOROPHENOL	2.40E-01	0.00E+00	0.00E+00	1.46E-03	0.00E+00	0.00E+00	1.46E-03	8.42E+00	2.27E+01	1.7E-04	6.4E-05
PHENANTHRENE	3.60E+01	0.00E+00	2.35E+01	2.19E-01	0.00E+00	1.52E+00	1.73E+00	1.00E+00	1.00E+01	1.7E+00	1.7E-01
PYRENE	4.20E+01	0.00E+00	2.74E+01	2.55E-01	0.00E+00	1.77E+00	2.02E+00	7.50E+00	1.25E+01	2.7E-01	1.6E-01
<b>Pesticides/PCBs</b>											
4,4'-DDD	4.80E+00	0.00E+00	3.02E+00	2.91E-02	0.00E+00	1.95E-01	2.24E-01	1.47E-01	2.74E-01	1.5E+00	8.2E-01
4,4'-DDE	7.20E-01	0.00E+00	1.25E-01	4.37E-03	0.00E+00	8.05E-01	8.09E-01	1.47E-01	2.74E-01	5.5E+00	3.0E+00
4,4'-DDT	2.90E+00	0.00E+00	1.08E-01	1.76E-02	0.00E+00	7.03E-01	7.21E-01	1.47E-01	2.74E-01	4.9E+00	2.6E+00
ALDRIN	3.20E-03	0.00E+00	1.29E-02	1.94E-05	0.00E+00	8.36E-04	8.56E-04	2.00E-01	1.00E+00	4.3E-03	8.6E-04
ALPHA-CHLORDANE	2.90E-02	0.00E+00	3.11E-01	1.76E-04	0.00E+00	2.01E-02	2.03E-02	4.58E+00	9.16E+00	4.4E-03	2.2E-03
AROCLOL-1260	1.50E+00	0.00E+00	2.16E+02	9.11E-03	0.00E+00	1.40E+01	1.40E+01	6.80E-02	6.80E-01	2.1E+02	2.1E+01
BETA-BHC	2.70E-03	0.00E+00	1.09E-02	1.64E-05	0.00E+00	7.05E-04	7.22E-04	4.00E-01	2.00E+00	1.8E-03	3.6E-04
DELTA-BHC	4.20E-03	0.00E+00	1.70E-02	2.55E-05	0.00E+00	1.10E-03	1.12E-03	1.40E-02	1.40E-01	8.0E-02	8.0E-03
DIELDRIN	2.60E-02	0.00E+00	1.05E-01	1.58E-04	0.00E+00	6.79E-03	6.95E-03	1.50E-02	1.27E-00	4.6E-01	5.5E-03
ENDOSULFAN I	1.10E-02	0.00E+00	4.45E-02	6.68E-05	0.00E+00	2.87E-03	2.94E-03	1.50E-01	1.50E+00	2.0E-02	2.0E-03
ENDOSULFAN II	3.10E-02	0.00E+00	1.25E-01	1.88E-04	0.00E+00	8.10E-03	8.29E-03	1.50E-01	1.50E+00	5.5E-02	5.5E-03
ENDOSULFAN SULFATE	1.40E-02	0.00E+00	5.67E-02	8.50E-05	0.00E+00	3.66E-03	3.74E-03	1.50E-01	1.50E+00	2.5E-02	2.5E-03
ENDRIN	1.60E-02	0.00E+00	6.47E-02	9.71E-05	0.00E+00	4.18E-03	4.28E-03	9.20E-02	9.20E-01	4.7E-02	4.7E-03
ENDRIN ALDEHYDE	1.60E-02	0.00E+00	6.47E-02	9.71E-05	0.00E+00	4.18E-03	4.28E-03	9.20E-02	9.20E-01	4.7E-02	4.7E-03
ENDRIN KETONE	2.00E-02	0.00E+00	8.09E-02	1.21E-04	0.00E+00	5.23E-03	5.35E-03	9.20E-02	9.20E-01	5.8E-02	5.8E-03
GAMMA-BHC (LINDANE)	3.50E-03	0.00E+00	1.42E-02	2.12E-05	0.00E+00	9.15E-04	9.36E-04	8.00E+00	8.00E+01	1.2E-04	1.2E-05
GAMMA-CHLORDANE	2.30E-02	0.00E+00	1.15E-01	1.40E-04	0.00E+00	7.41E-03	7.55E-03	4.58E+00	9.16E+00	1.6E-03	8.2E-04
HEPTACHLOR	4.50E-03	0.00E+00	1.82E-02	2.73E-05	0.00E+00	1.18E-03	1.20E-03	1.00E-01	1.00E+00	1.2E-02	1.2E-03
HEPTACHLOR EPOXIDE	4.50E-03	0.00E+00	1.82E-02	2.73E-05	0.00E+00	1.18E-03	1.20E-03	1.00E-01	1.00E+00	1.2E-02	1.2E-03
METHOXYCHLOR	3.80E-02	0.00E+00	1.54E-01	2.31E-04	0.00E+00	9.93E-03	1.02E-02	4.00E+00	8.00E+00	2.5E-03	1.3E-03
<b>Inorganics</b>											
ARSENIC	1.41E+01	2.90E-03	9.73E+00	8.56E-02	3.70E-04	6.28E-01	7.14E-01	2.47E+00	4.55E+00	2.9E-01	1.6E-01
CADMIUM	6.10E+00	1.26E-01	4.87E-01	3.70E-02	1.61E-02	3.15E+00	3.20E+00	7.70E-01	6.90E+00	4.2E+00	4.6E-01
CHROMIUM	9.88E+01	6.80E-03	4.53E+01	5.88E-01	8.67E-04	2.93E+00	3.51E+00	2.40E+00	5.82E+01	1.5E+00	6.0E-02
COPPER	1.73E+02	2.93E-02	9.08E+02	1.05E+00	3.74E-03	5.87E+01	5.97E+01	5.82E+00	8.14E+01	1.0E+01	7.3E-01
LEAD	2.41E+02	7.80E-03	1.46E+02	1.46E+00	9.95E-04	9.45E+00	1.09E+01	4.70E+00	1.86E+02	2.3E+00	5.9E-02
MERCURY	1.20E+00	2.10E-04	3.44E+00	7.28E-03	2.68E-05	2.22E-01	2.30E-01	3.20E-02	1.60E-01	7.2E+00	1.4E+00
NICKEL	6.15E+01	8.47E-02	1.43E+02	3.73E-01	1.08E-02	9.21E+00	9.60E+00	1.70E+00	1.48E+01	5.6E+00	6.5E-01
SELENIUM	6.80E+00	0.00E+00	6.80E-00	4.13E-02	0.00E+00	4.39E-01	4.80E-01	2.00E-01	3.30E-01	2.4E+00	1.5E+00
SILVER	9.60E-01	0.00E+00	9.60E-01	5.83E-03	0.00E+00	6.20E-02	6.78E-02	6.02E-00	1.19E-02	1.1E-02	5.7E-04
ZINC	7.02E+02	3.34E-01	5.28E+03	4.26E+00	4.26E-02	3.41E+02	3.46E+02	1.60E+02	3.20E+02	2.2E+00	1.1E+00

Cells are shaded if the value is greater than 1.0

Body Weight = (BW)	3.67E+00	kg	Dose (sediment) = (Cs * Is)(H)/BW	Conc = Concentration
Food Ingestion Rate = (If)	2.37E-01	kg/day	Dose (invertebrate) = (Ci * If)(H)/BW	LOAEL = Lowest Observed Adverse Effects Concentration
Water Ingestion Rate = (Iw)	4.68E-01	L/day	Dose (water) = (Cw * Iw)(H)/BW	NOAEL = No Observed Adverse Effects Concentration
Sediment Ingestion Rate = (Is)	2.23E-02	kg/day	Ci = Contaminant concentration in invertebrate	Sed = Sediment
Home Range = (HR)	Assume 100% on site		Cs = Contaminant concentration in sediment	SW = Surface Water
Contaminated Area = (CA)	Assume equal to home range		Cw = Contaminant concentration in water	
			Total Dose = Dose (sediment) + Dose (invertebrate) + Dose (water)	
			H=HR/CA (Assume = to 1)	

RACCOON - AVERAGE INPUTS  
 TERRESTRIAL WILDLIFE MODEL ECOLOGICAL EFFECTS QUOTIENT CALCULATION  
 SITE 2B - AREA A WETLAND, NSB-NLON, GROTON, CONNECTICUT

Chemical	Avg Sed Conc. (mg/kg)	Avg SW Conc (mg/L)	Invertebrate Conc (mg/kg)	Dose (mg/kg/d) from			Total Dose (mg/kg/d)	NOAEL (mg/kg/d)	LOAEL (mg/kg/d)	Hazard Quotients	
				Sediment	Surface Water	Invert.				NOAEL	LOAEL
<b>Semivolatile Organics</b>											
1,4-DICHLOROBENZENE	4.20E-02	0.00E+00	9.44E-02	1.29E-04	0.00E+00	3.08E-03	3.21E-03	3.00E+01	3.00E+02	1.1E-04	1.1E-05
2-METHYLNAPHTHALENE	4.88E-02	0.00E+00	1.10E-01	1.50E-04	0.00E+00	3.58E-03	3.73E-03	4.10E+01	4.10E+02	9.1E-05	9.1E-06
ACENAPHTHENE	1.22E-01	0.00E+00	7.96E-02	3.75E-04	0.00E+00	2.60E-03	2.97E-03	1.75E+01	3.50E+01	1.7E-04	8.5E-05
ACENAPHTHYLENE	1.37E-01	0.00E+00	8.90E-02	4.19E-04	0.00E+00	2.91E-03	3.32E-03	7.00E+01	7.00E+02	4.7E-05	4.7E-06
ANTHRACENE	6.35E-01	0.00E+00	4.14E-01	1.95E-03	0.00E+00	1.35E-02	1.55E-02	1.00E+02	1.00E+03	1.5E-04	1.5E-05
BENZO(A)ANTHRACENE	1.58E+00	0.00E+00	1.03E+00	4.85E-03	0.00E+00	3.36E-02	3.85E-02	1.70E+01	1.70E+02	2.3E-01	2.3E-02
BENZO(A)PYRENE	1.90E+00	0.00E+00	1.24E+00	5.82E-03	0.00E+00	4.03E-02	4.62E-02	1.00E+00	1.00E+01	4.6E-02	4.6E-03
BENZO(B)FLUORANTHENE	2.41E+00	0.00E+00	1.57E+00	7.40E-03	0.00E+00	5.13E-02	5.87E-02	4.00E+00	4.00E+01	1.5E-02	1.5E-03
BENZO(G,H,I)PERYLENE	1.43E+00	0.00E+00	9.30E-01	4.38E-03	0.00E+00	3.04E-02	3.48E-02	7.20E+00	7.20E+01	4.8E-03	4.8E-04
BENZO(K)FLUORANTHENE	2.20E+00	0.00E+00	1.43E+00	6.74E-03	0.00E+00	4.68E-02	5.35E-02	7.20E+00	7.20E+01	7.4E-03	7.4E-04
CHRYSENE	2.13E+00	0.00E+00	1.39E+00	6.53E-03	0.00E+00	4.53E-02	5.18E-02	1.70E+01	1.70E+02	3.0E-01	3.0E-02
DIBENZO(A,H)ANTHRACENE	1.25E-01	0.00E+00	8.15E-02	3.84E-04	0.00E+00	2.66E-03	3.04E-03	1.33E+00	1.33E+01	2.3E-03	2.3E-04
FLUORANTHENE	3.41E+00	0.00E+00	2.22E+00	1.05E-02	0.00E+00	7.25E-02	8.30E-02	1.25E+01	2.50E+01	6.6E-03	3.3E-03
FLUORENE	5.96E-01	0.00E+00	3.88E-01	1.83E-03	0.00E+00	1.27E-02	1.45E-02	1.25E+01	2.50E+01	1.2E-03	5.8E-04
INDENO(1,2,3-CD)PYRENE	1.40E+00	0.00E+00	9.10E-01	4.28E-03	0.00E+00	2.97E-02	3.40E-02	7.20E+00	7.20E+01	4.7E-03	4.7E-04
NAPHTHALENE	6.78E-02	0.00E+00	4.42E-02	2.08E-04	0.00E+00	1.44E-03	1.65E-03	7.10E+00	1.42E+01	2.3E-04	1.2E-04
PENTACHLOROPHENOL	2.40E-01	0.00E+00	0.00E+00	7.37E-04	0.00E+00	0.00E+00	7.37E-04	8.42E+00	2.27E+01	8.7E-05	3.3E-05
PHENANTHRENE	1.84E+00	0.00E+00	1.20E+00	5.65E-03	0.00E+00	3.92E-02	4.48E-02	1.00E+00	1.00E+01	4.5E-02	4.5E-03
PYRENE	2.23E+00	0.00E+00	1.45E+00	6.83E-03	0.00E+00	4.74E-02	5.42E-02	7.50E+00	1.25E+01	7.2E-03	4.3E-03
<b>Pesticides/PCBs</b>											
4,4'-DDD	2.39E-01	0.00E+00	1.51E-01	7.34E-04	0.00E+00	4.92E-03	5.65E-03	1.47E-01	2.74E-01	3.8E-02	2.1E-02
4,4'-DDE	4.25E-02	0.00E+00	7.36E-01	1.30E-04	0.00E+00	2.40E-02	2.41E-02	1.47E-01	2.74E-01	1.6E-01	8.8E-02
4,4'-DDT	1.46E-01	0.00E+00	5.48E-01	4.48E-04	0.00E+00	1.79E-02	1.83E-02	1.47E-01	2.74E-01	1.2E-01	6.7E-02
ALDRIN	3.20E-03	0.00E+00	1.29E-02	9.82E-06	0.00E+00	4.23E-04	4.33E-04	2.00E-01	1.00E+00	2.2E-03	4.3E-04
ALPHA-CHLORDANE	2.45E-02	0.00E+00	2.62E-01	7.51E-05	0.00E+00	8.57E-03	8.64E-03	4.58E+00	9.16E+00	1.9E-03	9.4E-04
AROCLOR-1260	1.81E-01	0.00E+00	1.47E+01	5.54E-04	0.00E+00	4.80E-01	4.80E-01	6.80E-02	6.80E-01	7.1E+00	7.1E-01
BETA-BHC	2.70E-03	0.00E+00	1.09E-02	8.29E-06	0.00E+00	3.57E-04	3.65E-04	4.00E-01	2.00E+00	9.1E-04	1.8E-04
DELTA-BHC	4.20E-03	0.00E+00	1.70E-02	1.29E-05	0.00E+00	5.55E-04	5.68E-04	1.40E-02	1.40E-01	4.1E-02	4.1E-03
DIELDRIN	1.17E-02	0.00E+00	4.74E-02	3.59E-05	0.00E+00	1.55E-03	1.58E-03	1.50E-02	1.27E+00	1.1E-01	1.2E-03
ENDOSULFAN I	4.63E-03	0.00E+00	1.87E-02	1.42E-05	0.00E+00	6.11E-04	6.26E-04	1.50E-01	1.50E+00	4.2E-03	4.2E-04
ENDOSULFAN II	9.58E-03	0.00E+00	3.88E-02	2.94E-05	0.00E+00	1.27E-03	1.30E-03	1.50E-01	1.50E+00	8.6E-03	8.6E-04
ENDOSULFAN SULFATE	8.65E-03	0.00E+00	3.50E-02	2.65E-05	0.00E+00	1.14E-03	1.17E-03	1.50E-01	1.50E+00	7.8E-03	7.8E-04
ENDRIN	9.02E-03	0.00E+00	3.65E-02	2.77E-05	0.00E+00	1.19E-03	1.22E-03	9.20E-02	9.20E-01	1.3E-02	1.3E-03
ENDRIN ALDEHYDE	6.22E-03	0.00E+00	2.52E-02	1.91E-05	0.00E+00	8.21E-04	8.40E-04	9.20E-02	9.20E-01	9.1E-03	9.1E-04
ENDRIN KETONE	9.15E-03	0.00E+00	3.70E-02	2.81E-05	0.00E+00	1.21E-03	1.24E-03	9.20E-02	9.20E-01	1.3E-02	1.3E-03
GAMMA-BHC (LINDANE)	3.50E-03	0.00E+00	1.42E-02	1.07E-05	0.00E+00	4.62E-04	4.73E-04	8.00E+00	8.00E+01	5.9E-05	5.9E-06
GAMMA-CHLORDANE	8.79E-03	0.00E+00	4.39E-02	2.70E-05	0.00E+00	1.43E-03	1.46E-03	4.58E+00	9.16E+00	3.2E-04	1.6E-04
HEPTACHLOR	4.49E-03	0.00E+00	1.82E-02	1.38E-05	0.00E+00	5.93E-04	6.07E-04	1.00E-01	1.00E+00	6.1E-03	6.1E-04
HEPTACHLOR EPOXIDE	3.53E-03	0.00E+00	1.43E-02	1.08E-05	0.00E+00	4.66E-04	4.77E-04	1.00E-01	1.00E+00	4.8E-03	4.8E-04
METHOXYCHLOR	3.80E-02	0.00E+00	1.54E-01	1.17E-04	0.00E+00	5.02E-03	5.14E-03	4.00E+00	8.00E+00	1.3E-03	6.4E-04
<b>Inorganics</b>											
ARSENIC	6.55E+00	2.83E-03	9.36E-01	2.01E-02	2.34E-04	3.06E-02	5.09E-02	2.47E+00	4.55E+00	2.1E-02	1.1E-02
CADMIUM	1.66E+00	4.01E-02	9.96E-01	5.10E-03	3.31E-03	3.25E-02	4.09E-02	7.70E-01	6.90E+00	5.3E-02	5.9E-03
CHROMIUM	4.39E+01	6.80E-03	4.39E+00	1.35E-01	5.61E-04	1.43E-01	2.78E-01	2.40E+00	5.82E+01	1.2E-01	4.8E-03
COPPER	5.36E+01	1.35E-02	8.34E+01	1.64E-01	1.11E-03	2.72E+00	2.89E+00	5.82E+00	8.14E+01	5.0E-01	3.5E-02
LEAD	5.89E+01	3.82E-03	4.18E+00	1.81E-01	3.15E-04	1.38E-01	3.17E-01	4.70E+00	1.86E+02	6.8E-02	1.7E-03
MERCURY	2.95E-01	2.10E-04	3.36E-01	9.06E-04	1.73E-05	1.10E-02	1.19E-02	3.20E-02	1.60E-01	3.7E-01	7.4E-02
NICKEL	2.02E-01	4.58E-02	9.83E+00	6.20E-02	3.78E-03	3.21E-01	3.87E-01	1.70E+00	1.48E+01	2.3E-01	2.6E-02
SELENIUM	1.18E+00	0.00E+00	1.18E+00	3.62E-03	0.00E+00	3.85E-02	4.21E-02	2.00E-01	3.30E-01	2.1E-01	1.3E-01
SILVER	6.15E-01	0.00E+00	6.15E-01	1.89E-03	0.00E+00	2.01E-02	2.20E-02	6.02E+00	1.19E+02	3.6E-03	1.9E-04
ZINC	1.27E+02	1.33E-01	2.45E+02	3.89E-01	1.10E-02	8.01E+00	8.41E+00	1.60E+02	3.20E+02	5.3E-02	2.6E-02

Cells are shaded if the value is greater than 1.0

Body Weight = (BW)	5.64E+00	kg	Dose (sediment) = (Cs * Is)(H)/BW	Conc = Concentration
Food Ingestion Rate = (If)	1.84E-01	kg/day	Dose (invertebrate) = (Ci * Ii)(H)/BW	LOAEL = Lowest Observed Adverse Effects Concentration
Water Ingestion Rate = (Iw)	4.65E-01	l/day	Dose (water) = (Cw * Iw)(H)/BW	NOAEL = No Observed Adverse Effects Concentration
Sediment Ingestion Rate = (Is)	1.73E-02	kg/day	Ci = Contaminant concentration in invertebrate	Sed = Sediment
Home Range = (HR)	1.56E+03	acres	Cs = Contaminant concentration in sediment	SW = Surface Water
Contaminated Area = (CA)	Assume equal to home range		Cw = Contaminant concentration in water	
			Total Dose = Dose (sediment) + Dose (invertebrate) + Dose (water)	
			H=HR/CA (Assume = to 1)	

**APPENDIX D**

**SURFACE WATER TECHNICAL MEMORANDUM**

## SUMMARY OF SURFACE WATER SAMPLING PROGRAM FOR THE AREA A LANDFILL

This memorandum describes the surface water sampling portion of the Groundwater Monitoring Plan (GMP) for the Area A Landfill (Tetra Tech NUS, Inc. [TtNUS], 1999). This memorandum also describes various modifications to the surface water sampling portion of the program based on recommendations from the annual Groundwater Monitoring Reports (GMRs).

Details of the sampling program are presented in the GMP for the Area A Landfill (TtNUS, 1999). In summary, surface water monitoring began at Area A Landfill began in 1999, to complement the groundwater sampling occurring the Area A Landfill (Site 2A) and Wetland (Site 2B). The results of the monitoring program were used to evaluate the success of the Remedial Action (RA) (i.e., installation of a multi-layer cover system and a surface water/shallow groundwater interception and diversion system upgradient of the cover system) to minimize contaminant migration from Area A Landfill.

A total of 19 rounds of sample collection were completed by December 2006. Clean unpreserved sample collection containers are filled directly from the surface water bodies and transferred to the appropriate sample containers. Dissolved metal samples are collected using a clean unpreserved sample collection container and a peristaltic pump to filter the water from the collection container, through a 0.45-micron filter, and into the sample container.

Initially, surface water samples were collected from eleven locations. Sampling took place at ten staff gauges that were placed near newly installed monitoring wells and one seep location. The samples taken from these locations were analyzed for the same parameters as groundwater, namely Target Compound List (TCL) organic compounds, Target Analyte List (TAL) metals (total and dissolved) analytes, and water chemistry parameters. Sample collection originally occurred on a quarterly basis, defined as rounds. After Round 10, sampling then took place twice a year. Modifications/deviations to the sampling program are presented below on a yearly basis:

### **Year 1 - Rounds 1, 2, 3, and 4 (TtNUS, 2006):**

- Surface water samples were not collected at SG-15, SG-16, and SG-17 during Rounds 1 through 4, because there was no surface water at these locations.
- None of the surface water samples were collected during Round 2 because the surface water within the wetland was frozen.
- Surface water samples were not collected at SG-22 and SG-24 during Round 4 because the locations were dry.

Surface water sampling results for the initial four sample rounds indicated that phenanthrene and arsenic exceeded primary criteria. Chromium, copper, lead, and zinc also exceeded secondary criteria for the initial four rounds of surface water samples.

**Year 2 - Rounds 5, 6, 7, and 8 (TtNUS, 2006):**

- Surface water samples were not collected at SG-15, SG-16, and SG-17 during Rounds 5 through 8, because there was no surface water at these locations.
- During Round 8, surface water was not collected at SG-22, SG-23, and SG-24 because there was no surface water at these locations.
- Also during Round 8, due to a problem with sample shipment, the samples collected at SG-22 and SG-24 were not shipped on time, and the holding times for the organic parameters and the miscellaneous parameters were exceeded. Subsequently, these samples were only analyzed for TAL metals (total and dissolved).

Surface water sampling results for the second year of monitoring indicated that the following constituents exceeded primary criteria:

- Benzo(a)anthracene
- Benzo(a)pyrene
- Benzo(b)fluoranthrene
- Benzo(k)fluoranthrene
- Phenanthrene
- Arsenic
- Zinc

All of the exceedances of primary criteria with the exception of phenanthrene, arsenic, and zinc occurred at the seep sample location 3MSP01. Copper and lead also exceeded secondary criteria in several of the Year 2 surface water samples.

The Year 2 Annual GMR recommended the following:

- The sampling frequency should be reduced from quarterly to biannually because no significant increasing contaminant trends have been observed to date.
- TCL Volatile Organic Compounds (VOCs) and TCL pesticides/PCBs should be eliminated from the analytical program because these contaminants have not been identified as a concern.

- Surface water sample locations SG-15, -16, and -17 should be eliminated because surface water was not present at these locations during the first two years of monitoring activities.

### **Year 3 - Rounds 9, 10, and 11 (TtNUS, 2006):**

- Surface water samples were not collected at SG-15 and SG-17 during Rounds 9 and 10 because there was no surface water at these locations. Surface water was sampled at SG-16 during the Round 9 sampling event but not during Round 10. Because of the sporadic sampling at staff gauges SG-15 through SG-17, these sampling points were eliminated from the monitoring program after Round 10. No surface water samples were collected from staff gauge SG-22 during Round 9 through 11 because surface water was not present at this location.
- VOCs, pesticides, and PCBs were eliminated from the analytical program after Round 10.

Surface water sampling results for the third year of monitoring indicated that the following constituents exceeded primary criteria:

- Phenanthrene
- Arsenic
- Copper
- Lead
- Zinc

No additional COPCs were detected in surface water samples at concentrations that exceeded secondary criteria during Year 3.

The Year 3 Annual GMR recommended that SG-18, SG-22, and SG-24 should also be eliminated from the program.

### **Year 4 - Rounds 12 and 13 (TtNUS, 2006):**

Recommendations from the Year 3 GMR (i.e., the removal of staff gauges SG-18, SG-22, and SG-24) were not implemented during Year 4 because of a delay in finalizing Volume II – GMP of the O&M Manual.

Surface water sampling results for the fourth year of monitoring indicated that the following constituents exceeded primary criteria:

- Benzo(b)fluoranthene
- Benzo(k)fluoranthene

- Phenanthrene
- Arsenic
- Copper
- Lead
- Zinc

Chromium was detected at concentrations in surface water samples that exceeded secondary criteria during Year 4.

The conclusions of the final Year 4 Annual GMR were as follows:

- Thirteen of the 20 COPCs were detected in samples collected from seven surface water locations and one seep location.

#### **Year 5 - Rounds 14 and 15 (ECC, 2005):**

All proposed surface water samples were collected during these rounds of sampling.

Surface water sampling results for the fifth year of monitoring indicated that the following constituents exceeded primary criteria:

- Cadmium
- Copper
- Lead
- Zinc

The conclusions of the final Year 6 Annual GMR were as follows:

- Nineteen of the 20 COPCs were detected in samples collected from seven surface water locations and one seep location.

#### **Year 6 - Rounds 16 and 17 (ECC, 2006):**

All proposed surface water samples were collected during these rounds of sampling.

Surface water sampling results for the sixth year of monitoring indicated that the following constituents exceeded primary criteria:

- Cadmium
- Chromium
- Copper
- Lead

- Zinc

The conclusions of the final Year 6 Annual GMR were as follows:

- Nineteen of the 20 COPCs were detected in samples collected from seven surface water locations and one seep location.

#### **Year 7 - Rounds 18 and 19 (ECC, 2007):**

Beginning with Round 18 (August 2006), SG18, SG22, and SG24 were eliminated from the monitoring program; therefore, a total of four staff gauges (SG19, SG20, SG21, and SG23) and one surface seep (3MSP01) were sampled.

Surface water sampling results for the seventh year of monitoring indicated that the following constituents exceeded primary criteria:

- Total cadmium
- Total chromium
- Copper (Total and dissolved)
- Total lead
- Zinc (Total and dissolved)

The conclusions of the draft Year 7 Annual GMR were as follows:

- Seventeen of the 20 COPCs were detected in samples collected from four surface water locations and one seep location.
- Exceedances of the primary monitoring criteria occurred at all of the surface water locations.

## References

ECC. 2005. Year 5 Annual Groundwater Monitoring Report for Area A Landfall at Naval Submarine Base – New London, Groton, Connecticut. Marlborough, Massachusetts, August.

ECC. 2006. Year 6 Annual Groundwater Monitoring Report for Area A Landfall at Naval Submarine Base – New London, Groton, Connecticut. Marlborough, Massachusetts, July.

ECC. 2007. Draft Year 7 Annual Groundwater Monitoring Report for Area A Landfall at Naval Submarine Base – New London, Groton, Connecticut. Marlborough, Massachusetts, June.

TiNUS. 1999. Groundwater Monitoring Plan for Area A Landfill Naval Submarine Base – New London, Groton, Connecticut. King of Prussia, Pennsylvania, January.

TiNUS. 2006. Operations and Maintenance Manual for Installation Restoration Program Sites at Naval Submarine Base – New London, Groton, Connecticut, Volume II. King of Prussia, Pennsylvania, January.





SURFACE WATER ANALYTICAL DATA FOR THE AREA A LANDFILL MONITORING PROGRAM  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 3 OF 30

ROUND	01	01	01	01	01	01	01	01	03	03	03	03	03	03	03	03	04
LOCATION	SG-18	SG-19	SG-20	SG-21	SG-21	SG-22	SG-23	SG-24	SG-18	SG-19	SG-20	SG-20	SG-21	SG-22	SG-23	SG-24	SG-18
NSAMPLE	SWSG18-01	SWSG19-01	SWSG20-01	SWSG21-01	SWSG21-01-D	SWSG22-01	SWSG23-01	SWSG24-01	SWSG18-03	SWSG19-03	SWSG20-03	SWSG20-03-D	SWSG21-03	SWSG22-03	SWSG23-03	SWSG24-03	SWSG18-04
SAMPLE	SWSG18-01	SWSG19-01	SWSG20-01	SWSG21-01	SWFD-102499-01	SWSG22-01	SWSG23-01	SWSG24-01	SWSG18-03	SWSG19-03	SWSG20-03	SWFD04000-03	SWSG21-03	SWSG22-03	SWSG23-03	SWSG24-03	SWSG18-04
MATRIX	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW
QC TYPE	NM	NM	NM	NM	FD	NM	NM	NM	NM	NM	-NM	FD	NM	NM	NM	NM	NM
SAMPLE DATE	19991027	19991027	19991027	19991024	19991024	19991027	19991027	19991027	20000405	20000405	20000404	20000404	20000404	20000405	20000405	20000405	20000718
SILVER	13 U	13 U	13 U	13 U	13 U	11 U	11 U	11 U	11 U	11 U	11 U	11 U	11 U				
SODIUM	88700	89400	87100	58000	76100	41500	41600	36200	53800	59200	47100	47300	47900	33900	43000	178000	56600
THALLIUM	43 U	43 U	43 U	43 U	41 U	41 U	41 U	41 U	41 U	41 U	41 U	41 U	5 U				
VANADIUM	25	28	11 U	11 U	11 U	5	2.6	16 J	07 U	15 U	07 U	07 U	077 U	12 U	07 U	23 U	44 U
ZINC	31 J	32 J	13.1 J	26 U	59.2	90.2 J	119 J	48.3 J	31	48.9	16.3	16	15.2	14.6	27.9	20.6	27.8 J
<b>Filtered Inorganics (ug/L)</b>																	
ALUMINUM	62.3 U	56.6 U	127 U	56.6 U	56.6 U	56.6 U	135 U	144 U	72.5 U	72.5 U	72.5 U	72.5 U	72.5 U	72.5 U	72.5 U	72.5 U	69.1 U
ANTIMONY	2.4 U	2.4 U	2.4 U	2.4 U	2.1 U	2.1 U	2.1 U	2.1 U	2.1 U	2.1 U	2.1 U	2.1 U	2.6 U				
ARSENIC	3.8 U	3.8 U	5.6 J	3.8 U	2.6 U	2.6 U	2.6 U	2.6 U	2.6 U	2.6 U	2.6 U	2.6 U	2.7 U				
BARIUM	317	335	24.9 J	40.8	43.4	55.3	61.3	57.6	27.8	43.4	20	19.8	22.2	31.6	36.1	17.8	53.3
BERYLLIUM	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.1 U				
CADMIUM	0.3 U	0.3 U	0.34 U	0.3 U	0.3 U	0.3 U	0.3 U	0.3 U	0.3 U	0.3 U	0.3 U	0.3 U	0.33 U				
CALCIUM	14200	15600	15000	27900	29400	39700	29700	9910	18500	24200	14500	14400	15700	20000	22800	16300	29100
CHROMIUM	2.4 U	2.4 U	2.4 U	2.4 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1.5 U	1.3 U				
COBALT	21 U	21 U	7.5	11.1	0.88 U	0.7 U	0.7 U	0.7 U	0.7 U	1.8 U	4.2 U	18.4	0.94 U				
COPPER	1.2 U	1.2 U	1.2 U	1.7 U	1.8 U	1.2 U	1.2 U	1.14	1.3 U	2.5 U	1.3 U	1.4 U	1.3 U	2.8 U	1.5 U	2 U	0.87 U
IRON	11600	2440	3070	5910	5980	17900	24100	3100	493	6210	227	219	239	15700	14600	32000	21700
LEAD	17 U	2.1 U	4.6 U	2.9 U	1.8 U	1.8 U	1.8 U	1.8 U	1.8 U	1.8 U	1.8 U	1.8 U	1.9 U				
MAGNESIUM	3480	3860	3740	5110	5500	5390	6540	3900	4270	5290	3890	3910	3950	3480	4550	23500	5560
MANGANESE	346	351	317	518	536	940	1400	2720	222	201	42.4	41.9	85.7	404	657	3590	794
MERCURY	0.1 UJ	0.1 UJ	0.1 UJ	0.2 J	0.13 J	0.1 UJ	0.1 UJ	0.1 UJ	0.17 U	0.17 U	0.17 U	0.17 U	0.17 U	0.17 U	0.17 U	0.17 U	0.1 U
MOLYBDENUM																	
NICKEL	2.6 U	3.7 J	9.6	10.9	2.5 J	1.9 U	1.9 U	1.9 U	1.9 U	3.7 J	3.1 J	8.9	1.5 U				
POTASSIUM	4290	4550	4440	5410 J	5910 J	3920	3720	5030	3280	4030	2960	3020	3140	3360	4170	9400	4740
SELENIUM	4.3 UJ	4.3 UJ	4.3 UJ	4.3 U	4.3 U	4.3 UJ	4.3 UJ	4.3 UJ	3.4 U	3.4 U	3.4 U	3.4 U	3.4 U	3.4 U	3.4 U	3.4 U	3.4 UJ
SILVER	13 U	13 U	1.4 J	13 U	13 U	13 U	3	13 U	11 U	11 U	11 U	11 U	11 U	11 U	11 U	11 U	11 U
SODIUM	91200	92600	89500	77900 J	91400 J	40300	46500	36900	50200	64200	47200	47300	48600	32500	46900	191000	56800
THALLIUM	43 U	43 U	43 U	43 U	41 U	41 U	41 U	41 U	41 U	41 U	41 U	41 U	5 U				
VANADIUM	1.8 J	1.1 U	1.1 U	1.1 U	1.1 U	1.1 U	1.1 U	1.1 U	0.7 U	0.7 U	0.7 U	0.7 U	0.7 U	0.7 U	0.85 U	1.1 U	1.9 U
ZINC	49.4 J	118 J	47.3 J	24.8 U	110	37 J	96.5 J	70.7 J	33.8	17.9	16.9	19.2	21.9	10.2	24	28.4	4.1 U
<b>Miscellaneous Parameters</b>																	
ALKALINITY (MG/L)	46.6	48.6	49.2	80.2	70.4	134	99.4	24.3	47.9	66.4	24	33.2	34.9	74	78.4	40.3	80.8
CARBONATE ALKALINITY (MG/L)																	
CHEMICAL OXYGEN DEMAND (MG/L)	26.4	26.4	23.8	21.1	20 U	42.2	58.1	55.4	20 U	20 U	20 U	20 U	20 U	31.4	20 U	21	50.6
CHLORIDE (MG/L)	164	162	161	116	129	64.2	89.1	78	95.5 J	121 J	87.2 J	87.9 J	89 J	53.4 J	89.2 J	398 J	105 J
HARDNESS (MG/L)	99.5	115	95.7	125	86.5	140	172	115	67	78.4	51.9	52	54.4	70.6	68.2	131	94
HYDROGEN (NMO/L)																	
SULFATE (MG/L)	10 U	14.5	14.5	12.1	12.5	10 U	10 U	10 U	20 U	20 U	20.8	20.9	20.5	20 U	20 U	20 U	20 U
SULFIDE (MG/L)																	
TOTAL DISSOLVED SOLIDS (MG/L)	268	32	296	315	326	268	267	201	202	248	211	224	193	179	280	643	302 J
TOTAL ORGANIC CARBON (MG/L)	7.5	7.2	6.3	5.4	5.6	9	10.3	14.2	3.4	3.9	4.5	3.3	4.5	6.2	3.3	9.2	9.7
TOTAL SUSPENDED SOLIDS (MG/L)																	105 J
<b>Field Parameters</b>																	
DISSOLVED OXYGEN (MG/L)																	
DISSOLVED OXYGEN - METER (MG/L)	3.81	5.05	4.95	1.8		4.35	4.01	6.46	1.89	2.04	6.61		7.68	0.65	5.61	1.12	1.18
MANGANESE (MG/L)																	
OXIDATION REDUCTION POTENTIAL (MV)	-24	23.3	7.4	46		-50	-19	-29	-221	-219	163		145	-57	-246	-176	-161
PH	6.45	6.62	6.63	6.68		6.39	6.16	7.32	7.7	8.11	6.96		7.74	6.9	7.84	6.8	6.64
SALINITY (NG/L)	0.31	0.31	0.31	0.28		0.27	0.24	0.19	0.21	0.26	0.17		0.19	0.17	0.3	2.54	0 U
SPECIFIC CONDUCTANCE (MS/CM)	0.639	0.628	0.624	0.568		0.559	0.5	0.403	0.442	0.536	0.36		0.401	0.36	0.617	4.5	0.734
TEMPERATURE (C)	10.1	10.5	11	9.4		14	11	12.7	9	9.7	11.8		12.1	8.6	6	13.7	19
TURBIDITY (NTU)	6.1	6.2	6.3	6.3		30.2	6.8	3.9	5.3	2.2	7.1		7	10.3	6.3	10.1	18

SURFACE WATER ANALYTICAL DATA FOR THE AREA A LANDFILL MONITORING PROGRAM  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 4 OF 30

ROUND	04	04	04	04	04	05	05	05	05	05	05	05	05	06	06	06	06
LOCATION	SG-18	SG-19	SG-20	SG-21	SG-23	SG-18	SG-19	SG-20	SG-21	SG-22	SG-23	SG-24	SG-24	SG-18	SG-19	SG-19	SG-20
NSAMPLE	SWSG18-04-D	SWSG19-04	SWSG20-04	SWSG21-04	SWSG23-04	SWSG18-05	SWSG19-05	SWSG20-05	SWSG21-05	SWSG22-05	SWSG23-05	SWSG24-05	SWSG24-05-D	SWSG18-06	SWSG19-06	SWSG19-06-D	SWSG20-06
SAMPLE	SWFD07180004	SWSG19-04	SWSG20-04	SWSG21-04	SWSG23-04	SWSG18-05	SWSG19-05	SWSG20-05	SWSG21-05	SWSG22-05	SWSG23-05	SWSG24-05	FD-SW-121800-01	SWSG18-06	SWSG19-06	SWFD031201-01	SWSG20-06
MATRIX	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW
QC TYPE	FD	NM	FD	NM	NM	FD	NM										
SAMPLE DATE	20000718	20000719	20000719	20000719	20000719	20001218	20001219	20001219	20001219	20001219	20001219	20001219	20001218	20010313	20010312	20010312	20010313
CONTRACTOR	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN
Volatiles (ug/L)																	
1,2,4-TRICHLOROENZENE																	
2-BUTANONE	5 U	2 J	5 U	5 U	5 U	5 UR	5 UR	5 UJ	5 UJ	5 UR							
ACETONE	4 J	6	5 U	15	4 J	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U
BENZENE	1 U	0.4 J	1 U	1 U	1 U	1 U	0.14 J	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
CARBON DISULFIDE	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	0.26 J	0.18 J	1 U	1 U	1 U	1 U
CHLOROBENZENE	1 U	1 U	1 U	1 U	1 U	1 U	0.17 J	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
CHLOROMETHANE	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
ETHYLBENZENE	1 U	0.1 J	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
METHANE																	
METHYLENE CHLORIDE	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U
TETRACHLOROETHENE	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
TOLUENE	2	20	2	9	2	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
TOTAL XYLENES	1 U	0.57 J	1 U	1 U	1 U	1 U	0.21 J	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
TRICHLOROETHENE	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
Semivolatile Organics (ug/L)																	
1-METHYLNAPHTHALENE																	
2,4-DIMETHYLPHENOL	5 U	5.1 U	5 U	5.2 U	5.1 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U
2-METHYLNAPHTHALENE	2 U	0.89 J	2 U	2.1 U	2 U												
2-METHYLPHENOL	5 U	5.1 U	5 U	5.2 U	5.1 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U
3&4-METHYLPHENOL	5 U	5.1 U	5 U	5.2 U	5.1 U												
4-METHYLPHENOL						10 U	10 U	10 U	10 U	10 U							
4-NITROANILINE	5 U	5.1 U	5 U	5.2 U	5.1 U	50 U	50 U	50 U	50 U	50 U	50 U	50 U	50 U	50 UJ	50 UJ	50 UJ	50 UJ
ACENAPHTHENE	0.2 UJ	1.3 J	0.2 UJ	0.21 UJ	0.2 UJ	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	0.21 J	1 U
ACENAPHTHYLENE	0.2 UJ	0.2 UJ	0.2 UJ	0.21 UJ	0.2 UJ	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
ANTHRACENE	0.2 UJ	0.2 UJ	0.2 UJ	0.21 UJ	0.2 UJ	0.2 U	0.037 J	0.2 U	0.2 U	0.2 U	0.2 U	0.3 J	0.16 J	0.2 U	0.2 U	0.2 U	0.2 U
BENZO(A)ANTHRACENE	0.2 UJ	0.2 UJ	0.2 UJ	0.21 UJ	0.2 UJ	0.2 U	0.2 U	0.2 U	0.029 J	0.2 U	0.2 U	0.093 J	0.093 J	0.061 J	0.2 U	0.2 U	0.037 J
BENZO(A)PYRENE	0.2 UJ	0.2 UJ	0.2 UJ	0.21 UJ	0.2 UJ	0.2 U	0.2 U	0.2 U	0.026 J	0.2 U	0.2 U	0.05 J	0.039 J	0.11 J	0.2 U	0.022 J	0.066 J
BENZO(B)FLUORANTHENE	0.2 UJ	0.2 UJ	0.2 UJ	0.21 UJ	0.2 UJ	0.2 U	0.2 U	0.2 U	0.037 J	0.2 U	0.2 U	0.059 J	0.053 J	0.2 U	0.2 U	0.2 U	0.2 U
BENZO(G,H,I)PERYLENE	0.2 UJ	0.2 UJ	0.2 UJ	0.21 UJ	0.2 UJ	0.2 U	0.2 U	0.2 U	0.029 J	0.023 J	0.2 U	0.2 U	0.2 U	0.2 J	0.2 U	0.031 J	0.11 J
BENZO(K)FLUORANTHENE	0.2 UJ	0.2 UJ	0.2 UJ	0.21 UJ	0.2 UJ	0.2 U	0.028 J	0.026 J	0.083 J	0.2 U	0.2 U	0.05 J					
BENZOIC ACID	20 UR	20 UR	20 UR	21 UR	20 UR	50 UJ	50 UJ	50 UJ	50 UJ	50 UJ							
BIS(2-ETHYLHEXYL)PHTHALATE	2 U	2 U	2 U	2.1 U	2 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U
BUTYL BENZYL PHTHALATE	2 U	2 U	2 U	2.1 U	2 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U
CARBAZOLE	2 U	2 U	2 U	2.1 U	2 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U
CHRYSENE	0.2 UJ	0.2 UJ	0.2 UJ	0.21 UJ	0.2 UJ	0.2 U	0.2 U	0.2 U	0.026 J	0.2 U	0.2 U	0.13 J	0.11 J	0.16 J	0.2 U	0.022 J	0.095 J
DI-N-BUTYL PHTHALATE	2 U	2 U	2 U	2.1 U	2 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U
DI-N-OCTYL PHTHALATE	2 U	2 U	2 U	2.1 U	2 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U
DIBENZO(A,H)ANTHRACENE	0.2 UJ	0.2 UJ	0.2 UJ	0.21 UJ	0.2 UJ	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U							
DIETHYL PHTHALATE	2 U	2 U	2 U	2.1 U	2 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U
FLUORANTHENE	0.2 UJ	0.2 UJ	0.2 UJ	0.21 UJ	0.2 UJ	0.03 J	0.11 J	0.03 J	0.064 J	0.065 J	0.038 J	0.38	0.36	0.42	0.2 U	0.074 J	0.26
FLUORENE	0.2 UJ	0.63 J	0.2 UJ	0.21 UJ	0.2 UJ	0.031 J	0.57	0.2 U	0.2 U	0.2 U	0.093 J	0.2 U					
INDENO(1,2,3-CD)PYRENE	0.2 UJ	0.2 UJ	0.2 UJ	0.21 UJ	0.2 UJ	0.2 U	0.2 U	0.2 U	0.022 J	0.2 U	0.2 U	0.036 J	0.2 U	0.091 J	0.2 U	0.2 U	0.054 J
NAPHTHALENE	0.2 UJ	5 J	0.2 UJ	0.21 UJ	0.2 UJ	1 U	3.6	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	0.42 J	1 U
PHENANTHRENE	0.2 UJ	0.16 J	0.2 UJ	0.21 UJ	0.2 UJ	0.037 J	0.39	0.024 J	0.043 J	0.042 J	0.031 J	0.19 J	0.14 J	0.21	0.2 U	0.067 J	0.14 J
PHENOL	5 UJ	5.1 UJ	5 UJ	5.2 UJ	5.1 UJ	10 U	10 U	10 U	10 U	10 U							
PYRENE	0.2 UJ	0.2 UJ	0.2 UJ	0.21 UJ	0.2 UJ	0.2 U	0.2 U	0.2 U	0.047 J	0.043 J	0.022 J	0.26	0.26	0.3	0.2 U	0.051 J	0.18 J

SURFACE WATER ANALYTICAL DATA FOR THE AREA A LANDFILL MONITORING PROGRAM  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 5 OF 30

ROUND	04	04	04	04	04	05	05	05	05	05	05	05	05	06	06	06	06
LOCATION	SG-18	SG-19	SG-20	SG-21	SG-23	SG-18	SG-19	SG-20	SG-21	SG-22	SG-23	SG-24	SG-24	SG-18	SG-19	SG-19	SG-20
NSAMPLE	SWSG18-04-D	SWSG19-04	SWSG20-04	SWSG21-04	SWSG23-04	SWSG18-05	SWSG19-05	SWSG20-05	SWSG21-05	SWSG22-05	SWSG23-05	SWSG24-05	SWSG24-05-D	SWSG18-06	SWSG19-06	SWSG19-06-D	SWSG20-06
SAMPLE	SWFD07180004	SWSG19-04	SWSG20-04	SWSG21-04	SWSG23-04	SWSG18-05	SWSG19-05	SWSG20-05	SWSG21-05	SWSG22-05	SWSG23-05	SWSG24-05	FD-SW-121800-01	SWSG18-06	SWSG19-06	SWSG19-06-D	SWSG20-06
MATRIX	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW
QC TYPE	FD	NM	FD	NM	NM	FD	NM										
SAMPLE DATE	20000718	20000719	20000719	20000719	20000719	20001218	20001219	20001219	20001219	20001219	20001219	20001219	20001218	20010313	20010312	20010312	20010313
<b>Polynuclear Aromatic Hydrocarbons (ug/L)</b>																	
2-METHYLNAPHTHALENE																	
ACENAPHTHENE																	
ACENAPHTHYLENE																	
ANTHRACENE																	
BAP EQUIVALENT																	
BENZO(A)ANTHRACENE																	
BENZO(A)PYRENE																	
BENZO(B)FLUORANTHENE																	
BENZO(G,H,I)PERYLENE																	
BENZO(K)FLUORANTHENE																	
CHRYSENE																	
DIBENZO(A,H)ANTHRACENE																	
FLUORANTHENE																	
FLUORENE																	
INDENO(1,2,3-CD)PYRENE																	
NAPHTHALENE																	
PHENANTHRENE																	
PYRENE																	
<b>Pesticides/PCBs (ug/L)</b>																	
4,4'-DDD	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.011 J	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U
4,4'-DDE	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U
4,4'-DDT	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U
ALDRIN	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	-0.01 U	0.01 U	0.01 U
ALPHA-CHLORDANE	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U
ENDOSULFAN I	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U
ENDOSULFAN II	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U
ENDRIIN ALDEHYDE	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U	0.02 U
GAMMA-CHLORDANE	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U	0.01 U
<b>Total Inorganics (ug/L)</b>																	
ALUMINUM	500	633	196	69.1 U	700	47.6 U	26.8 U	58.3 U	250 U	48.9 U	12.9 U	23.9 U	12.9 U	523	26.5 U	27.4 U	313 U
ANTIMONY	2.6 U	2.6 U	2.6 U	2.6 U	2.6 U	1.9 U	1.8 U	2.1 U	1.8 U	3.2 U	3.2 U	3.2 U	3.2 U				
ARSENIC	2.7 U	2.7 U	2.7 U	2.7 U	3 J	2.2 U	2.7 J	2.6 U	2.6 U	3 J							
BARIUM	60.3	108	26.8 J	59.6	40.8	24.7	43.3	17.7	23.8	43.4	12.7	18.8	18.6	20.2	32.3	33.7	15.4
BERYLLIUM	0.1 U	0.14 U	0.2 U	0.1 U	0.11 U	0.26 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 U	0.42	0.13 U	0.13 U	0.17 J
CADMIUM	0.32 U	0.49 U	0.34 U	0.32 U	0.48 U	0.2 U	0.2 U	0.2 U	0.28 U	0.2 U	0.2 U	0.26 U	0.31 U	2.6 U	2.6 U	2.6 U	2.6 U
CALCIUM	29400	45900	15700	33900	22700	10900	20300	7690	10300	17300	4050	3490	3630	9090	17400	18000	7110
CHROMIUM	1.3 U	1.3 U	1.3 U	1.3 U	1.3 U	0.5 U	2.6 U	2.6 U	2.6 U	2.6 U							
COBALT	1.2 U	0.98 U	1.2 U	1.5 U	1.9 U	0.6 U	0.6 U	0.6 U	0.6 U	0.8 U	0.6 U	0.85 U	0.8 U	4.1 U	4.1 U	4.1 U	4.1 U
COPPER	2 U	5.2 U	1.6 U	0.87 U	6.2	1.8 J	2.2	1 U	2.2	3.5	1 U	5	2.7	2 U	2 U	2 U	2.1 J
IRON	24700	36900	3470	13400	7860	1500	6120	1050	2000	2160	450	480	446	949	2120	2300	784
LEAD	2 J	3.4 J	2.3 J	1.9 U	5 J	1.6 U	2.1 U	1.3 U	5 U	2.9 U	1.3 U	1.8 U	1.5 U	2 U	2 U	2 U	2 U
MAGNESIUM	5730	8090	3950	5440	5070	2710	3550	2240	2450	2990	771	935	939	1630	2920	3030	1230
MANGANESE	804	1170	346	1670	355	154	230	111	191	401	66.3	64.5	70.6	146	188	193	125
MERCURY	0.22	0.38	0.1 U	0.079 UR	0.08 U	0.08 U	0.079 UR										
MOLYBDENUM																	
NICKEL	1.9 U	2.8 U	1.3 U	1.2 U	6.4	2.3 J	2.9 J	2.1 U	2.1 U	2.7 J	2.1 U	6.9	2.2 J	9.8 U	9.8 U	9.8 U	9.8 U
POTASSIUM	4760	5790	2750	3950	2950	4190	4780	3910	4200	3550	2080	1760	1920	1770	2470 U	2700 U	2170
SELENIUM	3.4 UJ	3.4 UJ	3.4 UJ	3.4 UJ	3.4 UJ	2.9 UJ	3.8 J	2.9 UJ	3.8 UJ	3.8 UJ	3.8 UJ	3.8 UJ					

SURFACE WATER ANALYTICAL DATA FOR THE AREA A LANDFILL MONITORING PROGRAM  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 6 OF 30

ROUND	04	04	04	04	04	05	05	05	05	05	05	05	05	06	06	06	06
LOCATION	SG-18	SG-19	SG-20	SG-21	SG-23	SG-18	SG-19	SG-20	SG-21	SG-22	SG-23	SG-24	SG-24	SG-18	SG-19	SG-19	SG-20
NSAMPLE	SWSG18-04-D	SWSG19-04	SWSG20-04	SWSG21-04	SWSG23-04	SWSG18-05	SWSG19-05	SWSG20-05	SWSG21-05	SWSG22-05	SWSG23-05	SWSG24-05	SWSG24-05-D	SWSG18-06	SWSG19-06	SWSG19-06-D	SWSG20-06
SAMPLE	SWFD07180004	SWSG19-04	SWSG20-04	SWSG21-04	SWSG23-04	SWSG18-05	SWSG19-05	SWSG20-05	SWSG21-05	SWSG22-05	SWSG23-05	SWSG24-05	FD-SW-121800-01	SWSG18-06	SWSG19-06	SWFD031201-01	SWSG20-06
MATRIX	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW
QC TYPE	FD	NM	FD	NM	NM	FD	NM										
SAMPLE DATE	20000718	20000719	20000719	20000719	20000719	20001218	20001219	20001219	20001219	20001219	20001219	20001219	20001218	20010313	20010312	20010312	20010313
SILVER	1.1 U	1.1 U	1.1 U	1.1 U	1.1 U	0.9 U	0.9 U	0.9 U	0.9 U	0.9 U							
SODIUM	57100	84700	47400	60200	47000	52300	52700	39800	35600	26700	3730	5790	6440	48600 J	59200 J	61200 J	33900 J
THALLIUM	5 U	5 U	5 U	5 U	5 U	7.1 UJ	6.1 J	6 U	6 U	6 UJ							
VANADIUM	4.3 U	12.6	1.1 U	0.71 U	4.6 U	0.99 U	0.6 U	0.6 U	0.93 U	0.73 U	0.6 U	1.2 U	1 U	4 U	2.6 U	2.6 U	7.2 U
ZINC	31.8 J	49.6 J	11.3 J	18.3 J	65.2 J	72.5	74.6	52.2	86.3	217	240	115	120	64.5	32.9	38	35.1
Filtered Inorganics (ug/L)																	
ALUMINUM	69.1 U	69.1 U	69.1 U	69.1 U	69.1 U	12.9 U	40.1 U	16 U	14.7 U	34 U							
ANTIMONY	2.6 U	2.6 U	2.6 U	2.6 U	2.6 U	1.8 U	1.8 U	1.8 U	2.3 U	2 U	3 U	1.8 U	1.8 U	3.2 U	3.2 U	3.2 U	3.2 U
ARSENIC	2.7 U	2.7 U	2.7 U	2.7 U	2.7 U	2.2 U	2.6 U	2.6 UJ	2.6 U	2.6 U							
BARIUM	54.6	108	23.2 J	53	31.8 J	22	40	15.9	23	42.8	12.9	16.9	16.7	16.7	36.7	34.2	14.5
BERYLLIUM	0.1 U	0.16 U	0.1 U	0.1 U	0.1 U	0.2 U	0.2 U	0.2 U	0.36 U	0.2 U	0.2 U	0.2 U	0.2 U	0.32	0.13 U	0.13 U	0.15 J
CADMIUM	0.32 U	0.44 U	0.32 U	0.32 U	0.32 U	0.2 U	0.2 U	0.2 U	0.37 U	0.2 U	0.2 U	0.2 U	0.2 U	2.6 U	2.6 U	2.6 UJ	2.6 U
CALCIUM	29500	49600	15400	32200	23000	10500	19500	7880	11000	18600	4870	3470	3600	8970	20200	18700	7490
CHROMIUM	1.3 U	1.3 U	1.3 U	1.3 U	1.3 U	0.5 U	2.6 U	2.6 U	2.6 U	2.6 U							
COBALT	0.98 U	1.6 U	1.7 U	1.2 U	1.8 U	0.6 U	0.6 U	0.6 U	0.69 U	1 U	0.6 U	0.6 U	0.6 U	4.1 U	4.1 U	4.1 U	4.1 U
COPPER	0.87 U	0.87 U	0.87 U	0.87 U	0.87 U	1 U	1 U	1.5 J	1 U	1 U	1.5 J	1.8 J	2 U	2 U	2 U	2 U	2 U
IRON	21900	33000	2770	7090	5090	705	5160	517	1380	489	268	150 U	185 U	152	2270	2110	226
LEAD	1.9 U	1.9 U	1.9 U	1.9 U	1.9 U	1.5 U	1.3 U	1.2 U	1 UJ	1 UJ	1.1 U	1.5 U	1.2 U	2 UJ	2 U	2 U	2 UJ
MAGNESIUM	5650	8460	3820	5160	5020	2590	3380	2300	2660	3170	907	892	939	1650	3550	3010	1270
MANGANESE	802	1210	331	1670	354	145	221	110	202	432	79.4	64	71.7	130	213	200	124
MERCURY	0.16 J	0.25	0.1 U	0.1 U	0.33	0.1 U	0.1 U	0.15 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.079 U	0.08 U	0.08 U	0.079 UR
MOLYBDENUM																	
NICKEL	1.2 U	1.2 U	1.5 U	1.2 U	4.5 U	2.1 J	2.3 J	2.1 U	2.1 J	2.5 J	3.8 J	2.1 U	2.1 U	9.8 U	9.8 U	9.8 U	9.8 U
POTASSIUM	4720	6160	2740	3670	2750	4040	4530	4100	4590	3750	2520	1830	1920	1830	3310 U	2550 U	1840
SELENIUM	3.4 UJ	3.4 UJ	3.4 UJ	3.4 UJ	3.4 UJ	2.9 UJ	3.8 U	3.8 UJ	3.8 UJ	3.8 U							
SILVER	1.1 U	1.1 U	1.1 U	1.1 U	1.1 U	0.9 U	2.7 U	2.1 U	2.1 U	2.7 U							
SODIUM	57800	90600	46700	59100	48700	50400	50200	41600	38800	28400	4800	6050	6600	47100 J	65400 J	59200 J	37100 J
THALLIUM	5 U	5 U	5 U	5 U	5 U	7.1 UJ	6 UJ	6 U	6 U	6 UJ							
VANADIUM	1.3 U	3.2 U	1.3 U	0.71 U	0.71 U	0.6 U	0.6 U	0.6 U	0.72 U	0.6 U	0.6 U	0.61 U	0.65 U	3.3 U	2.6 U	2.6 U	3.7 U
ZINC	7.6	25.2	24.4	13.9	6.4	72.4	31.8 U	32.7 U	34.5 U	204	290	115	114	56.7	37.1 J	35.8 J	25.3
Miscellaneous Parameters																	
ALKALINITY (MG/L)	71.8	144	40.1	95.6	35.9	21.2	53	14.8	17	42.4	10.6	6.4	6.4	13.4	35.7	40.1	11.2
CARBONATE ALKALINITY (MG/L)																	
CHEMICAL OXYGEN DEMAND (MG/L)	40.5	58.2	38	50.6	101	15.6	22.6	26.1	15.6	10 U	10 U	10 U	10 U	10 U	13.8	10 U	10 U
CHLORIDE (MG/L)	104 J	160 J	82.9 J	113 J	71.9 J	90.5	92.3	68.6	57	48	8	9.2	9.1	87.6	116	116	66.3
HARDNESS (MG/L)	97	150	56	110	78	38.4	65.3	28.4	35.8	55.5	13.3	12.6	12.9	29.4	55.5	57.3	22.8
HYDROGEN (NMO/L)																	
SULFATE (MG/L)	20 U	20 U	20 U	20 U	45.5	8.2	7.8	9.4	8.5	10.3	3.2	6.9	6.7	6.6	10.5	10.8	5.7
SULFIDE (MG/L)																	
TOTAL DISSOLVED SOLIDS (MG/L)	275 J	452 J	212 J	356 J	256 J	198	248	167	145	154	41	43	57	160	240 J	240 J	137
TOTAL ORGANIC CARBON (MG/L)	9.9	12.6	6.1	11.7	8.9	4.5	3.4	3.6	3.7	2.4	1.8	1.6	1.2	1.1 U	1.8 U	1.4 U	1.6 J
TOTAL SUSPENDED SOLIDS (MG/L)	71 J	141 J	24 J	51 J	108 J												
Field Parameters																	
DISSOLVED OXYGEN (MG/L)																	
DISSOLVED OXYGEN - METER (MG/L)		0.7	0.57	0.43	0.79	3.65	1.01	3.44	2.29	1.96	4.67	9.68		12.41	8.59		7.29
MANGANESE (MG/L)																	
OXIDATION REDUCTION POTENTIAL (MV)		-192	-101	-172	-82	-53.7	126	-17.2	-28.4	-10.5	-0.6	-211		104	-66		39
PH		6.56	6.28	6.65	6.12	7.34	6.81	6.98	6.53	6.69	6.57	9.29		6.57	6.56		6.67
SALINITY (NG/L)		0.1	0 U	0 U	0 U									0.13	0.19		0.18
SPECIFIC CONDUCTANCE (MS/CM)		1.26	0.422	0.599	0.65	0.332	0.395	0.267	0.253	0.258	0.055	0.371		0.285	0.389		0.38
TEMPERATURE (C)		18.5	18	17.9	16.7	3.88	2.83	1.99	2.33	3.68	2.57	3.22		1	2.8		1.7
TURBIDITY (NTU)		6.6	6.3	14.9	13.2	11	8.4	7.3	6.1	7	0 U	4.88		11	2.9		12

SURFACE WATER ANALYTICAL DATA FOR THE AREA A LANDFILL MONITORING PROGRAM  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 7 OF 30

ROUND	06	06	06	06	07	07	07	07	07	07	07	07	07	08	08	08	08	09	09
LOCATION	SG-21	SG-22	SG-23	SG-24	SG-18	SG-19	SG-20	SG-21	SG-22	SG-22	SG-23	SG-24	SG-18	SG-19	SG-20	SG-21	SG-16	SG-18	
NSAMPLE	SWSG21-06	SWSG22-06	SWSG23-06	SWSG24-06	SWSG18-07	SWSG19-07	SWSG20-07	SWSG21-07	SWSG22-07	SWSG22-07-D	SWSG23-07	SWSG24-07	SWSG18-08	SWSG19-08	SWSG20-08	SWSG21-08	SWSG16-09	SWSG18-09	
SAMPLE	SWSG21-06	SWSG22-06	SWSG23-06	SWSG24-06	SWSG18-07	SWSG19-07	SWSG20-07	SWSG21-07	SWSG22-07	SWFD06250101	SWSG23-07	SWSG24-07	SWSG18-08	SWSG19-08	SWSG20-08	SWSG21-08	SWSG16-09	SWSG18-09	
MATRIX	SW	SW	SW	SW	SW	SW	SW	SW	SW										
QC TYPE	NM	FD	NM																
SAMPLE DATE	20010311	20010311	20010311	20010313	20010620	20010622	20010622	20010624	20010625	20010625	20010621	20010625	20010925	20010925	20010925	20010925	20011219	20011218	
CONTRACTOR	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN										
Volatiles (ug/L)																			
1,2,4-TRICHLOROBENZENE					1 U	1 U	1 U	1 UJ			1 U		1 U	1 U	1 U	1 U			
2-BUTANONE	5 UR			5 UR		5 UR	5 U	5 U											
ACETONE	5 U	5 U	5 U	5 U	5 UR	5 UR	5 UR	5 UR			5 UR		5 UR	5 U	6 U				
BENZENE	1 U	1 U	1 U	1 U	1 U	1 UJ	1 U	1 U			1 U		1 U	1 U	1 U	1 U	1 U	1 U	0.3 J
CARBON DISULFIDE	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 UJ			1 U		1 U	1 U	1 U	1 U	1 U	1 U	1 U
CHLOROBENZENE	1 U	1 U	1 U	1 U	1 U	1 UJ	1 U	1 U			1 U		1 U	1 U	1 U	1 U	1 U	1 U	0.9 J
CHLOROMETHANE	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U			1 U		1 U	1 U	1 U	1 U	1 U	1 U	1 U
ETHYLBENZENE	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U			1 U		1 U	1 U	1 U	1 U	1 U	1 U	2
METHANE																			
METHYLENE CHLORIDE	2 U	2 U	2 U	2 U	0.7 J	2 U	2 U	2 U			2 U		2 U	2 U	2 U	2 U	2 U	2 U	2 U
TETRACHLOROETHENE	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U			1 U		1 U	1 U	1 U	1 U	1 U	1 U	1 U
TOLUENE	1 U	1 U	1 U	1 U	1 U	1 U	2.6	1 U			1.9		13	0.9 J	0.9 J	0.7 J	1 U	1 U	1
TOTAL XYLENES	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U			1 U						1 U	5	
TRICHLOROETHENE	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U			1 U		1 U	1 U	1 U	1 U	1 U	1 U	1 U
Semivolatile Organics (ug/L)																			
1-METHYLNAPHTHALENE														0.5 U	0.5 U	0.5 U	0.5 U	0.1 U	0.2
2,4-DIMETHYLPHENOL	10 U	10 U	10 U	10 U	5 U	5 U	5 U	5 U			5 U		5 U	5 U	5 U	5 U	5 UJ	5 UJ	5 UJ
2-METHYLNAPHTHALENE					5 U	5 U	5 U	5 UJ			5 U		0.5 UJ	0.5 UJ	0.5 UJ	0.06 J	0.1 U	0.1	
2-METHYLPHENOL	10 U	10 U	10 U	10 U	5 U	5 U	5 U	5 U			5 U		5 U	5 U	5 U	5 U	5 UJ	5 UJ	5 UJ
3&4-METHYLPHENOL					5 U	5 U	5 U	5 U			5 U		5 U	5 U	5 U	5 U			
4-METHYLPHENOL	10 U	10 U	10 U	10 U													5 UJ	5 UJ	
4-NITROANILINE	50 UJ	50 UJ	50 UJ	50 UJ	20 U	20 U	20 U	20 UJ			20 U		20 UJ	20 UJ	20 UJ	20 UJ	20 U	20 U	20 U
ACENAPHTHENE	1 U	1 U	1 U	1 U	0.016 U	0.016 UJ	0.016 U	0.016 U			0.016 U		0.016 U	0.1 U	0.1				
ACENAPHTHYLENE	1 U	1 U	1 U	1 U	0.013 U	0.013 UJ	0.013 U	0.013 U			0.013 U		0.013 U	0.013 U	0.013 U	0.2	0.1 U	0.1 U	
ANTHRACENE	0.2 U	0.2 U	0.2 U	0.2 U	0.13	0.35 J	0.23	0.03 U			0.03 U		0.03 U	0.1 U	0.1 U				
BENZO(A)ANTHRACENE	0.2 U	0.2 U	0.2 U	0.2 U	0.012 U	0.012 U	0.012 U	0.012 U			0.012 U		0.012 UJ	0.012 UJ	0.012 UJ	0.012 UJ	0.1 U	0.1 UJ	
BENZO(A)PYRENE	0.022 J	0.2 U	0.2 U	0.2 U	0.021 U	0.021 U	0.021 U	0.021 U			0.021 U		0.021 U	0.021-U	0.021 U	0.021 U	0.1 U	0.1 U	
BENZO(B)FLUORANTHENE	0.2 U	0.2 U	0.2 U	0.2 U	0.02 U	0.02 U	0.02-U	0.02 U			0.02 U		0.02 U	0.02 U	0.02 U	0.02 U	0.1 U	0.1 U	
BENZO(G,H)PERYLENE	0.028 J	0.2 U	0.2 U	0.2 U	0.009 U	0.009 U	0.009 U	0.009 U			0.009 U		0.009 U	0.009 U	0.009 U	0.009 U	0.1 U	0.1 U	
BENZO(K)FLUORANTHENE	0.2 U	0.2 U	0.2 U	0.2 U	0.02 U	0.02 U	0.02 U	0.02 U			0.02 U		0.02 U	0.02 U	0.02 U	0.02 U	0.1 U	0.1 U	
BENZOIC ACID	50 U	50 U	50 U	50 U	5 U	5 U	5 U	5 U			5 U		5 U	5 U	5 U	5 U	20 UJ	20 UJ	20 UJ
BIS(2-ETHYLHEXYL)PHTHALATE	10 U	10 U	10 U	10 U	2.1 J	1.5 J	1.1 J	1.9 J			2.1 J		5 U	1.3 J	5 U	4.1 J	5 U	3 J	
BUTYL BENZYL PHTHALATE	10 U	10 U	10 U	10 U	5 U	5 U	5 U	5 UJ			5 U		5 U	5 U	5 U	5 U	5 U	5 U	5 U
CARBAZOLE	10 U	10 U	10 U	10 U	5 U	5 U	5 U	5 UJ			5 U		5 UJ	5 UJ	5 UJ	5 UJ	5 U	5 U	5 U
CHRYSENE	0.023 J	0.2 U	0.2 U	0.2 U	0.012 U	0.012 U	0.012 U	0.012 U			0.012 U		0.012 UJ	0.012 U	0.012 UJ	0.012 UJ	0.2 U	0.1 U	
DI-N-BUTYL PHTHALATE	10 U	10 U	10 U	10 U	0.8 J	1.2 J	1.3 J	1.5 J			0.8 J		5 U	5 U	5 U	5 U	0.9 J	5 U	5 U
DI-N-OCTYL PHTHALATE	10 U	10 U	10 U	10 U	5 U	5 U	5 U	5 UJ			5 U		5 U	5 U	5 U	2.3 J	5 U	5 U	5 U
DIBENZO(A,H)ANTHRACENE	0.2 U	0.2 U	0.2 U	0.2 U	0.014 U	0.014 U	0.014 U	0.014 U			0.014 U		0.014 U	0.014 U	0.014 U	0.014 U	0.1 U	0.1 U	
DIETHYL PHTHALATE	10 U	10 U	10 U	10 U	5 U	5 U	5 U	0.5 J			5 U		5 U	5 U	5 U	5 U	5 U	5 U	5 U
FLUORANTHENE	0.069 J	0.2 U	0.022 J	0.2 U	0.009 U	0.009 U	0.009 U	0.009 U			0.009 U		0.009 U	0.009 UJ	0.009 U	0.009 U	0.4 U	0.1 U	
FLUORENE	0.2 U	0.2 U	0.2 U	0.2 U	0.007 U	0.007 U	0.007 U	0.007 U			0.007 U		0.007 U	0.25	0.007 U	0.36	0.1 U	0.1 U	
INDENO(1,2,3-CD)PYRENE	0.2 U	0.2 U	0.2 U	0.2 U	0.008 U	0.008 U	0.008 U	0.008 U			0.008 U		0.008 U	0.008 U	0.008 U	0.008 U	0.1 U	0.1 U	
NAPHTHALENE	1 U	1 U	1 U	1 U	0.008 U	0.008 U	0.008 U	0.008 U			0.008 U		0.008 U	0.008 U	0.008 U	0.008 U	0.1 U	0.2	
PHENANTHRENE	0.041 J	0.2 U	0.2 U	0.2 U	0.004 U	0.004 UJ	0.004 U	0.004 U			0.004 U		0.004 U	0.004 U	0.004 U	0.004 U	0.1	0.1 U	
PHENOL	10 U	10 U	10 U	10 U	5 U	5 U	5 U	5 U			5 U		5 U	5 U	5 U	5 U	5 UJ	5 UJ	5 UJ
PYRENE	0.049 J	0.2 U	0.2 U	0.2 U	0.008 U	0.008 U	0.008 U	0.008 U			0.008 U		0.008 U	0.23	0.19	0.008 U	0.2 U	0.1 U	

SURFACE WATER ANALYTICAL DATA FOR THE AREA A LANDFILL MONITORING PROGRAM  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 8 OF 30

ROUND	06	06	06	06	07	07	07	07	07	07	07	07	08	08	08	08	09	09
LOCATION	SG-21	SG-22	SG-23	SG-24	SG-18	SG-19	SG-20	SG-21	SG-22	SG-22	SG-23	SG-24	SG-18	SG-19	SG-20	SG-21	SG-16	SG-18
NSAMPLE	SWSG21-06	SWSG22-06	SWSG23-06	SWSG24-06	SWSG18-07	SWSG19-07	SWSG20-07	SWSG21-07	SWSG22-07	SWSG22-07-D	SWSG23-07	SWSG24-07	SWSG18-08	SWSG19-08	SWSG20-08	SWSG21-08	SWSG16-09	SWSG18-09
SAMPLE	SWSG21-06	SWSG22-06	SWSG23-06	SWSG24-06	SWSG18-07	SWSG19-07	SWSG20-07	SWSG21-07	SWSG22-07	SWSG22-07-D	SWSG23-07	SWSG24-07	SWSG18-08	SWSG19-08	SWSG20-08	SWSG21-08	SWSG16-09	SWSG18-09
MATRIX	SW	SW	SW	SW	SW	SW	SW	SW	SW									
QC TYPE	NM	FD	NM															
SAMPLE DATE	20010311	20010311	20010311	20010313	20010620	20010622	20010622	20010624	20010625	20010625	20010621	20010625	20010925	20010925	20010925	20010925	20011219	20011218
<b>Polynuclear Aromatic Hydrocarbons (ug/L)</b>																		
2-METHYLNAPHTHALENE																		
ACENAPHTHENE																		
ACENAPHTHYLENE																		
ANTHRACENE																		
BAP EQUIVALENT																		
BENZO(A)ANTHRACENE																		
BENZO(A)PYRENE																		
BENZO(B)FLUORANTHENE																		
BENZO(G,H)PERYLENE																		
BENZO(K)FLUORANTHENE																		
CHRYSENE																		
DIBENZO(A,H)ANTHRACENE																		
FLUORANTHENE																		
FLUORENE																		
INDENO(1,2,3-CD)PYRENE																		
NAPHTHALENE																		
PHENANTHRENE																		
PYRENE																		
<b>Pesticides/PCBs (ug/L)</b>																		
4,4'-DDD	0.02 U			0.02 U		0.02 U	0.02 U	0.02 U	0.02 U	0.31	0.02 U							
4,4'-DDE	0.02 U			0.02 U		0.02 U	0.02 U	0.02 U	0.02 U	0.15	0.02 U							
4,4'-DDT	0.02 U			0.02 U		0.02 U	0.02 U	0.02 U	0.02 U	0.92	0.02 U							
ALDRIN	0.01 U			0.01 U		0.01 U												
ALPHA-CHLORDANE	0.01 U	0.1 U	0.1 U	0.1 U			0.1 U		0.01 U	0.01 U	0.01 U	0.01 U	0.013 J	0.01 U				
ENDOSULFAN I	0.01 U			0.01 U		0.01 U												
ENDOSULFAN II	0.02 U			0.02 U		0.02 U	0.02 U	0.02 U	0.02 U	0.021 U	0.02 U							
ENDRIN ALDEHYDE	0.02 U			0.02 U		0.02 U	0.02 U	0.02 U	0.02 U	0.021 U	0.02 U							
GAMMA-CHLORDANE	0.01 U	0.1 U	0.1 U	0.1 U			0.1 U		0.01 U	0.01 U	0.01 U	0.01 U	0.017	0.01 U				
<b>Total Inorganics (ug/L)</b>																		
ALUMINUM	102 U	251 U	64 U	216 U	457 U	457 U	1690	505	204 U	431 J	457 U	402 U	2180	163 U	437	78 U	13500	1300
ANTIMONY	3.2 U	3.2 U	3.2 U	3.2 U	3.7 U	3.7 U	3.7 U	9.2 U	9.2 U	9.2 U	9.2 U	3.65 U	3.65 U					
ARSENIC	2.6 U	2.6 U	2.6 U	2.6 U	5 U	5 U	5.3 J	5 U	6.8 J	7.3 J	7.5 J	6.6 J	7.7	9.1 J	7.2 J	5 U	3.6	2.53 U
BARIUM	8.4 U	56.9	9.8 U	7.2 U	32.6	27.5 J	37.3 J	47.9	54.8	53.8	42.9 J	95.6	82.6	63.9	26.7	38.2	105	99.1
BERYLLIUM	0.13 U	0.13 U	0.43 U	0.43 U	0.1 U	0.1 U	0.21 U	0.1 U	0.1 U	0.1 U	0.58 U	0.1 U	0.38 U	0.1 U	0.1 U	0.13 U	0.57 U	0.18 U
CADMIUM	2.6 UJ	2.6 UJ	2.6 UJ	2.6 UJ	3 U	3 U	3 U	3 U	3 U	3 U	3 U	3 U	3 U	3 U	3 U	3 U	2.84 U	0.3 U
CALCIUM	2840	35500	3990	1630	15000	13600	14300	20600	21600	20300	12200	11800	19100	28000	11200	17500	5210	20800
CHROMIUM	2.6 U	2.6 U	2.6 U	2.6 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	7.2 U	1.8 U
COBALT	4.1 U	4.1 U	4.1 U	4.1 U	1.8 U	1.8 U	1.9 J	2.5 J	1.8 U	1.9 J	1.8 U	9.9	5 U	5 U	5 U	5 U	6.6 J	0.92 U
COPPER	2 U	2.4 U	2 U	2.1 U	2.2 U	2.2 U	7.6 U	15.5 U	7.4 U	7.8 U	5.3 U	10.2 U	17.2	16	18.4	5.7	79.4	8.5 U
IRON	350	9010	444	314	10600	3890	4110	1830	25700	25900	4750	32000	20700	25200	5610	7330	25700	20800
LEAD	2 U	2 U	2 U	2 U	3 U	3 UJ	9.5	5 J	3 UJ	3.2 J	3 UJ	3 UJ	8.2 J	3 U	3.8 J	3 U	75.7	5.2 U
MAGNESIUM	718 U	4460	715 U	591 U	2700	2700	2810	4350	4020	3870	2550	4310	3740	4890	3230	4750	3620	3640
MANGANESE	89.2	722	56.4	82.7	498	208	281	764	392	372	278	1790	566	518	263	315	330	477
MERCURY	0.08 U	0.08 U	0.08 U	0.08 U	0.2 UJ	0.2 U	0.2 U	0.2 UJ	0.2 UJ	0.2 UJ	0.2 U	0.2 UJ	0.2 U	0.2 U	0.2 U	0.2 U	0.38 J	0.03 U
MOLYBDENUM																		
NICKEL	9.8 U	10.1 J	9.8 U	9.8 U	4 U	4 U	4 U	5.3 J	4 U	4 U	4 U	4 U	4.3 J	4 U	4 U	4 U	16.4	4.6 U
POTASSIUM	1920 U	3490 U	1040 U	1530 U	1070	1780 J	2010 J	21700 J	2030 J	1890 J	1700 J	2310 J	2070 J	4020	2470	4530	4720	4200
SELENIUM	3.8 UJ	3.8 UJ	3.8 UJ	3.8 UJ	5 UJ	5 UJ	5 UJ	5 UJ	5 U	5 U	5 UJ	5 U	5 UJ	5 U	5 U	5 UJ	3.04 UJ	3.2 U

SURFACE WATER ANALYTICAL DATA FOR THE AREA A LANDFILL MONITORING PROGRAM  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 9 OF 30

ROUND	06	06	06	06	07	07	07	07	07	07	07	07	07	08	08	08	08	09	09
LOCATION	SG-21	SG-22	SG-23	SG-24	SG-18	SG-19	SG-20	SG-21	SG-22	SG-22	SG-23	SG-24	SG-18	SG-19	SG-20	SG-21	SG-16	SG-18	
NSAMPLE	SWSG21-06	SWSG22-06	SWSG23-06	SWSG24-06	SWSG18-07	SWSG19-07	SWSG20-07	SWSG21-07	SWSG22-07	SWSG22-07-D	SWSG23-07	SWSG24-07	SWSG18-08	SWSG19-08	SWSG20-08	SWSG21-08	SWSG16-09	SWSG18-09	
SAMPLE	SWSG21-06	SWSG22-06	SWSG23-06	SWSG24-06	SWSG18-07	SWSG19-07	SWSG20-07	SWSG21-07	SWSG22-07	SWSG22-07-D	SWSG23-07	SWSG24-07	SWSG18-08	SWSG19-08	SWSG20-08	SWSG21-08	SWSG16-09	SWSG18-09	
MATRIX	SW	SW	SW	SW	SW	SW	SW	SW	SW										
QC TYPE	NM	FD	NM																
SAMPLE DATE	20010311	20010311	20010311	20010313	20010620	20010622	20010622	20010624	20010625	20010625	20010621	20010625	20010925	20010925	20010925	20010925	20011219	20011218	
SILVER	2.1 U	2.1 U	2.1 U	2.1 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	4.15 UJ	1.2 J	
SODIUM	8690 J	48400 J	5080 J	4580 J	40300	49200	47300	63700	58400	54300	21600	30300	37800	60600	38800	51100	10100	33200	
THALLIUM	6 U	6 U	6 U	6 U	5.7 UJ	5.7 UJ	5.7 UJ	6.4 J	5.7 UJ	5.7 UJ	5.7 UJ	5.7 UJ	7.2 U	7.2 U	7.2 U	7.2 U	0.33 U	0.43 U	
VANADIUM	3.2 U	2.6 U	2.6 U	2.6 U	3.2 U	3.2 U	5.6 J	3.2 U	3.2 U	3.2 U	3.2 U	3.2 U	19.7 J	14.2	5.1 J	2.6 UJ	270	9.9	
ZINC	185	82.3	334	15.8 U	55.9	30.2	109	107	81.6	84.9	108	95.4	263	109	121	55.3 J	424	394	
<b>Filtered Inorganics (ug/L)</b>																			
ALUMINUM	15.9 U	14.7 U	14.7 U	71.2 U	32.5 U	45.7 U	47.6 U	45.7 U	89 U	71.9 U	45.7 U	56.4 U	46.5 U	65.3 U	78.1 U	31.4 U	158 U	44.5 U	
ANTIMONY	3.2 U	3.2 U	3.2 U	3.2 U	8.9 U	3.7 U	3.7 U	3.7 U	9.2 U	9.2 U	9.2 U	9.2 U	3.65 U	3.65 U					
ARSENIC	2.6 U	2.6 U	2.6 U	2.6 U	6.2 U	5 U	5 U	5 U	5.9 J	5.7 J	5 U	5.6 J	5 U	5 U	5.6 J	5 U	2.53 U	2.53 U	
BARIUM	8.2 U	47.3	7.6 U	233	31	24.1 J	23.6 J	21.7	48.7	48.6	48.9 J	75.4	66.9	70.4	19 U	37.8	17 U	78.5	
BERYLLIUM	0.13 U	0.13 U	0.13 U	0.13 U	0.1 U	0.1 UJ	0.13 U	0.1 UJ	0.1 U	0.1 U	0.1 UJ	0.1 U	1.3 U	0.42 U	0.46 U	1.3 U	0.41 U	0.41 U	
CADMIUM	2.6 UJ	3.3 U	2.6 UJ	2.6 UJ	0.6 U	3 U	3 U	3 U	3 U	3 U	3 U	3 U	3 U	3 U	3 U	3 U	2.84 U	2.84 U	
CALCIUM	3320	33100	3690	1570	14800	12700	14200	17100	21000	20700	17600	11500	20000	27900	9230	17700	3640	19400	
CHROMIUM	2.6 U	2.6 U	2.6 UJ	2.6 U	0.6 U	5 U	5 U	5 U	5 U	5 U	27.5	5 U	5 U	5 U	5 U	5 U	0.57 U	4.81 UJ	
COBALT	4.1 U	4.2 U	4.1 U	4.1 U	1.4 J	1.8 U	1.9 J	7.8	5 U	5 U	5 U	5 U	3.89 U	3.89 U					
COPPER	2 U	2 U	2 U	2.1 U	1.5 U	6.4 U	5.6 U	2.2 U	6.3 U	4.5 U	5.1 U	3.3 U	4.6 J	4.7 J	2.4 U	3.2 J	7.2 U	1.66 U	
IRON	236 U	4120	110 U	130 U	3000	941	612	265 J	18100	18500	8160	8980	12300	17500	2020	3180	516 J	12400 J	
LEAD	2 U	2 U	2 U	2 U	3 U	3 UJ	3 UJ	3 UJ	3 U	3 U	3 U	3 U	4.6 U	1.48 U					
MAGNESIUM	770 U	4100	740 U	553 U	2670	2580	2960	3890	3830	3940	4140	3410	4510	2710	4870	3700	700	3430	
MANGANESE	97.9	681	48	78.8	465	187	243	38.2	362	359	474	1650	561	473	204	320	7.2	451	
MERCURY	0.08 U	0.08 U	0.08 U	0.08 U	0.2 U	0.2 U	0.2 U	0.2 U	0.2 UJ	0.2 UJ	0.2 U	0.2 UJ	0.2 U	0.2 U	0.2 U	0.2 U	0.03 U	0.03 U	
MOLYBDENUM																			
NICKEL	9.8 U	9.8 U	9.8 U	9.8 U	1.9 J	4 U	4 U	4 U	4 U	4 U	15.1	4 U	4 U	4 U	4 U	4 U	5.89 U	5.89 U	
POTASSIUM	2270 U	3320 U	981 U	1510 U	2060	1210 J	1170 J	1190 J	2080 J	2040 J	1710 J	2320 J	7720 J	4070 J	1810 J	5900 J	2660	4200	
SELENIUM	3.8 UJ	3.8 UJ	3.8 UJ	3.8 UJ	3.2 U	5 UJ	5 UJ	5 U	5 U	5 U	5 UJ	5 U	5 UJ	5 UJ	5 UJ	5 UJ	3.04 UJ	3.04 UJ	
SILVER	2.1 U	2.1 U	2.1 U	2.1 U	1.7 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	4.15 UJ	4.15 UJ	
SODIUM	10600 J	44600 J	5430 J	4900 J	39800	40900	42000	67400 J	60500 J	58800 J	49100	30500 J	41500	62800	35700	52700	10600	34900	
THALLIUM	6 U	6 U	6 U	6 U	4.3 U	5.7 UJ	5.7 UJ	5.7 U	5.7 UJ	5.7 UJ	5.7 UJ	5.7 UJ	7.2 U	7.2 U	7.2 U	7.2 U	0.22 U	0.41 U	
VANADIUM	2.6 U	2.6 U	2.6 U	2.6 U	1.2 U	3.2 U	3.2 U	3.2 U	2.6 UJ	6.2 J	2.6 UJ	2.6 UJ	10.1	4.66 U					
ZINC	161 J	53.6 J	342 J	13.5 U	47 J	43.7	41	29.1	60	56.2	41.5	57.6	102	86.1	51.2 J	50.1	104	101	
<b>Miscellaneous Parameters</b>																			
ALKALINITY (MG/L)	5 U	100	8.9	5 U	31	27	28				35		55	85	25	57	20 UJ	70 J	
CARBONATE ALKALINITY (MG/L)								29											
CHEMICAL OXYGEN DEMAND (MG/L)	10 U	13.8	10 U	10 U	20	5 U	17	37			14		100	43	75	23	20	20	
CHLORIDE (MG/L)	15.7	89	7.4	61	70	60	80	90			80		70	100	70	90	13	51	
HARDNESS (MG/L)	10.1	107	12.9	6.5 U	49	45	37	1 U			41		63	90	41	63	27.9	66.9	
HYDROGEN (NMO/L)																			
SULFATE (MG/L)	3.8	5.8	2.9	1.8	6	7	9	13			5		1 U	1 U	1 U	2	1.6 J	10 J	
SULFIDE (MG/L)																			
TOTAL DISSOLVED SOLIDS (MG/L)	46	255	99	10 U	220	230	180	400 J			210		430	320	280	250	60	140	
TOTAL ORGANIC CARBON (MG/L)	2.7 U	2.8 U	0.52 U	4.5 U	7.3	4.4	7.6	12 J			6.8		16	15	14	10	8 J	7.5 J	
TOTAL SUSPENDED SOLIDS (MG/L)																			
<b>Field Parameters</b>																			
DISSOLVED OXYGEN (MG/L)																			
DISSOLVED OXYGEN - METER (MG/L)	11.25	1.27	6.28	14.47	6.49	4.56	6.21	9.7	3.16		5.25	3.22	7.22	7.67	6.92	6.62	6.21	3.1	
MANGANESE (MG/L)																			
OXIDATION REDUCTION POTENTIAL (MV)	5	-68	-45	89	-79.8	31.6	13.6	-9.5	-102.7		-95.6	-11.8	-88	-61	-13	-81	-92	-75	
PH	7.15	7.03	8.03	7.55	7.87	6.51	6.55	7.46	6.8		7.82	6.56	6.35	6.16	6.18	6.44	6.72	7.12	
SALINITY (NG/L)	0.05	0.24	0.03	0.02									0 U	0 U	0 U	0 U	12.27	0.25	
SPECIFIC CONDUCTANCE (MS/CM)	0.097	0.491	0.069	0.037	0.309	0.236	0.231	0.335	0.43		0.368	0.253	0.405	0.583	0.302	0.494	0.611	0.322	
TEMPERATURE (C)	1.2	3.5	0.8	0.5	24.33	20.07	20.23	23.69	14.83		20.09	24.8	19	20.1	21	20.6	10.74	5.92	
TURBIDITY (NTU)	4.7	10	5	64	89	5.8	7.2				5.2		159	28	17.8	15.2	250	132	

SURFACE WATER ANALYTICAL DATA FOR THE AREA A LANDFILL MONITORING PROGRAM  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 10 OF 30

ROUND	09	09	09	09	09	10	10	10	10	10	10	10	11	11	11	11	11	
LOCATION	SG-19	SG-20	SG-21	SG-21	SG-23	SG-18	SG-19	SG-20	SG-20	SG-21	SG-23	SG-24	SG-18	SG-19	SG-20	SG-21	SG-21	
NSAMPLE	SWSG19-09	SWSG20-09	SWSG21-09	SWSG21-09-D	SWSG23-09	SWSG18-10	SWSG19-10	SWSG20-10	SWSG20-10-D	SWSG21-10	SWSG23-10	SWSG24-10	SWSG18-11	SWSG19-11	SWSG20-11	SWSG21-11	SWSG21-11-D	
SAMPLE	SWSG19-09	SWSG20-09	SWSG21-09	FD121801-01	SWSG23-09	SWSG1810	SWSG1910	SWSG2010	FD03210201	SWSG2110	SWSG2310	SWSG2410	SWSG1811	SWSG1911	SWSG2011	SWSG2111	FD09240201	
MATRIX	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	
QC TYPE	NM	NM	NM	FD	NM	NM	NM	NM	FD	NM	FD							
SAMPLE DATE	20011218	20011218	20011218	20011218	20011220	20020321	20020325	20020321	20020321	20020325	20020325	20020325	20020924	20020924	20020924	20020924	20020924	
CONTRACTOR	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	TTN	
Volatiles (ug/L)																		
1,2,4-TRICHLOROBENZENE						1 U	1 U	1 U	1 U	1 U	1 U	0.2 J						
2-BUTANONE	5 U	5 U	5 U	5 U	5 U	5 U	5 UR	5 U	5 U	5 UR	5 UR	5 UR						
ACETONE	5 U	5 U	5 U	5 U	5 U	5 UJ	5 UR	3 J	5 UJ	5 UR	5 U	5 U						
BENZENE	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U						
CARBON DISULFIDE	1 U	1 U	1 U	1 U	1 U	0.2 J	1 U	1 UJ	0.5 J	1 U	1 U	1 U						
CHLOROBENZENE	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U						
CHLOROMETHANE	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U						
ETHYLBENZENE	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U						
METHANE																		
METHYLENE CHLORIDE	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U						
TETRACHLOROETHENE	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U						
TOLUENE	1 U	1 U	0.9 J	0.9 J	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U						
TOTAL XYLENES	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U						
TRICHLOROETHENE	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U						
Semivolatile Organics (ug/L)																		
1-METHYLNAPHTHALENE	0.1 U	0.1 U	0.06 J	0.05 J	0.1 U	0.1 U	0.1 UJ	0.1 U	0.1 U	0.1 UJ	0.1 UJ	0.1 UJ	0.2 UJ	0.3 J	0.2 UJ	0.2 UJ	0.2 UJ	
2,4-DIMETHYLPHENOL	5 UJ	5 UJ	5 UJ	5 UJ	6 UJ	5 U	5 U	5 U	5 U	5 U	5 U	5 U	10 U	10 U	10 U	10 U	10 U	
2-METHYLNAPHTHALENE	0.1 U	0.1 U	0.1 UJ	0.1 U	0.1 U	0.1 U	0.1 UJ	0.1 U	0.1 U	0.1 UJ	0.1 UJ	0.1 UJ	0.2 UJ	0.1 J	0.2 UJ	0.2 UJ	0.2 UJ	
2-METHYLPHENOL	5 UJ	5 UJ	5 UJ	5 UJ	6 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	10 U	10 U	10 U	10 U	10 U	
3&4-METHYLPHENOL																		
4-METHYLPHENOL	5 UJ	5 UJ	5 UJ	5 UJ	6 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	10 U	10 U	10 U	10 U	10 U	
4-NITROANILINE	20 U	20 U	20 U	20 U	22 U	20 U	20 U	20 U	20 U	20 U	20 U	20 U	25 U					
ACENAPHTHENE	0.06 J	0.1 U	0.1 J	0.09 J	0.1 U	0.1 U	0.1 UJ	0.1 U	0.1 U	0.1 UJ	0.1 UJ	0.1 UJ	0.2 U	0.6	0.2 U	0.2 U	0.08 J	
ACENAPHTHYLENE	0.1 U	0.1 U	0.1 UJ	0.1 U	0.1 U	0.1 UJ	0.1 UJ	0.1 UJ	0.1 UJ	0.1 UJ	0.1 UJ	0.1 UJ	0.2 U					
ANTHRACENE	0.1 U	0.04 J	0.1 UJ	0.1 U	0.1 U	0.1 UJ	0.1 U	0.1 UJ	0.1 UJ	0.1 UJ	0.1 U	0.1 U	0.2 U					
BENZO(A)ANTHRACENE	0.1 U	1 U	0.1 U	0.1 U	0.1 UJ	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.2 U	0.2 UJ	0.2 U	0.2 U	0.2 U	
BENZO(A)PYRENE	0.1 U	1 U	0.1 UJ	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.2 U					
BENZO(B)FLUORANTHENE	0.1 U	1 U	0.1 UJ	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.2 U					
BENZO(G,H,I)PERYLENE	0.1 U	1 U	0.1 UJ	0.1 U	0.1 U	0.1 U	0.1 UJ	0.1 U	0.1 U	0.1 UJ	0.1 UJ	0.1 UJ	0.2 U					
BENZO(K)FLUORANTHENE	0.1 U	1 U	0.1 UJ	0.1 U	0.1 U	0.1 U	0.1 UJ	0.1 U	0.1 U	0.1 UJ	0.1 UJ	0.1 UJ	0.2 U					
BENZOIC ACID	20 UJ	20 UJ	20 UJ	20 UJ	22 UJ	20 UJ	20 UJ	20 UJ	20 UJ	20 UJ	20 UJ	20 UJ						
BIS(2-ETHYLHEXYL)PHTHALATE	5 U	12 J	5 U	5 U	6 U	5 U	5 U	5 U	15	5 U	5 U	5 U	10 U	10 U	10 U	10 U	10 U	
BUTYL BENZYL PHTHALATE	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	10 U	10 U	10 U	10 U	10 U	
CARBAZOLE	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	10 U	10 U	10 U	10 U	10 U	
CHRYSENE	0.1 U	1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.06 J	0.2 U	0.2 UJ	0.2 U	0.2 U	0.2 U	
DI-N-BUTYL PHTHALATE	2 J	5 U	1 J	1 J	6 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	10 U	10 U	10 U	10 U	10 U	
DI-N-OCTYL PHTHALATE	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	10 U	10 U	10 U	10 U	10 U	
DIBENZO(A,H)ANTHRACENE	0.1 U	1.5 U	0.1 UJ	0.1 U	0.1 U	0.1 U	0.1 UJ	0.1 U	0.1 U	0.1 UJ	0.1 UJ	0.1 UJ	0.2 U					
DIETHYL PHTHALATE	5 U	5 U	5 U	5 U	6 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	10 U	10 U	10 U	10 U	10 U	
FLUORANTHENE	0.2 U	2	0.1 UJ	0.1 U	0.2	0.1 U	0.1 UJ	0.1 U	0.1 U	0.1 UJ	0.1 UJ	0.09 J	0.2 U					
FLUORENE	0.1 U	0.1 U	0.1 UJ	0.1 U	0.1 U	0.1 UJ	0.1 UJ	0.1 UJ	0.1 UJ	0.1 UJ	0.1 UJ	0.1 UJ	0.2 U	0.3	0.2 U	0.2 U	0.2 U	
INDENO(1,2,3-CD)PYRENE	0.1 U	1 U	0.1 UJ	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.2 U					
NAPHTHALENE	0.1 U	0.1 U	0.07 J	0.06 J	0.1 U	0.1 UJ	0.1 U	0.1 UJ	0.1 UJ	0.1 U	0.1 U	0.1 U	0.2 UJ	0.5 J	0.2 UJ	0.2 UJ	0.2 UJ	
PHENANTHRENE	0.09 J	0.5	0.1 UJ	0.1 U	0.07 J	0.1 UJ	0.1 UJ	0.1 UJ	0.1 UJ	0.1 UJ	0.1 UJ	0.1 UJ	0.4 J	0.2 U	0.2 U	0.2 U	0.2 U	
PHENOL	5 UJ	5 UJ	5 UJ	5 UJ	6 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	10 U	10 U	10 U	10 U	10 U	
PYRENE	0.2 U	1	0.1 UJ	0.1 U	0.1 U	0.1 UJ	0.1 U	0.1 UJ	0.1 UJ	0.1 U	0.1 U	0.09 J	0.2 UJ	0.2 U	0.2 UJ	0.2 UJ	0.2 UJ	

SURFACE WATER ANALYTICAL DATA FOR THE AREA A LANDFILL MONITORING PROGRAM  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 11 OF 30

ROUND	09	09	09	09	09	10	10	10	10	10	10	10	11	11	11	11	11
LOCATION	SG-19	SG-20	SG-21	SG-21	SG-23	SG-18	SG-19	SG-20	SG-20	SG-21	SG-23	SG-24	SG-18	SG-19	SG-20	SG-21	SG-21
NSAMPLE	SWSG19-09	SWSG20-09	SWSG21-09	SWSG21-09-D	SWSG23-09	SWSG18-10	SWSG19-10	SWSG20-10	SWSG20-10-D	SWSG21-10	SWSG23-10	SWSG24-10	SWSG18-11	SWSG19-11	SWSG20-11	SWSG21-11	SWSG21-11-D
SAMPLE	SWSG19-09	SWSG20-09	SWSG21-09	FD121801-01	SWSG23-09	SWSG1810	SWSG1910	SWSG2010	FD03210201	SWSG2110	SWSG2310	SWSG2410	SWSG1811	SWSG1911	SWSG2011	SWSG2111	FD09240201
MATRIX	SW	SW	SW	FD	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW
QC TYPE	NM	NM	NM	FD	NM	NM	NM	NM	FD	NM	FD						
SAMPLE DATE	20011218	20011218	20011218	20011218	20011220	20020321	20020325	20020321	20020321	20020325	20020325	20020325	20020924	20020924	20020924	20020924	20020924
<b>Polynuclear Aromatic Hydrocarbons (ug/L)</b>																	
2-METHYLNAPHTHALENE																	
ACENAPHTHENE																	
ACENAPHTHYLENE																	
ANTHRACENE																	
BAP EQUIVALENT																	
BENZO(A)ANTHRACENE																	
BENZO(A)PYRENE																	
BENZO(B)FLUORANTHENE																	
BENZO(G,H,I)PERYLENE																	
BENZO(K)FLUORANTHENE																	
CHRYSENE																	
DIBENZO(A,H)ANTHRACENE																	
FLUORANTHENE																	
FLUORENE																	
INDENO(1,2,3-CD)PYRENE																	
NAPHTHALENE																	
PHENANTHRENE																	
PYRENE																	
<b>Pesticides/PCBs (ug/L)</b>																	
4,4'-DDD	0.02 U	0.02 U	0.019 U	0.019 U	0.022 U	0.019 U	0.019 U	0.019 U	0.02 U	0.019 U	0.019 UJ	0.021 U					
4,4'-DDE	0.02 U	0.02 U	0.019 U	0.019 U	0.022 U	0.019 U	0.019 U	0.019 U	0.02 U	0.019 U	0.019 UJ	0.021 U					
4,4'-DDT	0.02 U	0.02 U	0.019 U	0.019 U	0.022 U	0.019 U	0.019 U	0.019 U	0.02 U	0.019 U	0.019 UJ	0.021 U					
ALDRIN	0.01 U	0.014 J	0.02 J	0.01 UJ	0.011 U	0.009 U	0.009 U	0.01 U	0.01 U	0.01 U	0.01 UJ	0.01 U					
ALPHA-CHLORDANE	0.01 U	0.01 U	0.01 U	0.01 U	0.011 U	0.009 U	0.009 U	0.01 U	0.01 U	0.01 U	0.01 UJ	0.01 U					
ENDOSULFAN I	0.01 U	0.01 U	0.01 U	0.01 U	0.011 U	0.009 U	0.009 U	0.01 U	0.01 U	0.01 U	0.01 UJ	0.01 U					
ENDOSULFAN II	0.02 U	0.02 U	0.019 U	0.019 U	0.022 U	0.019 U	0.019 U	0.019 U	0.02 U	0.019 U	0.019 UJ	0.021 U					
ENDRIN ALDEHYDE	0.02 U	0.024 J	0.019 U	0.019 U	0.022 U	0.019 U	0.019 U	0.019 U	0.02 U	0.019 U	0.019 UJ	0.021 U					
GAMMA-CHLORDANE	0.01 U	0.01 U	0.01 U	0.01 U	0.011 U	0.009 U	0.009 U	0.01 U	0.01 U	0.01 U	0.01 UJ	0.01 U					
<b>Total Inorganics (ug/L)</b>																	
ALUMINIUM	2920	16500	109 U	961 U	1440	529 U	385 U	106 U	115 U	132 U	376	1140	245 U	472	285 U	120 U	126 U
ANTIMONY	3.65 U	3.65 U	3.65 U	3.65 U	5	0.26 U	0.36 U	0.24 U	0.61 U	0.33 U	0.52 U	0.41 U	2.13 U				
ARSENIC	2.53 U	7.4	2.53 U	2.53 U	2.53 U	0.8 U	0.8 U	0.8 U	3.2 U	0.8 U	1.8	2.2	3.8 J	3.3 J	2 U	2 U	2 U
BARIIUM	47.9	138	55.7	55.7	35	24.9	16.6	14.4	13.9	19.4	21.1	69.4	55	77.2	30.3	38	33.4
BERYLLIUM	0.18 U	0.72 U	0.18 U	0.18 U	0.41 U	0.29 U	0.29 U	0.29 U	0.29 U	0.29 U	0.29 U	0.29 U	0.26 U	0.15 U	0.15 U	0.15 U	0.15 U
CADMIUM	0.76 U	1.3 U	0.24 U	0.53 U	2.84 U	2.98 UJ	2.98 U	2.98 UJ	2.98 UJ	2.98 U	2.98 U	2.98 U	0.25 U				
CALCIUM	11600	25600	34100	33700	8080	12400	9720	8130	8320	11100	5130	7130	14400	27000	8920	11000	10800
CHROMIUM	4.2 U	21.1 U	0.57 U	1.9 U	1.7 U	0.35 U	0.56 U	0.36 U	1.4 U	0.43 U	1.3 U	1.9 U	1.1	1.8	0.86 J	0.92 J	0.63 J
COBALT	1.3 J	6.8 J	0.92 U	0.92 U	3.89 U	2.93 U	2.93 U	2.93 U	2.93 U	2.93 U	2.93 U	2.93 U	1.6	1.7	1.1 J	1.4	1.2 J
COPPER	25.6	64.8	0.84 U	0.84 U	14 U	1.74 U	1.74 U	1.74 U	1.74 U	1.74 U	7.4	11.7	6.3	12.2	5.6 U	4.3 U	3.4 U
IRON	11100	23600	6120	5940	4380	2200 J	1450	953 J	934 J	838	3110	5250	16800	29200	7580	7500	6290
LEAD	13.3	41.8	2.3 U	3.4 U	7.4 U	0.34 U	0.64 U	0.41 U	2.2 U	1.9 U	3.3 U	4.2 U	2.6	5.3	2.6	2.1 J	2 J
MAGNESIUM	2480	9670	6480	6440	4290	3150	3140	2730	2790	3120	1460	3560	2360	4570	3370	4050	4000
MANGANESE	311	655	510	509	331	53 J	13.6	43.8 J	40.5 J	34.8	151	100	411	502	449	613 J	426 J
MERCURY	0.05 J	0.13 J	0.03 U	0.03 U	0.03 U	0.06 U	0.1 U	0.05 U	0.09 U	0.05 U	0.04 U	0.08 U	0.12 U	0.12 U	0.09 U	0.14 U	0.12 U
MOLYBDENUM																	
NICKEL	11.1	31	1.7 U	2 U	5.89 U	9.25 U	9.25 U	9.25 U	9.25 U	9.25 U	10.3 U	9.25 U	5.1	9	2.9	2.2	1.7 J
POTASSIUM	4530	14900	7480	7510	3960	3500 J	3260	3410 J	3000 J	3380	2750	4120	1230	5300	3630	4100	3760
SELENIUM	5.4 U	4.7 U	5.5 U	3.04 UJ	3.04 UJ	2 U	2 UJ	2 U	8 U	2 UJ	2 UJ	2 UJ	2.36 U				

SURFACE WATER ANALYTICAL DATA FOR THE AREA A LANDFILL MONITORING PROGRAM  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 12 OF 30

ROUND	09	09	09	09	09	10	10	10	10	10	10	10	11	11	11	11	11	
LOCATION	SG-19	SG-20	SG-21	SG-21	SG-23	SG-18	SG-19	SG-20	SG-20	SG-20	SG-21	SG-23	SG-24	SG-18	SG-19	SG-20	SG-21	SG-21
NSAMPLE	SWSG19-09	SWSG20-09	SWSG21-09	SWSG21-09-D	SWSG23-09	SWSG18-10	SWSG19-10	SWSG20-10	SWSG20-10-D	SWSG21-10	SWSG23-10	SWSG24-10	SWSG18-11	SWSG19-11	SWSG20-11	SWSG21-11	SWSG21-11-D	
SAMPLE	SWSG19-09	SWSG20-09	SWSG21-09	FD121801-01	SWSG23-09	SWSG1810	SWSG1910	SWSG2010	FD03210201	SWSG2110	SWSG2310	SWSG2410	SWSG1811	SWSG1911	SWSG2011	SWSG2111	FD09240201	
MATRIX	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	
QC TYPE	NM	NM	NM	FD	NM	NM	NM	NM	FD	NM								
SAMPLE DATE	20011218	20011218	20011218	20011218	20011220	20020321	20020325	20020321	20020321	20020325	20020325	20020325	20020924	20020924	20020924	20020924	20020924	
SILVER	1.03 UJ	1.1 J	1.1 J	1 J	4.15 UJ	3.89 U	3.89 U	3.89 U	3.89 U	3.89 U	3.89 U	3.89 U	1.02 U					
SODIUM	10800	49500	58000	58200	53200	40400 J	40100	34100 J	34400 J	38400	13200	29700	21900	53500	34400	43100	42600	
THALLIUM	0.18 U	0.99 U	0.1 U	0.2 U	0.16 U	0.1 U	0.1 U	0.1 U	0.4 U	0.1 U	0.1 U	0.1 U	4.38 U	4.38 U	4.38 U	4.38 U	4.38 U	
VANADIUM	30.6	46	1.6 U	1.7 U	6.8 J	3.84 U	3.84 U	3.84 U	3.84 U	3.84 U	4.7 J	3.84 U	9.3	18.8	2.7	1.9	1.6	
ZINC	259	1160	14.6	14.3	305	69.8 J	15.6	19.5 J	20.1 J	10.2	344	123	156	62.5	27.7	11.7	8.9	
Filtered Inorganics (ug/L)																		
ALUMINUM	21.39 U	51.7 U	34.3 U	21.39 U	92.9 U	15.6 U	29.1 U	15.6 U	15.6 U	21.2 U	87.8 U	21.4 U	7.7 U	67.1 U	53.2 U	25.37 U	25.37 U	
ANTIMONY	3.65 U	3.65 U	3.65 U	3.65 U	3.7	0.26 U	0.35 U	0.3 U	0.16 U	0.42 U	0.6 U	0.39 U	2.13 U					
ARSENIC	2.53 U	2.53 U	2.53 U	2.53 U	2.6	0.8 U	0.8 U	0.93 J	0.8 U	0.8 U	1.1 U	0.8 U	2.3 J	3.4 J	2 U	2 U	2 U	
BARIUM	24	47.2	52.1	50.3	22	27.1	17.6	14	13.5	19	19.2	59.9	53.1	69.2	26.3	31.8	33	
BERYLLIUM	0.41 U	0.41 U	0.41 U	0.42 U	0.41 U	0.29 U	0.29 U	0.29 U	0.29 U	0.29 U	0.29 U	0.29 U	0.37 U					
CADMIUM	2.84 U	2.84 U	2.84 U	2.84 U	2.84 U	2.98 UJ	2.98 U	2.98 UJ	2.98 UJ	2.98 U	2.98 U	3 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	
CALCIUM	9040	20500	30000	28800	8110	13200	10400	8960	3770	11400	5330	7190	15900	27100	8540	11100	11200	
CHROMIUM	4.81 U	4.81 UJ	4.81 UJ	4.81 U	0.57 U	0.35 U	0.6 U	0.8 U	0.45 U	0.47 U	0.82 U	0.44 U	2.75 U	2.75 U	2.75 U	2.75 U	0.55 U	
COBALT	3.89 U	3.89 U	3.89 U	3.89 U	3.89 U	2.93 U	2.93 U	2.93 U	2.93 U	2.93 U	2.93 U	2.93 U	5.13 U	5.13 U	5.13 U	5.13 U	0.91 J	
COPPER	1.66 U	2.1 U	1.66 U	2.6 U	2.8 U	1.74 U	1.8 J	1.74 U	1.74 U	1.74 U	3 J	3.7	2.2 U	3.6 U	2.4 U	2.1 U	2.1 U	
IRON	2820 J	153 U	944 J	481 J	2050 J	749 J	385	427 J	394 J	392	2140	440	12600	23100	5090	2380	2020	
LEAD	2.3 U	1.48 U	1.48 U	2.5 U	2.5 U	0.31 U	0.4 U	0.16 U	0.13 U	0.6 U	1.5 U	0.46 U	1.26 U					
MAGNESIUM	1670	5380	6240	5960	4080	3420	3390 J	2960	1290	3140 J	1480 J	3330 J	2600	4560	3220	4050	4100	
MANGANESE	230	332	462	436	280	54.6 J	15.1	30.1 J	12 J	38.1	160	87.5	436	499	430	414	430	
MERCURY	0.03 U	0.03 U	0.03 U	0.03 U	0.03 U	0.03 U	0.1 U	0.04 U	0.09 U	0.11 U	0.03 U	0.13 U	0.05 U	0.06 U	0.05 U	0.05 U	0.07 U	
MOLYBDENUM																		
NICKEL	5.89 U	5.89 U	5.89 U	5.89 U	5.89 U	9.25 U	9.25 U	9.25 U	9.25 U	9.25 U	9.25 U	9.25 U	4.3	6.5	1.8	1.6 J	1.4 J	
POTASSIUM	4360	12400	7360	7680	3820	3630 J	3850	2890 J	2950 J	3380	3000	3890	985	5140	3710	3870	3880	
SELENIUM	3.04 UJ	3.04 UJ	3.04 UJ	3.3 U	3.04 UJ	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2.36 U	2.36 U	2.36 U	2.36 U	2.36 U	
SILVER	4.15 UJ	4.15 UJ	4.15 UJ	4.15 UJ	4.15 UJ	3.89 U	3.89 U	3.89 U	3.89 U	3.89 U	3.89 U	3.89 U	1.02 U					
SODIUM	11500	52600	60000	58800	56500	42100 J	43400 J	35100 J	34500 J	38600 J	14200 J	31100 J	25000	54900	33400	43600	43600	
THALLIUM	0.16 U	0.69 U	0.15 U	0.26 U	0.1 U	0.47 U	0.1 U	0.1 U	0.1 U	0.1 U	0.18 U	0.1 U	4.38 U	4.38 U	4.38 U	4.38 U	4.38 U	
VANADIUM	4.66 U	4.66 U	4.66 U	4.66 U	4.66 U	3.84 U	3.84 U	3.84 U	3.84 U	3.84 U	3.84 U	3.84 U	3.3	7.7	1.2 J	0.88 J	0.7 U	
ZINC	28.2	145	7.1 U	10 U	126	60.6 J	15	21 J	9.1 U	9.1	290	109	86.9	7.3	9.6	3.7	3.8	
Miscellaneous Parameters																		
ALKALINITY (MG/L)	30 J	40 J	90 J	100 J	20 J	30	20 U	20 U	20 U	20	20 U	20 U	20	74	17.46 J	20 J	30 J	
CARBONATE ALKALINITY (MG/L)																		
CHEMICAL OXYGEN DEMAND (MG/L)	30	30	30	30	30	15 U	15 U	15 U	15 U	15 U	15 U	15 U	30 U	40 U	40 U	56 U	40 U	
CHLORIDE (MG/L)	17	99	100	100	84	70	80	60	60	70	20	70	44	98	64	80	82	
HARDNESS (MG/L)	39.2	104	112	111	37.8	44	37.2	31.6	32.2	40.5	18.8	32.4	45.7	86.2	36.1	44.1	43.6	
HYDROGEN (NMO/L)																		
SULFATE (MG/L)	12 J	18 J	5 J	5.1 J	15 J	9.6	14	11	11	13	3	4	22	4.5	15	13 J	8.9 J	
SULFIDE (MG/L)																		
TOTAL DISSOLVED SOLIDS (MG/L)	56	250	260	280	250	190	300	130	170	170	110	270	210	200	180	190	200	
TOTAL ORGANIC CARBON (MG/L)	20 J	14 J	11 J	20 J	20 J	4	5.5	5	4	5.4	6.6	4	28	40	21	20	26	
TOTAL SUSPENDED SOLIDS (MG/L)																		
Field Parameters																		
DISSOLVED OXYGEN (MG/L)																		
DISSOLVED OXYGEN - METER (MG/L)	2.1	4.24	2.4		5.4	4.87	8.45	9.88		9.73	3.67	6.72	0.79	1.14	0.68	1.23		
MANGANESE (MG/L)																		
OXIDATION REDUCTION POTENTIAL (MV)	-72	151	97		-15	-241	26	-240		2	-93	64	-39	-133	-113	-14		
PH	7.23	6.57	6.63		5.5	8.42	6.68	8.86		6.84	7.45	6.37	6.18	6.37	6.28	6.19		
SALINITY (NG/L)	0.09	0.27	0.36		0.22	0.3	0.15	0.13		0.14	0.1	0.11	0.13	0.25	0.15	0.16		
SPECIFIC CONDUCTANCE (MS/CM)	0.188	0.558	0.739		0.461	0.414	0.304	0.274		0.291	0.207	0.23	0.272	0.508	0.311	0.334		
TEMPERATURE (C)	6.1	5.6	5.3		4.7	6.3	5.4	5.8		5.2	3.4	4.6	17.6	17.8	19.6	18.5		
TURBIDITY (NTU)	1.5	8.74	28.1		6.32	4.5	3.1	4.6		3.5	1.2	3.4	3.7	7.9	4.5	10		



SURFACE WATER ANALYTICAL DATA FOR THE AREA A LANDFILL MONITORING PROGRAM  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 14 OF 30

ROUND LOCATION	11 SG-23	11 SG-24	12 SG-18	12 SG-19	12 SG-22	12 SG-23	12 SG-24	12 SG-24	12 SG-24	13 SG-18	13 SG-19	13 SG-19
NSAMPLE	SWSG23-11	SWSG24-11	2-SW18-01-20030411	2-SW19-01-20030408	2-SW22-01-20030411	2-SW23-01-20030411	2-SW24-01-20030411	2-SW24-01-20030411-D	2-SW24-01-20030411-D	2-SW18-03-20030815	2-SW19-03-20030818	2-SW19-03-20030818-D
SAMPLE MATRIX	SWSG2311	SWSG2411	2-SW18-01	2-SW19-01	2-SW22-01	2-SW23-01	2-SW24-01	2-SW24-01D	2-SW24-01D	2-SW18-03	2-SW19-03	FD-081803-03
QC TYPE	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	FD
SAMPLE DATE	20020924	20020924	20030411	20030408	20030411	20030411	20030411	20030411	20030411	20030815	20030818	20030818
Polynuclear Aromatic Hydrocarbons (ug/L)												
2-METHYLNAPHTHALENE			0.20 U	0.83	0.20 U	0.20 U	0.20 U	0.20 U	0.20 U	0.20 UJ	0.20 UJ	0.20 UJ
ACENAPHTHENE			0.10 U	1.6	0.10 U	0.10 U	0.10 U	0.10 U	0.10 U	0.31 J	1	1.1
ACENAPHTHYLENE			0.10 U	0.10 U	0.20 UJ	0.20 UJ	0.20 UJ					
ANTHRACENE			0.10 U	0.10 U	0.20 UJ	0.20 U	0.20 U					
BAP EQUIVALENT												
BENZO(A)ANTHRACENE			0.10 U	0.10 U	0.16 J	0.20 U	0.20 U					
BENZO(A)PYRENE			0.10 U	0.10 U	0.15 J	0.20 U	0.20 U					
BENZO(B)FLUORANTHENE			0.10 U	0.10 U	0.23 J	0.20 U	0.20 U					
BENZO(G,H,I)PERYLENE			0.10 U	0.10 U	0.25 UJ	0.25 U	0.25 U					
BENZO(K)FLUORANTHENE			0.10 U	0.10 U	0.21 J	0.20 U	0.20 U					
CHRYSENE			0.10 U	0.10 U	0.22 J	0.20 U	0.20 U					
DIBENZO(A,H)ANTHRACENE			0.10 U	0.10 U	0.20 UJ	0.20 U	0.20 U					
FLUORANTHENE			0.10 U	0.12	0.10 U	0.10 U	0.10 U	0.10 U	0.10 U	0.18 J	0.13 J	0.14 J
FLUORENE			0.10 U	0.85	0.10 U	0.10 U	0.10 U	0.10 U	0.10 U	0.18 J	0.74	0.79
INDENO(1,2,3-CD)PYRENE			0.10 U	0.10 U	0.20 UJ	0.20 U	0.20 U					
NAPHTHALENE			0.10 U	4.6	0.10 U	0.10 U	0.10 U	0.10 U	0.10 U	0.43 J	0.24 J	0.15 J
PHENANTHRENE			0.10 U	0.48	0.10 U	0.10 U	0.10 U	0.10 U	0.10 U	0.12 J	0.62	0.57
PYRENE			0.10 U	0.10 U	0.20 J	0.10 J	0.11 J					
Pesticides/PCBs (ug/L)												
4,4'-DDD												
4,4'-DDE												
4,4'-DDT												
ALDRIN												
ALPHA-CHLORDANE												
ENDOSULFAN I												
ENDOSULFAN II												
ENDRIN ALDEHYDE												
GAMMA-CHLORDANE												
Total Inorganics (ug/L)												
ALUMINIUM	753	667	100 U	100 U	130	100 U	140 J	100 UJ	530 J	72 J	76 J	
ANTIMONY	2.13 U	2.13 U	50 U	50 U	50 U	50 U	50 U	50 U	2.05 U	50 U	2.09 U	
ARSENIC	6	2 U	4 U	4 U	4 U	4 U	4 J	4 UJ	4 UJ	4 U	4 U	
BARIUM	22.4	103	30	40	50	30	160	110	60 J	40	40	
BERYLLIUM	0.33 U	0.18 U	4 U	4 U	4 U	4 U	4 U	4 U	2 UJ	2 U	2 U	
CADMIUM	0.25 U	0.25 U	5 U	5 U	5 U	5 U	5 U	5 U	2 UJ	2 U	2 U	
CALCIUM	6960	5180	18000	27000	26000	11000	18000	16000	21000 J	18000	19000	
CHROMIUM	2	1.3	10 U	10 UJ	10 U	10 U						
COBALT	1.8	5.7	20 U	1 U	0.96 U	0.97 U						
COPPER	14.4	24	10 U	6 J	10 U	1.8 J						
IRON	4020	4380	2700	10000	8300	2900	28000	17000	18000 J	7800	8600	
LEAD	5.4	4.2	10 U	5 UJ	5 U	5 U						
MAGNESIUM	1630	2880	3700	4300	4100	2100	17000	12000	4100 J	4000	4000	
MANGANESE	130	312	150	230	300	220	1200	900	420 J	420	420	
MERCURY	0.13 U	0.12 U	0.20 J	0.20 UJ	0.20 U	0.20 U						
MOLYBDENUM									4.6 U	50 U	4.6 J	
NICKEL	8.8	11.4	25 U	6.37 J	25 U	25 U						
POTASSIUM	1320	4830	3500	4300	4000	2000	7700	5600	2500 J	2200	2200	
SELENIUM	2.36 U	2.36 U	10 U	10 U	10 U	10 U	10 U	10 U	10 UJ	10 U	10 U	





SURFACE WATER ANALYTICAL DATA FOR THE AREA A LANDFILL MONITORING PROGRAM  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 17 OF 30

ROUND	13	13	13	13	13	14	14	14	14	14
LOCATION	SG-20	SG-21	SG-22	SG-23	SG-24	SG-18	SG-19	SG-19	SG-20	SG-21
NSAMPLE	2-SW20-03-20030815	2-SW21-03-20030815	2-SW22-03-20030815	2-SW23-03-20030815	2-SW24-03-20030815	2-SW18-02-20040602	2-SW19-02-20040602	2-SW19-02-20040602-D	2-SW20-02-20040603	2-SW21-02-20040602
SAMPLE	2-SW20-03	2-SW21-03	2-SW22-03	2-SW23-03	2-SW24-03	2-SW18-02	2-SW19-02	60204	2-SW20-02	2-SW21-02
MATRIX	SW	SW	SW							
QC TYPE	NM	FD	NM	NM						
SAMPLE DATE	20030815	20030815	20030815	20030815	20030815	20040602	20040602	20040602	20040603	20040602
<b>Polynuclear Aromatic Hydrocarbons (ug/L)</b>										
2-METHYLNAPHTHALENE	0.20 UJ	0.20 U	0.20 U	0.20 U	0.20 U	0.20 J				
ACENAPHTHENE	0.20 UJ	0.20	0.20 J	0.20 UJ	0.20 U	0.20				
ACENAPHTHYLENE	0.20 UJ	0.20 U	0.20 U	0.20 U	0.20 U	0.20 U				
ANTHRACENE	0.20 UJ	0.20 U	0.20 U	0.20 U	0.20 U	0.20 U				
BAP EQUIVALENT										
BENZO(A)ANTHRACENE	0.20 UJ	0.071 J	0.20 UJ	0.20 UJ	0.12 J	0.20 UJ	0.20 J	0.20 UJ	0.20 UJ	0.20 UJ
BENZO(A)PYRENE	0.20 UJ	0.20 UJ	0.20 UJ	0.20 UJ	0.22 J	0.20 U	0.20 U	0.20 U	0.20 U	0.20 U
BENZO(B)FLUORANTHENE	0.20 UJ	0.20 UJ	0.20 UJ	0.20 UJ	0.40 J	0.20 UJ	0.20 UJ	0.20 UJ	0.20 U	0.20 UJ
BENZO(G,H,I)PERYLENE	0.25 UJ	0.25 U	0.25 U	0.25 U	0.25 UJ	0.25 U				
BENZO(K)FLUORANTHENE	0.20 UJ	0.20 UJ	0.20 UJ	0.20 UJ	0.42 J	0.20 UJ	0.20 J	0.20 UJ	0.20 UJ	0.20 UJ
CHRYSENE	0.20 UJ	0.081 J	0.20 UJ	0.20 UJ	0.23 J	0.20 U	0.20 J	0.20 U	0.20 U	0.20 U
DIBENZO(A,H)ANTHRACENE	0.20 UJ	0.20 U	0.20 U	0.20 U	0.20 U					
FLUORANTHENE	0.20 UJ	0.20 UJ	0.20 UJ	0.08 J	0.36 J	0.20 U	0.20 J	0.20 U	0.20 U	0.20 J
FLUORENE	0.20 UJ	0.20 U	0.20 U	0.20 U	0.20 U	0.20 J				
INDENO(1,2,3-CD)PYRENE	0.20 UJ	0.20 U	0.20 U	0.20 U	0.20 U	0.20 U				
NAPHTHALENE	0.20 UJ	0.20 U	0.20 J	0.20 U	0.20 U	0.20 J				
PHENANTHRENE	0.20 UJ	0.20 UJ	0.20 UJ	0.20 UJ	0.18 J	0.20 UJ	0.20 J	0.20 UJ	0.20 U	0.20 J
PYRENE	0.20 UJ	0.20 UJ	0.20 UJ	0.086 J	0.33 J	0.20 UJ	0.20 J	0.20 UJ	0.20 U	0.20 J
<b>Pesticides/PCBs (ug/L)</b>										
4,4'-DDD										
4,4'-DDE										
4,4'-DDT										
ALDRIN										
ALPHA-CHLORDANE										
ENDOSULFAN I										
ENDOSULFAN II										
ENDRIN ALDEHYDE										
GAMMA-CHLORDANE										
<b>Total Inorganics (ug/L)</b>										
ALUMINUM	51 J	39 J	260 J	720 J	56000 J	100 J	550 J	240 J	100 J	320
ANTIMONY	50 UJ	50 UJ	50 UJ	50 UJ	2.5 U	1 J	1.1	1 J	1 J	1 J
ARSENIC	4 UJ	4 UJ	9 J	4 J	190 J	1 J	2.9 J	1.6 J	1 J	1 J
BARIUM	30 J	30 J	80 J	80 J	1100 J	30.5	51.3	31.5	20.4	24.8
BERYLLIUM	2 UJ	2 UJ	2 UJ	2 UJ	2 U	1 U	1 U	1 U	1 U	1 U
CADMIUM	2 UJ	0.10 U	0.20 J	0.10 UJ	0.10 U	0.10 U				
CALCIUM	16000 J	17000 J	36000 J	38000 J	30000 J	11000	12000	11000	9300	11000
CHROMIUM	10 UJ	10 UJ	10 UJ	10 UJ	70 J	1 J	2	1.3	1 J	1 J
COBALT	1.2 U	0.83 U	1.6 U	5 J	60 J	1 J	1	1 J	1 U	1 J
COPPER	4.6 J	1.9 J	5.5 J	7.3 J	290 J	2	12.7 J	5.7 J	1.3	2
IRON	4200 J	2900 J	40000 J	20000 J	430000 J	9800 J	42000 J	17000 J	3000 J	3800 J
LEAD	5 UJ	5 UJ	5 UJ	5 UJ	198 J	0.50 J	5.5 J	2.2 J	0.60	3.4
MAGNESIUM	3400 J	3400 J	5500 J	8500 J	33000 J	3000	3100	3000	2800	3100
MANGANESE	390 J	300 J	580 J	760 J	3800 J	260	160	110	70	110
MERCURY	0.20 UJ	0.20 UJ	0.20 UJ	0.20 UJ	0.40 J	0.20 U	0.20 J	0.20 J	0.20 UJ	0.20 U
MOLYBDENUM	4.6 U	50 UJ	2.2 U	50 UJ	13 U	1 U	1 U	1 U	1 U	1 U
NICKEL	25 UJ	25 UJ	4.79 J	10.8 J	132 J	1.3	4 J	2.2 J	1 J	1.4
POTASSIUM	1700 U	2100 U	2700 J	1700 U	7500 J	2500 J	3800	3100	2500	3000
SELENIUM	10 UJ	2 J	2 J	2 J	2 U	2 J				





SURFACE WATER ANALYTICAL DATA FOR THE AREA A LANDFILL MONITORING PROGRAM  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 20 OF 30

ROUND LOCATION	14 SG-22	14 SG-23	14 SG-24	15 SG-18	15 SG-19	15 SG-19	15 SG-20	15 SG-21	15 SG-22	15 SG-23
NSAMPLE	2-SW22-02-20040602	2-SW23-02-20040602	2-SW24-02-20040603	2-SW18-04-20041005	2-SW19-04-20041006	2-SW19-04-20041006-D	2-SW20-04-20041006	2-SW21-04-20041006	2-SW22-04-20041006	2-SW23-04-20041006
SAMPLE	2-SW22-02	2-SW23-02	2-SW24-02	2-SW18-04	2-SW19-04	2-SW100604	2-SW20-04	2-SW21-04	2-SW22-04	2-SW23-04
MATRIX	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW
QC TYPE	NM	NM	NM	NM	NM	FD	NM	NM	NM	NM
SAMPLE DATE	20040602	20040602	20040603	20041005	20041006	20041006	20041006	20041006	20041006	20041006
Polynuclear Aromatic Hydrocarbons (ug/L)										
2-METHYLNAPHTHALENE	0.20 U	0.20 U	0.20 U	0.24	0.20 J	0.20 J	0.20 U	0.20 U	0.20 U	0.20 U
ACENAPHTHENE	0.20 UJ	0.20 UJ	0.20 U	0.23	0.32	0.31	0.20 U	0.21	0.20 U	0.20 U
ACENAPHTHYLENE	0.20 J	0.20 U	0.20 U	0.20 J	0.20 U	0.20 U	0.20 U	0.20 U	0.20 U	0.20 U
ANTHRACENE	0.20 U	0.20 U	0.20 U	0.20 J	0.20 UJ	0.20 J	0.20 U	0.20 U	0.20 U	0.20 U
BAP EQUIVALENT										
BENZO(A)ANTHRACENE	0.20 J	0.20 UJ	0.20 J	0.20 J	0.20 J	0.20 J	0.20 U	0.20 U	0.20 J	0.20 U
BENZO(A)PYRENE	0.20 J	0.20 U	0.20 J	0.20 J	0.20 UJ	0.20 J	0.20 U	0.20 U	0.20 J	0.20 U
BENZO(B)FLUORANTHENE	0.20 UJ	0.20 UJ	0.20 J	0.20 J	0.20 J	0.20 J	0.20 U	0.20 U	0.20 J	0.20 U
BENZO(G,H,I)PERYLENE	0.25 U	0.25 U	0.25 UJ	0.25 J	0.25 J	0.25 J	0.25 U	0.25 U	0.25 J	0.25 U
BENZO(K)FLUORANTHENE	0.20 J	0.20 UJ	0.20 J	0.20 J	0.20 J	0.20 J	0.20 U	0.20 U	0.20 J	0.20 U
CHRYSENE	0.20 J	0.20 U	0.20 J	0.20 J	0.20 J	0.20 J	0.20 U	0.20 U	0.20 J	0.20 U
DIBENZO(A,H)ANTHRACENE	0.20 U	0.20 U	0.20 U	0.20 J	0.20 U	0.20 U	0.20 U	0.20 U	0.20 U	0.20 U
FLUORANTHENE	0.21	0.20 U	0.26	0.20 J	0.20 J	0.24	0.20 J	0.20 J	0.20 J	0.20 J
FLUORENE	0.20 U	0.20 U	0.20 U	0.20 J	0.20	0.20	0.20 U	0.20 J	0.20 U	0.20 U
INDENO(1,2,3-CD)PYRENE	0.20 U	0.20 U	0.20 U	0.20 J	0.20 J	0.20 J	0.20 U	0.20 U	0.20 J	0.20 U
NAPHTHALENE	0.20 U	0.20 U	0.20 J	0.27 U	0.20 J	0.20 J	0.20 U	0.20 J	0.20 U	0.20 U
PHENANTHRENE	0.20 J	0.20 UJ	0.20 J	0.20 J	0.20	0.21	0.20 U	0.20 J	0.20 J	0.20 J
PYRENE	0.20 J	0.20 UJ	0.24	0.20 J	0.20 J	0.21	0.20 U	0.20 J	0.20 J	0.20 J
Pesticides/PCBs (ug/L)										
4,4'-DDD										
4,4'-DDE										
4,4'-DDT										
ALDRIN										
ALPHA-CHLORDANE										
ENDOSULFAN I										
ENDOSULFAN II										
ENDRIN ALDEHYDE										
GAMMA-CHLORDANE										
Total Inorganics (ug/L)										
ALUMINUM	2500	100 J	26000	510	470 J	310 J	200 J	210 J	2500 J	4100 J
ANTIMONY	2	1 J	1.3	1 J	1 J	1 J	1 U	1 J	2.9	1.3
ARSENIC	5.3	1 U	44.9	2.9	2.3	2.1	2.2	1.4	49.5	12
BARIUM	59.3	18.9	276.6	99.3	47.5	42	31.5	38.7	106.9	67.9
BERYLLIUM	1 U	1 U	1.1 J	1 J	1 U	1 U	1 U	1 U	1 J	1 J
CADMIUM	0.20	0.10 U	0.50	0.20 J	0.20 J	0.20 U	0.20 U	0.20 U	0.70	1
CALCIUM	11000	5300	9700	19000	15000	14000	13000	18000	22000	8700
CHROMIUM	5.3	1 J	34	1.5	1.8	2.3	1 J	1.2	7.7	6.9
COBALT	2.1	1 J	18.3	1.1	1 J	1 J	1.5	1 J	4.1	5.3
COPPER	25.9	1.5	109.9	6.8	13.6	8.6	3.8	2.9	52.1	39.3
IRON	34000 J	2400 J	77000 J	28000	21000	19000	5200	4800	33000	13000
LEAD	14.5	0.50 J	70.7	2.6	4.1	3.2	1.4	1.9	24.8	20.2
MAGNESIUM	2900	980	12000	3500	3500	3500	3300	4800	5900	3300
MANGANESE	390	110	1200	440	210	200	320	280	690	410
MERCURY	0.20 J	0.20 U	0.20 J	0.20 UJ	0.20 U	0.20 U	0.20 U	0.20 U	0.20 J	0.20 J
MOLYBDENUM	1.4	1 U	3.1	1 U	1 U	1 U	1 U	1 U	3.8 U	1.3
NICKEL	10.5	2.7	41.5	4.9	5.2	4.1	3.4	2.3	27.4	25.3
POTASSIUM	3300	2500 J	11000	2800	3200 J	2900 J	2600 J	3800 J	4300 J	4300 J
SELENIUM	2 J	2 U	2 U	2 U	2 U	2 J	2 U	2 U	2 J	2 U





SURFACE WATER ANALYTICAL DATA FOR THE AREA A LANDFILL MONITORING PROGRAM  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 23 OF 30

ROUND	15	16	16	16	16	16	16	16	16	17
LOCATION	SG-24	SG-18	SG-19	SG-19	SG-20	SG-21	SG-22	SG-23	SG-24	SG-18
NSAMPLE	2-SW24-04-20041005	2-SW18-02-20050601	2-SW19-02-20050601	2-SW19-02-20050601-D	2-SW20-02-20050601	2-SW21-02-20050601	2-SW22-02-20050601	2-SW23-02-20050601	2-SW24-02-20050602	2-SW18-04-20051207
SAMPLE	2-SW24-04	2-SW18-02	2-SW19-02	2-SW060501-01	2-SW20-02	2-SW21-02	2-SW22-02	2-SW23-02	2-SW24-02	2-SW18-04
MATRIX	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW
QC TYPE	NM	NM	NM	FD	NM	NM	NM	NM	NM	NM
SAMPLE DATE	20041005	20050601	20050601	20050601	20050601	20050601	20050601	20050601	20050602	20051207
<b>Polynuclear Aromatic Hydrocarbons (ug/L)</b>										
2-METHYLNAPHTHALENE	0.20 U	0.20 U	0.20 J	0.19 J	0.19 U	0.20 U				
ACENAPHTHENE	0.20 U	0.20 U	0.27	0.92	0.19 U	0.20 U				
ACENAPHTHYLENE	0.20 U	0.20 U	0.20 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.20 U
ANTHRACENE	0.20 U	0.20 U	0.20 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.20 U
BAP EQUIVALENT			0.2 U	0.19 U	0.19 U	0.19 U		0.19 U		
BENZO(A)ANTHRACENE	0.20 U	0.20 U	0.20 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.20 U
BENZO(A)PYRENE	0.20 U	0.20 U	0.20 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.20 U
BENZO(B)FLUORANTHENE	0.20 U	0.20 UJ	0.20 UJ	0.19 UJ	0.19 UJ	0.19 UJ	0.19 UJ	0.19 UJ	0.19 UJ	0.20 U
BENZO(G,H,I)PERYLENE	0.25 U	0.24 U	0.25 U	0.24 U	0.24 U	0.24 U	0.24 U	0.24 U	0.24 U	0.26 U
BENZO(K)FLUORANTHENE	0.20 U	0.20 U	0.20 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.20 U
CHRYSENE	0.20 J	0.20 U	0.20 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.20 U
DIBENZO(A,H)ANTHRACENE	0.20 U	0.20 U	0.20 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.20 U
FLUORANTHENE	0.20 J	0.20 U	0.20 U	0.19 U	0.19 U	0.19 U	0.24 J	0.19 U	0.19 U	0.20 U
FLUORENE	0.20 U	0.20 U	0.20 J	0.37	0.19 U	0.20 U				
INDENO(1,2,3-CD)PYRENE	0.20 U	0.20 U	0.20 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.20 U
NAPHTHALENE	0.20 U	0.20 U	0.20 U	0.36 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.20 U
PHENANTHRENE	0.20 U	0.20 U	0.20 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.20 U
PYRENE	0.20 U	0.20 U	0.20 U	0.19 U	0.19 U	0.19 U	0.19 J	0.19 U	0.19 U	0.20 U
<b>Pesticides/PCBs (ug/L)</b>										
4,4'-DDD										
4,4'-DDE										
4,4'-DDT										
ALDRIN										
ALPHA-CHLORDANE										
ENDOSULFAN I										
ENDOSULFAN II										
ENDRIN ALDEHYDE										
GAMMA-CHLORDANE										
<b>Total Inorganics (ug/L)</b>										
ALUMINUM	250	100 J	110	100 J	100 J	100 J	12000	400	2200	1600 V
ANTIMONY	1 J	1 J	1 J	1 J	1 J	1 J	7	1 J	1 J	1.1 V
ARSENIC	3.1	1 J	1 J	1 J	1 J	1 J	59.1	1 J	29	10.9 V
BARIUM	51.3	28	66.6	68.4	24.9	27.5	332.6	26.5	162.7	100 V
BERYLLIUM	1 U	2.5 U	2.5 U	2.5 U	2.5 U	2.5 U	5 U	2.5 U	5 U	5 J
CADMIUM	1 J	0.20 U	0.20 U	0.20 U	0.20 U	0.20 U	3.5	0.20	0.20 U	0.40 V
CALCIUM	5400	14000	23000	23000	12000	12000	14000	6300	13000	14000 V
CHROMIUM	1	1 U	1.1	1 J	1 U	1 U	38.8	1.1	4	5.6 V
COBALT	4.1	1 J	1 J	1 J	1 J	1 J	10.7	1.1	10.1	2.7 V
COPPER	24.2	1 J	5.1	4.5	1 J	1	270.6	4.8	13.3	19.2 V
IRON	4600	1700	50 J	21000	1200	1100	220000	6800	85000	180000 V
LEAD	2.5	0.50 J	1.1	1	0.50 J	0.60	130.8	2.3	8.7	9.5 V
MAGNESIUM	2200	4400	2800	4300	3400	3200	6100	1500	9700	29000 V
MANGANESE	330	130	200	200	30	70	590	90	1100	450 V
MERCURY	0.20 UJ	0.20 U	0.20 U	0.20 U	0.20 U	0.20 U	0.50 J	0.20 U	0.20 U	0.20 J
MOLYBDENUM	1 U	1 U	1 U	1 U	1 U	1 U	8.9	1 U	3.6	2.6 V
NICKEL	6.4	1.4	3.1	3.2	1 J	1 J	98.1	4.3	13.7	14.2 V
POTASSIUM	2800	2500 U	3900 U	3900 U	2500 U	2500 U	7000	2500 U	8000	4300 V
SELENIUM	2 U	2 J	2 J	2 J	2 J	2 J	7	2 J	2 J	2 V





SURFACE WATER ANALYTICAL DATA FOR THE AREA A LANDFILL MONITORING PROGRAM  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 26 OF 30

ROUND	17	17	17	17	17	17	17	17	18	18	18
LOCATION	SG-19	SG-19	SG-20	SG-21	SG-22	SG-23	SG-24	SG-19	SG-19	SG-20	
NSAMPLE	2-SW19-04-20051207	2-SW19-04-20051207-D	2-SW20-04-20051207	2-SW21-04-20051207	2-SW22-04-20051208	2-SW23-04-20051208	2-SW24-04-20051208	2-SW19-03-20060829	2-SW19-03-20060829-D	2-SW20-03-20060824	
SAMPLE	2-SW19-04	2-SW120705	2-SW20-04	2-SW21-04	2-SW22-04	2-SW23-04	2-SW24-04	2-SW19-03	2-SW-03-FD01	2-SW20-03	
MATRIX	SW	SW	SW	SW	SW	SW	SW	SW	SW	SW	
QC TYPE	NM	FD	NM	NM	NM	NM	NM	NM	FD	NM	
SAMPLE DATE	20051207	20051207	20051207	20051207	20051208	20051208	20051208	20060829	20060829	20060824	
<b>Polynuclear Aromatic Hydrocarbons (ug/L)</b>											
2-METHYLNAPHTHALENE	0.22 U	0.21 J	0.21 U	0.21 U	0.22 U	0.22 U	0.20 U	1.6	1.5	0.20 U	
ACENAPHTHENE	0.22 J	0.21 V	0.21 U	0.21 U	0.22 U	0.22 U	0.20 U	3.8	3.3	0.20 U	
ACENAPHTHYLENE	0.22 U	0.21 U	0.21 U	0.21 U	0.22 U	0.22 U	0.20 U	0.20 U	0.20 J	0.20 U	
ANTHRACENE	0.22 U	0.21 U	0.21 U	0.21 U	0.22 U	0.22 U	0.20 J	0.20	0.21	0.20 U	
BAP EQUIVALENT	0.37692	0.21 U	0.21 U	0.21 U	0.22 U	0.26557	0.24135	0.2 U	0.2 U	0.3627	
BENZO(A)ANTHRACENE	0.22 U	0.21 U	0.21 U	0.21 U	0.22 U	0.22 J	0.28 V	0.20 J	0.20 U	0.20 J	
BENZO(A)PYRENE	0.22 U	0.21 U	0.21 U	0.21 U	0.22 U	0.22 U	0.42 V	0.20 U	0.20 U	0.20 J	
BENZO(B)FLUORANTHENE	0.22 U	0.21 U	0.21 U	0.21 U	0.22 U	0.22 U	0.78 V	0.20 U	0.20 U	0.20 J	
BENZO(G,H,I)PERYLENE	0.27 J	0.26 U	0.26 U	0.26 U	0.28 U	0.27 U	0.37 V	0.25 U	0.25 U	0.25 J	
BENZO(K)FLUORANTHENE	0.22 U	0.21 U	0.21 U	0.21 U	0.22 U	0.22 U	0.51 V	0.20 J	0.20 U	0.20 J	
CHRYSENE	0.22 J	0.21 U	0.21 U	0.21 U	0.22 U	0.22 J	0.54 V	0.20 U	0.20 U	0.20 J	
DIBENZO(A,H)ANTHRACENE	0.22 J	0.21 U	0.21 U	0.21 U	0.22 U	0.22 U	0.20 J	0.20 U	0.20 U	0.20 U	
FLUORANTHENE	0.22 J	0.21 J	0.21 U	0.21 J	0.22 U	0.22 J	1.1 V	0.27	0.25	0.20 J	
FLUORENE	0.22 J	0.21 J	0.21 U	0.21 U	0.22 U	0.22 U	0.20 U	2.1	1.8	0.20 U	
INDENO(1,2,3-CD)PYRENE	0.22 J	0.21 U	0.21 U	0.21 U	0.22 U	0.22 U	0.37 V	0.20 U	0.20 U	0.20 J	
NAPHTHALENE	0.22 U	0.21 J	0.21 U	0.21 U	0.22 U	0.22 U	0.20 U	6	5.8	0.20 U	
PHENANTHRENE	0.22 J	0.21 J	0.21 U	0.21 U	0.22 U	0.22 J	0.32 V	1.3	1.3	0.20 J	
PYRENE	0.22 J	0.21 J	0.21 U	0.21 U	0.22 U	0.22 J	0.86 V	0.20 J	0.20 J	0.20 J	
<b>Pesticides/PCBs (ug/L)</b>											
4,4'-DDD											
4,4'-DDE											
4,4'-DDT											
ALDRIN											
ALPHA-CHLORDANE											
ENDOSULFAN I											
ENDOSULFAN II											
ENDRIN ALDEHYDE											
GAMMA-CHLORDANE											
<b>Total Inorganics (ug/L)</b>											
ALUMINUM	320 V	360 V	100 J	220 V	1300 V	1100 V	20000 V	100 J	100 J	910	
ANTIMONY	1 J	1 J	1 J	1 J	19 V	1 J	15 V	1 J	1 J	1 J	
ARSENIC	1.6 V	1.9 V	1 J	1 J	17 V	19 V	127.4 V	1 J	1 J	4.1 J	
BARIUM	50 V	60 V	20 V	20 V	70 V	30 V	460 V	120	100	30	
BERYLLIUM	5 J	5 J	5 J	5 J	5 J	5 J	5 U	2.5 U	2.5 U	2.5 U	
CADMIUM	0.20 J	0.20 J	0.20 J	0.20 J	0.60 V	0.40 V	13 V	0.20 U	0.20 U	0.20	
CALCIUM	19000 V	19000 V	8900 V	9800 V	18000 V	8300 V	20000 V	100 J	40000	8800	
CHROMIUM	1 J	1.5 V	1 J	1.1 V	4.3 V	2.1 V	37 V	1 U	1 U	1.8	
COBALT	1 J	1 J	1 J	1 J	2 V	2.5 V	52.6 V	1 J	1 J	1.7	
COPPER	1 J	11.6 V	2 V	2.7 V	34.7 V	10 V	143.7 V	1 J	3.3 J	10.1 J	
IRON	50 J	26000 V	1200 V	1900 V	38000 V	8400 V	35000 V	50 J	18000	6400	
LEAD	3.8 V	4.6 V	0.60 V	1.6 V	19.3 V	5.2 V	102 V	1.2	1.2	5.1	
MAGNESIUM	3400 V	3400 V	2600 V	2800 V	3600 V	2300 V	13000 V	6000	5500	2400	
MANGANESE	160 V	170 V	60 V	110 V	320 V	190 V	3600 V	400	370	300	
MERCURY	0.20 UJ	0.20 U	0.20 U	0.20 U	0.20 J	0.20 U	0.30 U	0.20 U	0.20 U	0.20 U	
MOLYBDENUM	2 J	2 J	2 J	2 J	2. V	2 J	10.2 V	2 U	2 J	2 J	
NICKEL	4.5 V	5.2 V	1.3 V	1.6 V	16.8 V	9.7 V	71.5 V	3.4	3.7	6.2	
POTASSIUM	2500 J	5300 V	3700 V	4400 V	4600 V	2500 V	14000 V	2500 J	8400 J	2700 J	
SELENIUM	1 U	1 J	1 U	1 U	1 V	1 J	5 V	1 J	1 J	1 J	



SURFACE WATER ANALYTICAL DATA FOR THE AREA A LANDFILL MONITORING PROGRAM  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 28 OF 30

ROUND	18	18	19	19	19	19	19
LOCATION	SG-21	SG-23	SG-19	SG-19	SG-20	SG-21	SG-23
NSAMPLE	2-SW21-03-20060824	2-SW23-03-20060830	2-SW19-04-20061212	2-SW19-04-20061212-D	2-SW20-04-20061212	2-SW21-04-20061212	2-SW23-04-20061212
SAMPLE	2-SW21-03	2-SW23-03	2-SW19-04	2-SW-04-FD01	2-SW20-04	2-SW21-04	2-SW23-04
MATRIX	SW	SW	SW	SW	SW	SW	SW
QC TYPE	NM	NM	NM	FD	NM	NM	NM
SAMPLE DATE	20060824	20060830	20061212	20061212	20061212	20061212	20061212
CONTRACTOR	ECC	ECC	ECC	ECC	ECC	ECC	ECC
<b>Volatiles (ug/L)</b>							
1,2,4-TRICHLOROBENZENE							
2-BUTANONE							
ACETONE							
BENZENE							
CARBON DISULFIDE							
CHLOROBENZENE							
CHLOROMETHANE							
ETHYLBENZENE							
METHANE							
METHYLENE CHLORIDE							
TETRACHLOROETHENE							
TOLUENE							
TOTAL XYLENES							
TRICHLOROETHENE							
<b>Semivolatile Organics (ug/L)</b>							
1-METHYLNAPHTHALENE							
2,4-DIMETHYLPHENOL	10 U	10 U	10 U	10 U	10 U	10 U	10 U
2-METHYLNAPHTHALENE							
2-METHYLPHENOL	6 U	6 U	6.3 U	6.2 U	6.3 U	6.3 U	6.2 U
3&4-METHYLPHENOL	6 U	6 U	6.3 U	6.2 U	6.3 U	6.3 U	6.2 U
4-METHYLPHENOL							
4-NITROANILINE	7 U	7 U	7.4 U	7.2 U	7.4 U	7.4 U	7.2 U
ACENAPHTHENE							
ACENAPHTHYLENE							
ANTHRACENE							
BENZO(A)ANTHRACENE							
BENZO(A)PYRENE							
BENZO(B)FLUORANTHENE							
BENZO(G,H,I)PERYLENE							
BENZO(K)FLUORANTHENE							
BENZOIC ACID	50 U	50 U	53 U	52 U	53 U	53 U	52 U
BIS(2-ETHYLHEXYL)PHTHALATE	10 U	10 U	5.3 U	5.2 U	5.3 U	5.3 U	5.2 U
BUTYL BENZYL PHTHALATE	5 U	5 U	5.3 U	5.2 U	5.3 U	5.3 U	5.2 U
CARBAZOLE	5 U	5 U	5.3 U	5.2 U	5.3 U	5.3 U	5.2 U
CHRYSENE							
DI-N-BUTYL PHTHALATE	5 U	5 U	5.3 U	5.2 U	5.3 U	5.3 U	5.2 U
DI-N-OCTYL PHTHALATE	5 U	5 U	5.3 U	5.2 U	5.3 U	5.3 U	5.2 U
DIBENZO(A,H)ANTHRACENE							
DIETHYL PHTHALATE	5 U	5 U	5.3 U	5.2 U	5.3 U	5.3 U	5.2 U
FLUORANTHENE							
FLUORENE							
INDENO(1,2,3-CD)PYRENE							
NAPHTHALENE							
PHENANTHRENE							
PHENOL	7 U	7 U	7.4 U	7.2 U	7.4 U	7.4 U	7.2 U
PYRENE							



SURFACE WATER ANALYTICAL DATA FOR THE AREA A LANDFILL MONITORING PROGRAM  
 SITE 2B - AREA A WETLAND  
 NSB-NLON, GROTON, CONNECTICUT  
 PAGE 30 OF 30

ROUND	18	18	19	19	19	19	19
LOCATION	SG-21	SG-23	SG-19	SG-19	SG-20	SG-21	SG-23
NSAMPLE	2-SW21-03-20060824	2-SW23-03-20060830	2-SW19-04-20061212	2-SW19-04-20061212-D	2-SW20-04-20061212	2-SW21-04-20061212	2-SW23-04-20061212
SAMPLE	2-SW21-03	2-SW23-03	2-SW19-04	2-SW-04-FD01	2-SW20-04	2-SW21-04	2-SW23-04
MATRIX	SW	SW	SW	SW	SW	SW	SW
QC TYPE	NM	NM	NM	FD	NM	NM	NM
SAMPLE DATE	20060824	20060830	20061212	20061212	20061212	20061212	20061212
SILVER	1 U	1 U	1 UJ	1 J	1 U	1 U	1 U
SODIUM	61000	19000	44200	40700	34400	35800	19500
THALLIUM	1 U	1 U	1 U	1 U	1 U	1 U	1 U
VANADIUM	1.1	2.6	4.1	3	1.8	6.8	1.5
ZINC	22.7	558.4	97.7	89	92.5	118.3	493.1
<b>Filtered Inorganics (ug/L)</b>							
ALUMINUM	100 U	100 U	100 U	100 U	100 U	100 U	100 U
ANTIMONY	1 U	1 U	1 U	1 U	1 U	1 U	1 U
ARSENIC	1 J	3.3	1 J	1 J	1 J	1 J	1 J
BARIUM	30	30	36	32	16	16	14
BERYLLIUM	2.5 U	2.5 U	4 U	4 U	4 U	4 U	4 U
CADMIUM	0.20 U	0.20 U	0.20 J	0.20 UJ	0.20 U	0.20 U	0.20 U
CALCIUM	15000	8600	15000	14000	8100	8400	5700
CHROMIUM	1 U	1 U	1 U	1 U	1 U	1 U	1 U
COBALT	1 J	2.4	1 U	1 U	1 U	1 U	1 J
COPPER	1 J	62.8	1 J	1 J	1 J	1 J	1 U
IRON	160	50 J	1900 J	50 UJ	100	80	50 U
LEAD	0.50 U	0.50 U	0.50 U	0.50 U	0.50 U	0.50 U	0.50 U
MAGNESIUM	4000	1800	3000	2400	2100	2100	1400
MANGANESE	100	310	78	72	23	32	29
MERCURY	0.20 U	0.20 U	0.20 J	0.20 J	0.20 J	0.20 J	0.20 J
MOLYBDENUM	2 U	2 U	2 U	2 U	2 U	2 U	2 U
NICKEL	1 J	4.4	1	1 J	1 J	1 J	1
POTASSIUM	3300 J	3000 J	4100	3400	3000	2700	2500 J
SELENIUM	1 J	1 U	1 J	1 J	1 J	1 J	1 U
SILVER	1 U	1 U	1 U	1 U	1 U	1 U	1 U
SODIUM	57000	15000	48000	40000	32000	33000	17000
THALLIUM	1 U	1 U	1 U	1 U	1 U	1 U	1 U
VANADIUM	1 U	1 U	1 J	1 UJ	1 U	1 U	1 U
ZINC	5.6	405.3	14	16	11	11	99
<b>Miscellaneous Parameters</b>							
ALKALINITY (MG/L)	55	28	41	43	23	24	11
CARBONATE ALKALINITY (MG/L)							
CHEMICAL OXYGEN DEMAND (MG/L)	60	95	36	20 J	52	130	140
CHLORIDE (MG/L)	93	30	92	67	54	53	26
HARDNESS (MG/L)	57	33	48	47	32	36	21
HYDROGEN (NMO/L)							
SULFATE (MG/L)	10 U	10 U	11	12	13	13	10
SULFIDE (MG/L)							
TOTAL DISSOLVED SOLIDS (MG/L)	250	140	200	200	170	160	99
TOTAL ORGANIC CARBON (MG/L)	10	12	7.3	4.8	2.7	5.1	4.3
TOTAL SUSPENDED SOLIDS (MG/L)	30	22	44	44	130	140	100
<b>Field Parameters</b>							
DISSOLVED OXYGEN (MG/L)							
DISSOLVED OXYGEN - METER (MG/L)							
MANGANESE (MG/L)							
OXIDATION REDUCTION POTENTIAL (MV)							
PH							
SALINITY (NG/L)							
SPECIFIC CONDUCTANCE (MS/CM)							
TEMPERATURE (C)							
TURBIDITY (NTU)							

**APPENDIX E**

**LABORATORY STATEMENT OF WORK**

## ATTACHMENT NO. 2

### STATEMENT OF WORK/PRICE TABLES

#### TECHNICAL SPECIFICATION FOR LABORATORY SERVICES NAVAL SUBMARINE BASE (NSB) NEW LONDON, GROTON, CONNECTICUT

#### COMPREHENSIVE LONG-TERM ENVIRONMENTAL ACTION - NAVY (CLEAN) CONTRACT N62472-03-D-0057, CONTRACT TASK ORDER (CTO) NO. 0439

#### PHASE 1 INVESTIGATION CHEMICAL ANALYSES

### 1.0 INTRODUCTION

Tetra Tech NUS, Inc. (TtNUS) under CLEAN Contract **N62472-03-D-0057**, is procuring a laboratory for NSB New London in support of a Phase 1 Investigation. Requested analyses include Target Compound List (TCL) Pesticide, and Polychlorinated Biphenyl (PCB) Organic Compounds; Target Analyte List (TAL) Metals; Total Organic Carbon (TOC); pH; and Polycyclic Aromatic Hydrocarbons (PAHs).

The laboratory performing these analyses must be certified in the state of Connecticut.

After award, the laboratory will be required to submit all relevant practical quantitation limits (PQLs), method detection limits (MDLs), Standard Operating Procedures (SOPs) and all relevant precision and accuracy limits for all preparation and analysis methods required under this scope of work. The laboratory will also be asked to provide tabular information for inclusion in the Quality Assurance Project Plan (QAPP). The QAPP will be prepared according to the Uniform Federal Policy (UFP) for QAPPs (March 2005) and utilize the UFP QAPP worksheets 1 through 37.

### 2.0 SAMPLE INFORMATION

Samples will be shipped to the laboratory during in Fall or Winter of 2007.

The approximate number of samples to be submitted, the type of analyses to be conducted, and the analytical methods to be used are summarized in the attached Table 1.

Field duplicate samples will be submitted with "blinded" identification to the laboratory. The field crew will designate samples (one per twenty samples of like matrices) upon which matrix spike/matrix spike duplicate (organic analyses) and matrix spike/laboratory duplicate (inorganic analyses) are to be performed; additional volumes of these samples will be provided as necessary.

All samples are expected to be of low or moderate contaminant concentration. The field crew will attempt to identify any potentially high concentration samples.

All positive identifications for gas chromatography (GC) analyses MUST be confirmed on a second column that possesses retention characteristics different than those exhibited by the primary column. Identification using a single column with dual detectors does not meet second column confirmation requirements. Confirmed positive results less than the reporting limit but greater than the Method Detection Limit (MDL) must be reported by the laboratory, the laboratory must "J" flag these results. For SW-846 methodology, the higher of two results generated from dual column analyses must be reported as the primary result by the laboratory unless RPDs between columns are greater than 40% and an interference is found to be present or there is some other justifiable reason to select the lower of the two results. This provides the data user with the most conservative result. The use of CLP criteria or the argument that pesticide interferences are usually positive in nature are not justifications for selection of the lower result.

### 3.0 ANALYSIS/REPORTING INFORMATION

8/10/2007  
Rev. 0  
KAC

**TECHNICAL SPECIFICATION FOR LABORATORY SERVICES  
CONTRACT N62472-03-D-0057, CTO 0439  
NSB NEW LONDON, GROTON. CT  
CHEMICAL ANALYSES  
PAGE 2**

**Data package deliverables must be submitted as a hardcopy and PDF (2 copies/2 CDs).** The analytical requirements and approximate number of samples to be submitted for analysis are detailed in Table 1. Any analysis and reporting requirements addressed in the DOD Quality Systems Manual (January 2006) and the requested methods must be followed. Additionally, it is a requirement of TtNUS that the associated PDF data packages for PAHs, pesticide, PCB, and metals analysis must meet Contract Laboratory Program (CLP) format, reporting, and PDF data package deliverable requirements. The PDF data packages for all the remaining analyses must be fully validatable and contain raw data, summary forms for all sample and laboratory method blank data, and summary forms containing all method specific quality control (results, recoveries, relative percent differences, relative standard deviations, and/or percent differences etc.).

**Additionally, each PDF data package must contain a summary data package. This summary data package shall consist of only the summary forms (i.e., for CLP Forms 1 through 15 and non-CLP it shall be the CLP-like equivalent of Forms 1 through 15).**

**Attachment A details the required target compound list and provides a space for bidding labs to fill in their reporting limits.** Attachment B details the required summary forms for CLP-like data packages and requirements for organization/bookmarking of PDF data packages.

Nondetected metals results must be reported to the Instrument Detection Limits (IDLs). Nondetected Organic compound results must be reported down to the method reporting limits; however, positive results must be reported down to the Method Detection Limits (MDLs). Positive results reported at concentrations between the reporting limit and the MDL must be qualified with a "J".

**The PDF data package deliverable must contain a detailed case narrative for all analytical fractions. This case narrative must also include the Contract Task Order (CTO) number, the site name, and the TtNUS Project Manager's name. Data from all analytical runs (i.e., original, dilution, re-analysis) must be reported. All soil and sediment matrix sample results shall be reported on an adjusted dry-weight basis.**

As stipulated in the CLEAN Master Services Agreement (MSA), Sample Delivery Group (SDG) and fractionally-specific text (TXT) files containing all environmental sample and field quality control blank analysis results must be generated in accordance with the requirements outlined in Attachment C of this specification.

**As part of the laboratory case narrative, it is required that the Laboratory Quality Assurance Manager sign an attestation statement verifying that all electronic diskette deliverables exactly match the data summary forms (i.e. Form Is).**

**Maximum holding time allowances, as defined in the following table, are to be strictly observed. Calculation of holding time is in calendar days and is to begin from the time of collection. The holding times are as follows:**

<b>Analyses</b>	<b>Holding Time</b>
TCL Pesticide, PCB, and PAHs Organic	7 days to extraction (aqueous); 14 days until

8/10/2007  
Rev. 0  
KAC

**TECHNICAL SPECIFICATION FOR LABORATORY SERVICES**  
**CONTRACT N62472-03-D-0057, CTO 0439**  
**NSB NEW LONDON, GROTON. CT**  
**CHEMICAL ANALYSES**  
**PAGE 3**

<b>Analyses</b>	<b>Holding Time</b>
Compounds	extraction (solids); 40 days to analysis
pH	Analyze immediately
TOC	14 days to analysis
TAL Metals	6 months to analysis except mercury which is 28 days to analysis and cyanide which is 14 days to analysis

These holding times are based on 40 CFR 136, data validation criteria, and method specific requirements, and are measured from date of collection. The holding time criteria depicted apply to all analyses necessary to successfully determine the contaminant level contained in the sample. Hence, the holding time criteria apply to any/all subsequent sample dilutions and re-analyses.

The TtNUS Project Manager for this project is Mr. Aaron Bernhardt and he must be contacted in the event of any laboratory problems that could impact project deadlines (i.e., late deliverables, technical problems in the lab that could lead to late deliverables.) To ensure good communication it is required that the laboratory's appointed project manager contact Mr. Bernhardt once a week for the entire project duration.

Contact information for Mr. Bernhardt is as follows:

Tetra Tech NUS, Inc.  
Foster Plaza 7  
661 Andersen Drive  
Pittsburgh, PA 15220  
Phone: 412-921-8433  
Fax: 412-921-4040  
e-mail: [aaron.bernhardt@ttnus.com](mailto:aaron.bernhardt@ttnus.com)

**Analytical data turnaround times are to be measured from receipt of each sample shipment.** All PDF analytical data package (2 CDs) and associated electronic (TXT) deliverables are due to the TtNUS Sample Management Coordinator, Ms. Amy Thomson, within the standard MSA turnaround term of 21 calendar days from receipt of the last sample in a Sample Delivery Group (SDG). Additionally all SDGs must contain 20 samples. The frequency in which SDGs contain less than 20 samples should be minimal. All PDF data packages and electronic deliverables must be received at the same time or the deliverable will be considered incomplete and payment deductions may be imposed. **Additionally the laboratory Project Manager must fax copies of chain of custody forms to Ms. Thomson as samples are received by the laboratory.**

Contact information for Ms. Thomson follows:

Tetra Tech NUS, Inc.  
Foster Plaza 7  
661 Andersen Drive

8/10/2007  
Rev. 0  
KAC

TECHNICAL SPECIFICATION FOR LABORATORY SERVICES  
CONTRACT N62472-03-D-0057, CTO 0439  
NSB NEW LONDON, GROTON. CT  
CHEMICAL ANALYSES  
PAGE 4

Pittsburgh, PA 15220  
Phone: 412-921-8182  
Fax: 412-921-4040  
e-mail: [amy.thomson@ttnus.com](mailto:amy.thomson@ttnus.com)

#### 4.0 PERIOD OF PERFORMANCE/BOTTLEWARE INFORMATION

All samples will be shipped to the laboratory via express carrier within 24-hours of collection. Please circle the Yes or No at the bottom of Table 1 which will indicate if the laboratory will provide courier service at no extra charge. Samples will be shipped to the laboratory in the Fall or Winter of 2007.

**Bottleware shipments will be coordinated at a later date.**

The laboratory is to provide all necessary sample containers (**plus approximately 10% extra for breakage**). All sample containers must meet ICHM series 300 cleanliness criteria (or equivalent), and documentation of certified cleanliness must be provided. All of the appropriate sample bottleware must be pre-preserved. The bottleware must be shipped to the designated location in Coleman-like coolers. **The laboratory must also provide any extra coolers needed for return shipment of sample to the laboratory for analysis.** The laboratory is also requested to provide a packing slip indicating the analytical parameters for which each container type is designated, sample labels, and chain-of-custody forms.

**The laboratory must provide Material Safety Data Sheets (MSDSs) for all preservatives sent with each bottleware shipment to the field. MSDSs must be representative of the chemicals provided as preservatives with regard to mixtures and/or purity of the chemicals. For example if a 35% sulfuric acid solution is the preservative, the MSDS provided should be for 35% sulfuric acid solution not 96% sulfuric acid.**

#### 5.0 ADDITIONAL COMMENTS/CONTACTS

Within a laboratory, internal transfers of samples, extracts, and digestates must be accomplished and documented as controlled custody transfers. The laboratory must maintain documentation that supports an unbroken chain of custody for samples, digestates and extracts from time of receipt or production in the laboratory until disposal.

The laboratory is to provide a minimum of one (1) year storage of sample extracts and sixty (60) days storage of intact sample aliquots, as stipulated in the MSA. **Additionally, the laboratory must store hardcopy and/or PDF data packages for 5 years.**

All analyses conducted under this subcontract assignment are to be performed at the solicited facility only. The laboratory is not permitted to lower-tier subcontract these analyses, or analyze these samples at a corporate facility other than the facility stipulated without prior notification and consent from the CLEAN Subcontracting Officer.

The unit cost for analysis is to include compensation for containers, preservatives, coolers, shipping costs, storage, disposal, and laboratory quality control analyses (such as matrix spike, matrix spike duplicate, laboratory duplicate, and laboratory control sample analyses.) These items are not to be billed as separate line items.

8/10/2007  
Rev. 0  
KAC

TECHNICAL SPECIFICATION FOR LABORATORY SERVICES  
CONTRACT N62472-03-D-0057, CTO 0439  
NSB NEW LONDON, GROTON. CT  
CHEMICAL ANALYSES  
PAGE 5

Technical, quality assurance, and data format concerns are to be directed to Ms. Kelly Carper at 412-921-7273 or via e-mail [kelly.carper@ttnus.com](mailto:kelly.carper@ttnus.com). Ms. Carper must be contacted and informed of any difficulties encountered during the conduct of the requested analyses.

Questions regarding electronic diskette deliverable concerns are to be directed to Mr. Ricky DePaul at 412-921-7112 or via e-mail [ricky.depaul@ttnus.com](mailto:ricky.depaul@ttnus.com). Mr. DePaul must be contacted and informed of any difficulties encountered preparing the required electronic deliverables.

Contract concerns, and response to this solicitation, are to be directed to:

Ms. Meg Price  
CLEAN Subcontracting Officer  
Tetra Tech NUS, Inc.  
234 Mall Boulevard, Suite 260  
King of Prussia, PA 19406  
Phone: 610-491-9688  
Fax: 610-491-9646  
e-mail: [meg.price@ttnus.com](mailto:meg.price@ttnus.com)

**Triplicate copies of invoices associated with the analyses contracted herein are to be submitted to the attention of the Accounting Supervisor:**

Tetra Tech NUS, Inc.  
661 Andersen Drive, Foster Plaza 7  
Pittsburgh, PA 15220  
Phone: 412-921-8506  
Fax: 412-921-4040

Please confirm the laboratory's ability to perform the methodologies requested at the analyte detection limits indicated. Also confirm available laboratory capacity, and complete/confirm the costing information indicated in Table 1. **All costing information must reflect the terms and conditions established by the 2007 CLEAN MSA.**

8/10/2007  
Rev. 0  
KAC

**TABLE 1  
NUMBER OF SAMPLES/ANALYTICAL METHODS  
NSB NEW LONDON, GROTON, CT  
CTO 0439, PHASE I**

Matrix	Parameter	Method	# Samples	Unit Price	Total Cost
Please fill in IDLs and reporting limits in Attachment A					
Aqueous Field Quality Control	PAHs	SW-846 8270C SIM	2	\$	\$
	TCL Pesticide Organic Compounds	SW-846 8081A	2	\$	\$
	TCL PCB Organic Compounds	SW-846 8082	2	\$	\$
	TAL Metals	SW-846 6010B or 6020	2	\$	\$
Soil	PAHs	SW-846 8270C SIM	20	\$	\$
	TCL Pesticide Organic Compounds	SW-846 8081A	20	\$	\$
	TCL PCB Organic Compounds	SW-846 8082	20	\$	\$
	TAL Metals	SW-846 6010B or 6020	20	\$	\$
	TOC	Lloyd Kahn	20	\$	\$
	pH	SW-846 9045C	20	\$	\$

**TOTAL COST \$**

**Can the laboratory provide sample pick-up on site? YES or NO (circle one)**  
**If yes is there an additional charge and what is that charge? \_\_\_\_\_**

**Name of Laboratory \_\_\_\_\_**

**Signature \_\_\_\_\_**

**ATTACHMENT A**  
**Required Target Compound Lists**

<b>INORGANICS</b>	<b>IDL (mg/kg)</b>
Aluminum	
Antimony	
Arsenic	
Barium	
Beryllium	
Cadmium	
Calcium	
Chromium	
Cobalt	
Copper	
Iron	
Lead	
Magnesium	
Manganese	
Mercury	
Nickel	
Potassium	
Selenium	
Silver	
Sodium	
Thallium	
Vanadium	
Zinc	
<b>PESTICIDES</b>	<b>RL (ug/kg)</b>
4,4'-DDD	
4,4'-DDE	
4,4'-DDT	
Aldrin	
alpha-BHC	
alpha-chlordane	
beta-BHC	
delta-BHC	
Dieldrin	
Endosulfan I	
Endosulfan II	
Endosulfan sulfate	
Endrin	
Endrin aldehyde	
Endrin ketone	
gamma-BHC (lindane)	
gamma-chlordane	
Heptachlor	
Heptachlor epoxide	
Methoxychlor	
Toxaphene	

POLYCHLORINATED BIPHENYLS RL (ug/kg)	
Aroclor-1016	
Aroclor-1221	
Aroclor-1232	
Aroclor-1242	
Aroclor-1248	
Aroclor-1254	
Aroclor-1260	
Semivolatile Organic Compounds RL (ug/kg)	
1-methylnaphthalene	
2-methylnaphthalene	
Acenaphthene	
Acenaphthylene	
Anthracene	
Benzo(a)anthracene	
Benzo(a)pyrene	
Benzo(b)fluoranthene	
Benzo(g,h,i)perylene	
Benzo(k)fluoranthene	
Chrysene	
Dibenzo(a,h)anthracene	
Fluoranthene	
Fluorene	
Indeno(1,2,3-cd)pyrene	
Naphthalene	
Phenanthrene	
Pyrene	

**ATTACHMENT B**  
**Summary Form Requirements for PDF Deliverable and PDF Data Package Deliverables**

## PDF DATA PACKAGE DELIVERABLE REQUIREMENTS

The laboratory is to provide 2 compact disks (CDs) containing a PDF file in the following format:

1. Table of Contents
2. Case Narrative
3. Chain-of-Custody
4. Data Summary Package (contains summary of all CLP or CLP like Forms 1 through 14 per analytical fraction)
5. Analytical Fractions (VOA, SVOC, General Chemistry, etc.)
  - a. QC Summary (summary of all CLP or CLP like Forms 1 through 14 for a particular analytical fraction)
  - b. Raw Sample Data (includes all sample dilutions, sample re-analyses, QC samples, etc.)
  - c. Calibration Data (includes all initial and continuing calibrations)
  - d. Miscellaneous (includes extraction forms, IDLs, MDLs, etc.)

Each of the above sections should be bookmarked in the PDF for easy access.

### Summary Form Requirements for PDF data package deliverable for non-CLP Methods:

The following summary forms are required as part of the data package deliverable for SW-846 6020/6010B/7000A series for metals:

- Results Report** - must present all information presented on CLP FORM I.
- Initial and Continuing Calibration Summary** - must present all information presented on CLP FORM 2A.
- CRDL Summary** - must present all information presented on CLP FORM 2B.
- Blanks** - must present all information presented on CLP FORM 3.
- ICP Interference Check Sample Summary** - must present all information presented on CLP FORM 4.
- Matrix Spike Summary** - must present all information presented on CLP FORM 5A.
- Post Digestion Spike** - must present all information presented on CLP FORM 5P.
- Lab Duplicate Results** - must present all information presented on CLP FORM 6.
- LCS Summary** - must present all information presented on CLP FORM 7.
- MSA Summary (Method of Standard Addition)** - must present all information presented on CLP FORM 8.
- ICP Serial Dilution Summary** - must present all information presented on CLP FORM 9.
- Detection Limits** - must present all information presented on CLP FORM 10.
- Prep Log** - must present all information presented on CLP FORM 13.
- Analysis Run Log with Post Digestion Spike Results or tunes for ICP/MS** - must present all information presented on CLP FORM 14.
- Internal Standard forms for ICP/MS** - must present all information presented on CLP FORM 15.

Also must include: Instrument Calibration Records, Chain-of-Custody Forms, and Case Narrative.

**Summary Forms for SW-846, 8260B and 8270C (i.e., Any SW-846 GC/MS analysis of Volatile and Semivolatile Organic Compounds) should be presented in a CLP-Like format. The following Summary Forms must be included:**

- |                         |   |
|-------------------------|---|
| Result Summary          | One Sample per summary page.<br>Presentation of analytical results for both method blanks and environmental samples, date of collection, preparation, and analysis. Environmental samples should be identified with the field identification numbers on the COCs. |
| Surrogate Recovery Form | Present all information contained on CLP Form II.   |

Summary of Matrix Spike/Matrix Spike Duplicate Recovery	Present all information contained on CLP Form III.
Instrument Performance Check Summary Form - Mass Spec Tuning Form	Present all information Contained on CLP Form V.
Initial Calibration Summary	Present all information contained CLP Form VI.
Continuing Calibration Summary	Present All Information contained on CLP Form VII.
Internal Standard Area and Retention Time Summary	Present all information contained CLP Form VIII.

8/10/2007  
Rev. 0  
KAC

**Summary Forms for SW-846 8081A and 8082 Pesticide and Polychlorinated Biphenyl (PCB) Organic Compounds (and 8151A, 8141A, and all other SW-846 GC methods) should be presented in a CLP-Like format. The following Summary Forms must be included:**

Result Summary	One Sample per summary page. Presentation of analytical results for both method blanks and environmental samples, date of collection, preparation, and analysis. Environmental samples should be identified with the field identification numbers on the COCs.
Surrogate Recovery Form	Present all information contained on CLP Form II for both Analytical Columns.
Summary of Matrix Spike/Matrix Spike Duplicate Recovery	Present all information contained on CLP Form III.
Summary of Pesticide Initial Calibration of Single Component Analytes	Present all information contained on CLP Form VI-PEST-2.
Summary of Pesticide Calibration Verification	Present all information contained on CLP Form VII-PEST-1 and Form VII-PEST-2.
Pesticide Analytical Sequence	Present all information contained on CLP Form VIII-PEST.
Pesticide Identification Summary For Single Component Analytes and for Multiple Component Analytes	Present all information contained on CLP Form X PEST 1 and 2.

**ATTACHMENT C  
ELECTRONIC DATA DELIVERABLE REQUIREMENTS**

## ELECTRONIC DATA FORMAT REQUIREMENTS

### 1.0 INTRODUCTION

The laboratory is to provide a compact disk (CD) containing separate text (TXT) files in the format specified in this Attachment. The electronic deliverable includes all environmental samples, sample dilutions, sample reanalyses, and laboratory quality control samples. **All entries in the electronic deliverable must agree exactly with the final entries reported on the hardcopy data package sample result summaries.** The LAB\_RESULT for nondetects should be populated with sample quantitation limits. Any corrections made to the hardcopy data must also be made to the electronic file. Appropriate qualifiers as identified by the analytical protocol must also be designated; laboratory QC non-compliance codes are not to be depicted.

Each CD is to be properly labeled with the laboratory name, project name, file name(s), and laboratory point of contact. Electronic files should be delivered in the same fashion, as are the hard copy data packages. A separate .txt file shall be made for each analytical fraction (by method) and each sample delivery group (SDG). The files shall be named with the first character being the analytical fraction designator, followed by an underscore, followed by the SDG name. For example, the file for the volatile fraction for SDG TT001 should be named V\_TT001.TXT. Additionally, the laboratory must provide a hardcopy listing all electronic files saved to the CD, indicating what analytical fraction and matrix the file data contained therein pertain to. All electronic data deliverables are due within the same time established for the associated hardcopy data packages.

In addition, the laboratory QC officer must read and sign a copy of the Quality Assurance Review Form displayed on the next page of this Attachment. Electronic deliverables are not considered to be complete without the accompanying Quality Assurance Review Form.

I \_\_\_\_\_, as the designated Quality Assurance Officer, hereby attest that all electronic deliverables have been thoroughly reviewed and are in agreement with the associated hardcopy data. The enclosed electronic files have been reviewed for accuracy (including significant figures), completeness and format. The laboratory will be responsible for any labor time necessary to correct enclosed electronic deliverables that have been found to be in error. I can be reached at (\_\_\_\_\_) \_\_\_\_\_ if there are any questions or problems with the enclosed electronic deliverables.

Signature: \_\_\_\_\_ Title: \_\_\_\_\_ Date: \_\_\_\_\_

The analytical data shall be delivered electronically in an ASCII comma delimited (double quotes around text fields) text file (filename.txt). The exact structure of the database is described in the table below. It shall be the responsibility of the laboratory to ensure that all electronic entries are in strict accordance with the information provided on the Form I.

An example database shall be sent for review prior to the first electronic deliverable in .txt format. The example file will be examined for completeness and comments will be sent to the laboratory. Any questions regarding the electronic deliverable shall be directed to Ricky DePaul at Tetra Tech NUS (412)921-7112.

DATA FIELD	DATA TYPE	FIELD WIDTH	DATA FIELD DESCRIPTION
SAMPLE_NO	C	25	Field sample ID as listed on the chain-of-custody. The sample number indicated in this field should never be truncated. The only exception for this field not matching the chain-of-custody is for reanalyses, dilution, and matrix spike results in which a RE, DL, or MS suffix will be added to the sample number respectively.
MATRIX	C	2	Matrix as indicated on the Chain of Custody.
COLL_METH	C	2	"G" (Grab) or "CP" (Composite) as indicated on the Chain of Custody.
LAB_ID	C	15	Laboratory number for the given sample.
LABORATORY	C	25	Laboratory name.
BATCH_NO	C	10	Laboratory code for batch of samples included in a given run.
ASSOC_BLNK	C	15	Laboratory name of the method blank associated with that particular batch of samples.
QC_TYPE	C	10	Normal Environmental Sample = "NORMAL", Laboratory Duplicate = "DUPLICATE", Matrix Spike = "MS", Matrix Spike Duplicate = "MSD", Laboratory Control Sample = "LCS", Laboratory Control Sample Duplicate = "LCSD", Method Blank = "M_BLANK", Preparation Blank = "P_BLANK".
RUN_TYPE	C	12	Initial, dilution 1, dilution 2, dilution 3, reanalysis 1, reanalysis 2, reanalysis 3
RES_TYPE	C	5	Surrogate Recoveries = "SUR", Target Compound = "TRG", Internal standards = "IS", Tentatively Identified Compounds = "TIC"
SAMP_DATE	D	8	Date of sample collection as indicated on the Chain of Custody. Example: 11/07/93.

DATA FIELD	DATA TYPE	FIELD WIDTH	DATA FIELD DESCRIPTION
SAMP_TIME	T	5	Time of sample collection as indicated on the Chain of Custody. Reported as five character string.
REC_DATE	D	8	Date sample was received by the laboratory.
EXTR_DATE	D	8	Date sample was extracted or prepared by the laboratory.
ANAL_DATE	D	8	Date sample was analyzed by the laboratory.
RUN_NUMBER	N	2 (0)	The number of the analytical run for a given sample in sequence. For example, if a sample is diluted and reanalyzed, the original run number would be 1 and the reanalysis would be 2.
SDG	C	15	Sample delivery group identifier assigned by the laboratory. This number should <u>exactly</u> match the SDG designated on the hardcopy data package.
PROJECT_NO	C	10	Identification of Project Number or Contract Task Order (CTO) number.
PROJ_MNGR	C	25	The Tetra Tech NUS Project Manager's last name, followed by a comma, followed by the first initial of the Project Manager. Example: HUTSON, D.
PARAMETER	C	45	Chemical or analyte name <u>exactly</u> as reported on Form I.
CAS_NO	C	10	Chemical Abstract Service number for the parameter listed. The CAS number should be reported exactly as it is listed in publications such as the Merck Index. This field should be left blank for those parameters not having CAS numbers (e.g. Total Organic Carbon).
FRACTION	C	8	Metals = 'M', Volatiles = 'OV', Semivolatiles/BNAs = 'OS', Pesticides = 'PEST', Herbicides = 'HERB', Polychlorinated Biphenyls = 'PCB', Explosives = 'EXP', Any petroleum hydrocarbon or fuel = 'TPH', Radionuclide = 'RAD', Miscellaneous = 'MISC', Dioxin/Furans = 'DIOX
SORT	C	5	Leave this field blank. To be filled in by Tetra Tech NUS, Inc.
EXTR_METH	C	20	Extraction method used. Example: '5035' for SW-846 Method 5035.

DATA FIELD	DATA TYPE	FIELD WIDTH	DATA FIELD DESCRIPTION
ANAL_METH	C	20	Analytical method used to quantitate parameter concentrations as listed in the laboratory technical specification. Example: 8270C for SW-846 Method 8270C.
LAB_RESULT	N	20	Reported value in units specified in the UNITS field containing the proper number of significant digits. Nondetects must be reported as sample quantitation limits (i.e. reporting limits adjusted for sample volume, percent moisture, and dilution factors as appropriate). <b>The % Recovery for matrix spikes, laboratory control samples, and surrogates shall ALSO be placed in this field.</b>
UNITS	C	5	The units of measure as reported on the Form I.
LAB_QUAL	C	2	The laboratory qualifier as reported on the Form I. For example, a 'U' qualifier should be used for all nondetected results.
IDL	N	15 (6)	Instrument detection limit in units specified in the UNITS field.
MDL	N	15 (6)	Method detection limit in units specified in the UNITS field and method specified in the METHOD field.
CRDL_CRQL	N	15 (6)	Contract Required Detection/Quantitation Limit in the units specified in the UNITS field. RDL for non-CLP parameters.
DIL_FACTOR	N	6 (1)	Dilution factor.
PCT_MOIST	N	5 (1)	Percent moisture for soil samples; blank for water samples.
RET_TIME	T	10	Retention time of analyte. Required for TICs. Format requested as HHHH:MI:SS (e.g. 1 day - 1 hr -10 min as 25:10:00)
COMMENTS	C	20	Analytical result qualifier or comment other than that listed in the LAB_QUAL field. Example: 'Reanalysis'.

C = Character string (everything shall be reported in capital letters)

N = Numeric string (decimal places are in parentheses in field width column)

D = Date (Ex: 010/07/04)

T = Time HHHH:MI:SS (e.g. 1 day - 1 hr -10 min as 25:10:00)