

8/1/03-00256

SIGNATURE PAGE

Final
Work Plan for
Baseline Ecological Risk Assessment (Step 4)
Blows Creek Sites 3, 4, and 5

St. Juliens Creek Annex
Chesapeake, Virginia

Contract Task Order Number—0027
Contract Number N62470-95-D-6007
Navy CLEAN II Program

Prepared by

CH2M HILL

August 2003

Approved by: _____
William J. Friedmann, Jr., P.G.
Activity Manager

Date: _____

Approved by: _____
Roger Huddleston, P.G.
Senior Reviewer

Date: _____

Approved by: _____
Stephen Petron, Ph.D
Senior Reviewer

Date: _____

Contents

Acronyms and Abbreviations.....	v
1 Introduction	1-1
1.1 Work Plan Organization.....	1-2
2 Site Background and Overview of Proposed ERA Investigation	2-1
2.1 St. Juliens Creek Annex Background.....	2-1
2.2 Blows Creek Watershed Site Descriptions.....	2-1
2.3 Summary of Ecological Risks for Sites 3, 4, and 5 and Conceptual Model for Blows Creek	2-2
2.3.1 Summary of Ecological Risks for Sites 3, 4, and 5	2-2
2.3.2 Preliminary Conceptual Model for Blows Creek.....	2-3
2.4 Objectives of Blows Creek Investigation.....	2-5
3 Approach to Blows Creek Investigation.....	3-1
3.1 Phase I.....	3-1
3.2 Phase II.....	3-7
3.2.1 Tissue Residue	3-7
3.2.2 Subsurface Sediment.....	3-8
4 Investigation Tasks and Methodology	4-1
4.1 Field Investigation.....	4-1
4.1.1 Fieldwork Support	4-1
4.1.2 Field Investigation and Sampling Activities	4-2
4.1.3 Sample Designation	4-6
4.1.4 Surveying.....	4-6
4.1.5 Investigation-Derived Waste Management.....	4-6
4.2 Sample Analysis and Data Validation.....	4-6
4.2.1 Sample Analysis	4-8
4.2.2 Field Quality Control Procedures	4-8
4.2.3 Data Validation.....	4-10
5 Data Evaluation	5-1
5.1 Evaluation of Bioassay Outcomes.....	5-1
5.2 Evaluation of Chemical Analytical and Physical Data	5-2
5.2.1 Weight of Evidence Analysis.....	5-2
5.2.2 Evaluation of Chemicals Potentially Causing Bioassay Organism Response	5-3
5.3 Evaluation of Tissue Residue Data (Phase II).....	5-4
5.4 BERA Report.....	5-4
6 Project Management and Staffing	6-1
7 Project Schedule	7-1
8 References	8-1

Appendixes

- A Health and Safety Checklist and SOPs
- B Bioassay SOPs

Tables

3-1	Sediment Sample Location Objectives	3-3
4-1	Analytical Methods, Required Containers, Preservative, and Holding Times for Samples	4-3
4-2	Summary of Sample ID Scheme	4-7
4-3	General Requirements for QC Sample Collection	4-9
5-1	Determination of Toxicity—All Sediment Samples Compared to Control.....	5-2
5-2	Integration of Bioassay and Chemistry Outcomes – All Sediment Samples Locations	5-3
7-1	Project Schedule.....	7-1

Figures (Figures are located at the end of each section.)

- 1-1 Location of St. Juliens Creek Annex
- 1-2 Site Locations and Blows Creek Watershed
- 2-1 Sites 3, 4, and 5 Habitat Composition and Drainage
- 3-1 Sediment Sample Locations
- 7-1 Project Schedule

Acronyms and Abbreviations

µg/kg	Microgram per kilogram
µg/L	Microgram per liter
AOC	Area of Concern
ASTM	American Society for Testing Materials
AVS/SEM	acid-volatile sulfides/simultaneously extractable metals
BERA	Baseline Ecological Risk Assessment
BTAG	Biological Technical Assistance Group
CLEAN	Comprehensive Long-Term Environmental Action Navy
CLP	Contract Laboratory Program
COC	Chain of Custody
COPC	Chemicals contaminants of potential concern
CTO	Contract Task Order
DO	Dissolved oxygen
DQO	Data Quality Objectives
EE/CA	Engineering Evaluation/Cost Analysis
EPIC	Environmental Photographic Interpretation Center
ERA	Ecological Risk Assessment
ERS	Ecological Risk Screening
FS	Feasibility Study
GIS	Geographic Information System
GPS	Global Positioning System
HHRA	Human Health Risk Assessment
IDW	Investigation-derived waste
IR	Installation Restoration
LANTDIV	Atlantic Division, Naval Facilities Engineering Command
MFSP	Master Field Sampling Plan
mg/kg	Milligrams per kilogram
mg/L	Milligrams per liter
MHASP	Master Health and Safety Plan
MIDWMP	Master Investigation-Derived Waste Management Plan
MPP	Master Project Plan
MQAPP	Master Quality Assurance Project Plan
MS/MSD	Matrix spike/matrix spike duplicate
MWP	Master Work Plan

NAVFACENGCOM	Naval Facilities Engineering Command
PAH	Polycyclic aromatic hydrocarbon
PCB	Polychlorinated biphenyl
PPE	Personal protective equipment
QA/QC	Quality assurance/quality control
RI	Remedial Investigation
SERA	Screening Ecological Risk Assessment
SJCA	St. Juliens Creek Annex
SOP	Standard Operating Procedures
SSA	Site Screening Assessment
SVOC	Semivolatile organic compound
TAL	Target Analyte List
TCL	Target Compound List
TNT	Trinitrotoluene
TOC	total organic carbon
USEPA	U.S. Environmental Protection Agency
VOC	Volatile organic compounds
WP	Work Plan

SECTION 1

Introduction

This document presents a work plan (WP) for conducting Steps 5 and 6 of the eight-step Ecological Risk Assessment (ERA) process for Sites 3, 4, and 5 and the Blows Creek watershed at St. Juliens Creek Annex (SJCA), Chesapeake, Virginia. A regional location map of SJCA is shown on Figure 1-1 and the Blows Creek watershed is shown on Figure 1-2.

Blows Creek is located within the SJCA boundary (Figure 1-2). Runoff from the Craddock District of Portsmouth enters the SJCA and Blows Creek through a drainage pipe beneath the base fence line and patrol road. Sites 3, 4, and 5 at SJCA are currently believed to pose the greatest potential risk to Blows Creek due to the transport of contaminants from drainage ditches and swales associated with these sites. Therefore, the Navy is assessing the impacts primarily from Sites 3, 4, and 5. In the *Final Remedial Investigation (RI)/Human Health Risk Assessment (HHRA)/Ecological Risk Assessment (ERA) Report for Sites 3, 4, 5, and 6* (CH2M HILL 2003), potential ecological risks were identified for exposure of receptors to metals and polycyclic aromatic hydrocarbons (PAHs) in surface soil, sediments, and surface water. Concurrent with the Remedial Investigation (RI), the SJCA Partnering Team developed an Engineering Evaluation/Cost Analysis (EE/CA) for waste and debris removal at Site 3. The removal activities at Site 3 will be completed in early 2004. Presumptive remedies are currently under consideration for Sites 4 and 5. An additional active Installation Restoration (IR) site and Areas of Concern (AOC) located along Blows Creek have the potential to affect the Blows Creek watershed. The additional site and AOC are shown in Figure 1-2 and include the Environmental Photographic Interpretation Center (EPIC) AOC 1 and Site 19.

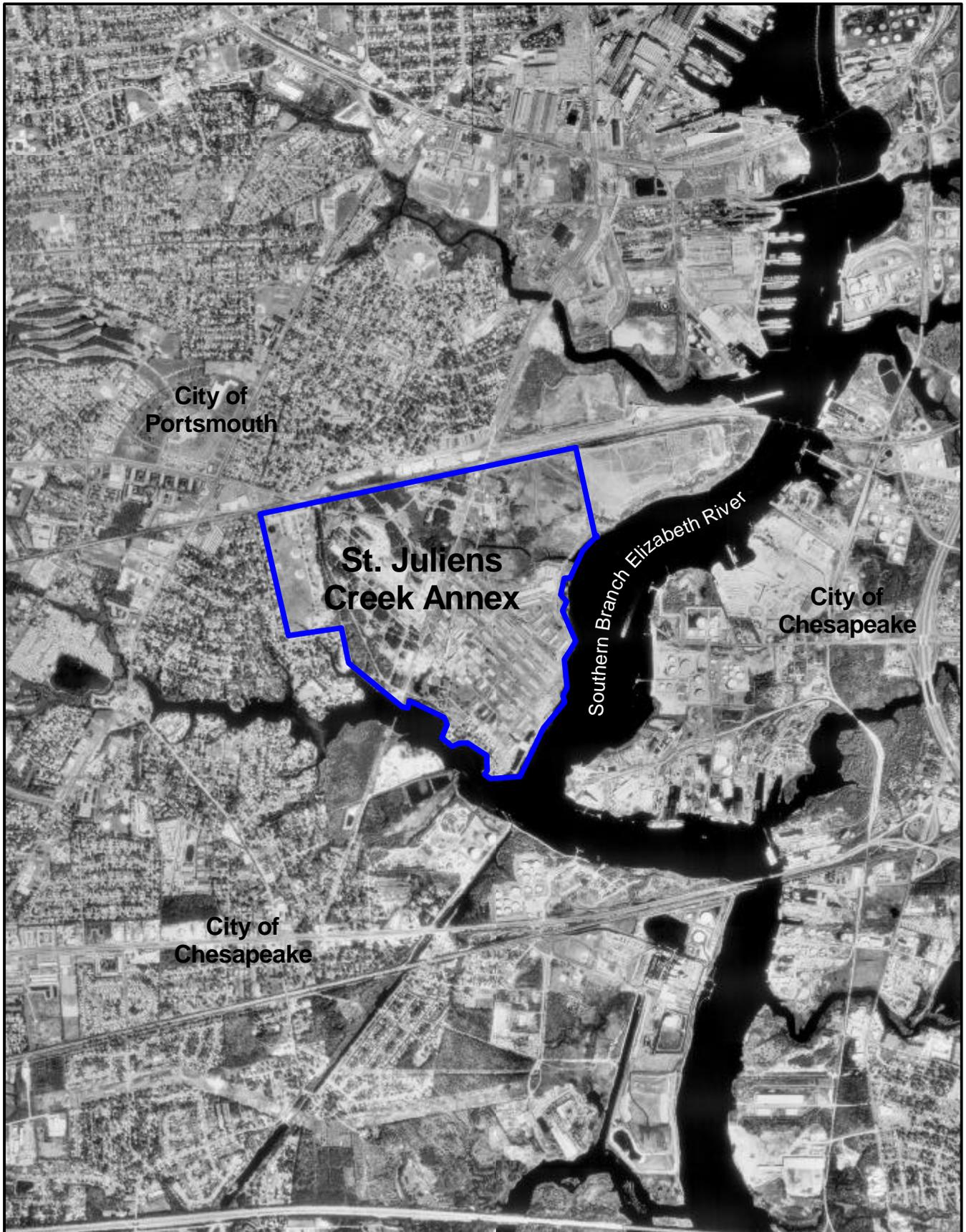
This WP outlines the phased site investigation that will be used to obtain site-sampling data to fill data gaps and address areas of uncertainty identified in the problem-formulation stage of the Baseline ERA (BERA) (Step 3b) for Sites 3, 4, and 5 and the Blows Creek watershed. The ERA process will use these data to identify risk associated with potential historical contributions to Blows Creek via transport in upland drainages. The data will aid in identifying whether contamination is present in Blows Creek and if it is site related. Additionally, other potential contaminant contributors to Blows Creek will be evaluated. Identifying the sources of potential contamination to Blows Creek will help to verify the remediation of Sites 3, 4, and 5. Although this WP focuses on the BERA, the collected data will also be reviewed for possible human health impacts from exposure to Blows Creek sediment, and this information will be addressed in the Feasibility Study (FS), as appropriate.

This WP was prepared under the Naval Facilities Engineering Command (NAVFACENGCOM) U.S. Naval Facilities Engineering Command, Atlantic Division (LANTDIV) Navy Contract N62470-95-D-6007, Navy Comprehensive Long-Term Environmental Action Navy (CLEAN), District III, Contract Task Order (CTO) 0027 in accordance with guidance provided by the U.S. Environmental Protection Agency (USEPA) (USEPA 1997) and the U.S. Navy (CNO 1999).

1.1 Work Plan Organization

This WP contains the following sections:

- Section 1—Introduction
- Section 2—Site Background and Overview of Proposed ERA Investigation
- Section 3—Approach to Blows Creek Investigation
- Section 4—Investigation Tasks and Methodology
- Section 5—Data Evaluation
- Section 6—Project Management and Staffing
- Section 7—Project Schedule
- Section 8—References



LEGEND

 St. Juliens Creek Annex



0 2000 4000 Feet



Figure 1-1
Location of St. Juliens Creek Annex
St. Juliens Creek Annex
Chesapeake, Virginia

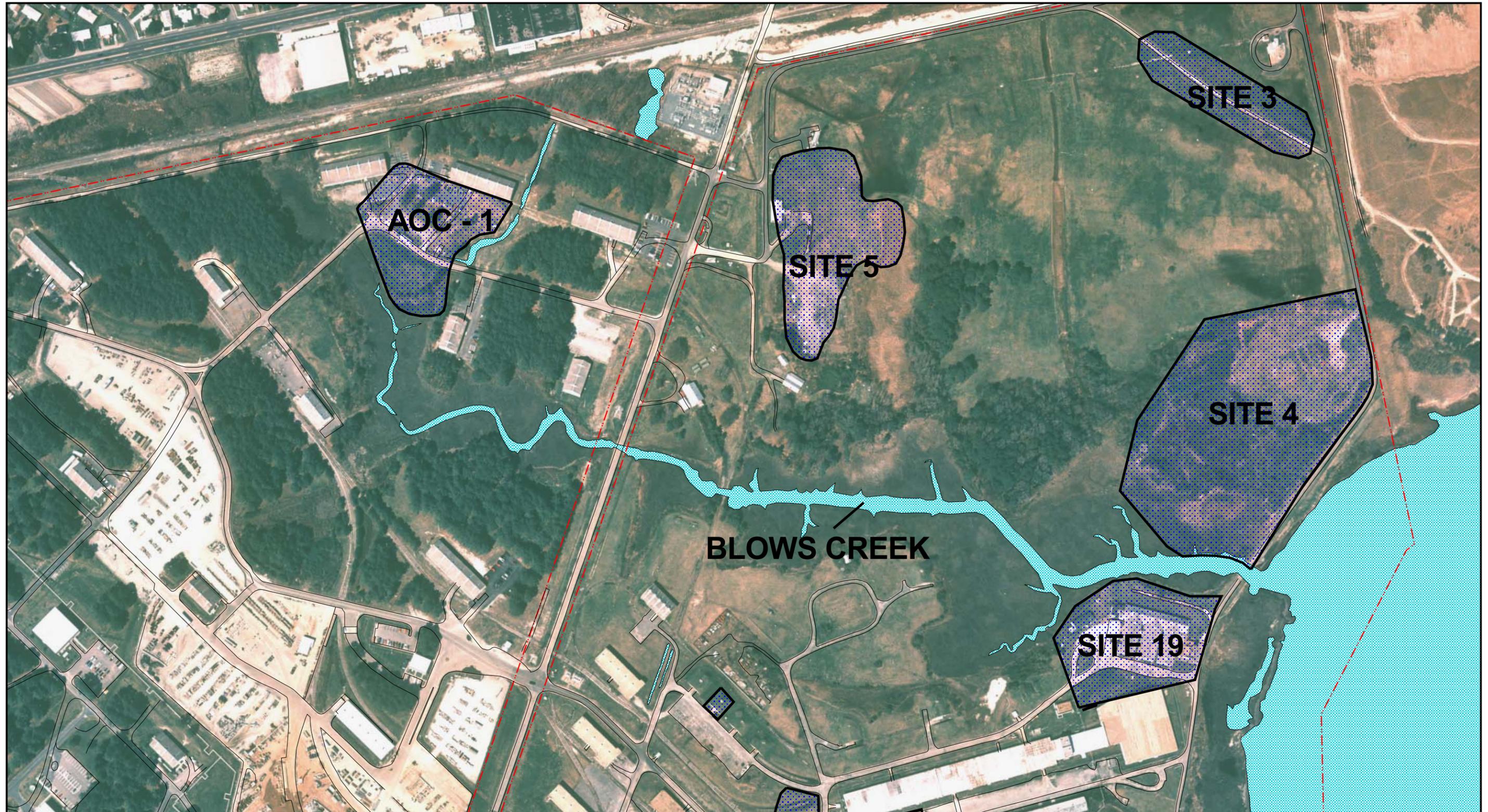


Figure 1-2
Site Locations and Blows Creek Watershed
Blows Creek BERA Work Plan
St. Juliens Creek Annex
Chesapeake, Virginia

SECTION 2

Site Background and Overview of Proposed ERA Investigation

2.1 St. Juliens Creek Annex Background

The SJCA facility is situated at the confluence of St. Juliens Creek and the Southern Branch of the Elizabeth River in the City of Chesapeake, located in southeastern Virginia (Figure 1-1). The facility covers about 490 acres and includes administrative buildings, wharf areas to the Elizabeth River, a central heating plant, numerous non-operational industrial facilities, and a radar-testing range.

The SJCA began operations as a naval ammunition facility in 1849 and has been used for activities such as the loading, assembling, and storage of ordnance and materials. SJCA has also been involved in non-ordnance operations, including degreasing operations, paint shops, machine shops, maintenance shops, pest-control shops, and fire-training operations. Many of these operations have been discontinued.

Activity at the Annex has decreased in recent years and the SJCA facility's current primary mission is to provide a radar testing range and various administrative and warehousing facilities for nearby Norfolk Naval Shipyard and other local Naval activities. SJCA also provides administrative offices, light industrial shops, and storage facilities for tenant Naval commands.

2.2 Blows Creek Watershed Site Descriptions

This section briefly describes activities that occurred at SJCA sites that could potentially contribute to Blows Creek. Figure 1-2 shows the Blows Creek watershed and location of these sites and AOC.

CH2M HILL completed a Final RI/HHRA/ERA in March 2003 that presents results and conclusions for data collected during various phases of the site investigations. The Final RI/HHRA/ERA report identified the potential exposure of receptors to metals and PAHs in surface soil, sediment, and surface water. The following three sites from the RI were identified for evaluation:

- **Site 3 (Waste Disposal Area)**—An approximately 2.1-acre unlined waste-disposal area, formerly referred to as Landfill C, that operated from 1940 to 1970 where wastes such as solvents, acids, bases, oils/oily sludges, and mixed municipal waste were disposed of and/or burned.
- **Site 4 (Landfill D)**—An approximately 10-acre unlined trench-and-fill landfill that reportedly operated from 1970 to 1981 and received drums of unknown wastes and polychlorinated biphenyls (PCBs).

- **Site 5 (Burning Grounds)**—An approximately 3-acre waste ordnance disposal area where materials (e.g., black powder, trinitrotoluene [TNT], fuzes, carbon tetrachloride, trichloroethylene, paint sludge, and pesticides) were reportedly disposed of by open burning between the 1930s and 1970s.

Ecological risk screening (ERS) was conducted for Site 19 and EPIC AOC 1 in the Site Screening Assessment (SSA) Report (CH2M HILL 2002). The study evaluated the site and AOC to determine the course of further action (i.e., additional investigation, removal action, or no further action). The additional site and AOC, located in the Blows Creek watershed, were identified as potential contributors to Blows Creek. The following activities occurred at the site and AOC:

- **Site 19 (Former Building 190)**—Site 19 is located near the former Building 190 to the south of the mouth of Blows Creek. It was reported that various ordnance were loaded and some items may have been dropped in the area. Soil and groundwater sample results along the southern portion of the site indicated impacts from metals and volatile organic compounds (VOCs). Additionally, drainage pipes have been identified from the site leading to Blows Creek. Further evaluation of Site 19 is scheduled for August 2003.
- **EPIC AOC 1 (E Street and Marsh Road Ground Scarring)**—Ground scarring was identified in the EPIC study in the 1937 photograph. By 1949, the area had been developed. Soil sample results of soils indicated that the AOC has been impacted by PAHs. Further evaluation of AOC 1 is scheduled for August 2003.

2.3 Summary of Ecological Risks for Sites 3, 4, and 5 and Conceptual Model for Blows Creek

A screening ecological risk assessment (SERA) and Step 3 of the BERA for Sites 3, 4, and 5 were conducted as part of the Final RI/HHRA/ERA in March 2003. The SERA evaluated the potential for adverse effects to ecological receptors in terrestrial and aquatic habitats at each of the four sites as summarized below. A preliminary conceptual model was developed for Blows Creek to establish a basis for the sampling activities proposed within this WP.

2.3.1 Summary of Ecological Risks for Sites 3, 4, and 5

2.3.1.1 Terrestrial Receptors

The SERA indicated the potential for adverse effects to:

- Lower-trophic-level receptors (plants and soil invertebrates) from the presence of chemicals (primarily inorganic chemicals and PAHs) in soils at Sites 3, 4, and 5; and
- Avian and mammalian vermivores from lead and zinc in Site 3 and Site 5 soils.

The SERA did not recommend further investigation of potential risks to terrestrial receptors from the presence of Contaminants of Potential Concern (COPCs) in soil because remediation/presumptive remedies are currently planned for these sites. The removal action for Site 3 is scheduled for completion by fall 2003.

2.3.1.2 Aquatic Receptors

The SERA indicated the potential for adverse effects to:

- Aquatic life primarily from inorganics, pesticides, and PAHs in upland drainage sediment.
- Aquatic life primarily from inorganic chemicals in upland drainage surface water.
- Avian piscivores from the presence of mercury in upland drainage sediments at Sites 4 and 5.

The SERA also evaluated potential risks associated with a series of upland drainages located adjacent to the sites. Surficial runoff from all sites has the potential to enter these drainages, where it could be transported to Blows Creek and ultimately to the Southern Branch of the Elizabeth River. Figure 2-1 shows this drainage system.

The SERA concluded that, based on the limited presence of surface water, the upland drainages (above the area of tidal influence) provide very little (Sites 4 and 5) or no (Site 3) viable habitat for aquatic species. The aquatic habitats present in the tidally influenced portion of Blows Creek (consisting of the main body of Blows Creek and the lower reaches of the site-related drainages) are meanwhile expected to support a variety of both resident and migratory aquatic species similar to those in the Southern Branch of the Elizabeth River. It was further concluded that a broader range of aquatic species and aquatic-based wildlife could be exposed to chemicals if transported via the site-related drainages to the tidally influenced portion of Blows Creek.

Based on these conclusions, the SERA recommended the following investigations to evaluate the potential for adverse effects to aquatic habitats:

- Further evaluation of the potential for adverse effects to aquatic life in Blows Creek based on the potential chemical transport from Sites 3, 4, and 5 (via the site-related drainages) to Blows Creek sediment and surface water.
- Further characterization of the elevated mercury concentrations (greater than 6 mg/kg) detected in the Site 4 drainage sediments adjacent to Blows Creek. With the exception of mercury in the Site 4 drainage, further evaluation of the potential for adverse effects to aquatic life in the site-related drainage ditches is not recommended, given the minimal habitat these drainages provide.
- Further evaluation of the potential adverse effects to avian piscivores foraging in Blows Creek, based on the potential for mercury in drainage sediments at Sites 4 and 5 to have been transported to Blows Creek.

2.3.2 Preliminary Conceptual Model for Blows Creek

The upland drainages associated with Sites 3, 4, and 5 and EPIC-AOC 1 represent a pathway by which site-related chemicals could be transported to Blows Creek. These upland drainages contain water only during storm events and have the potential to transport chemicals while they are flowing. Between storm events, the drainages typically do not represent viable transport pathways. Site 19 is located immediately adjacent to Blows Creek. Although there are no defined drainage channels associated with this site, surficial runoff from Site 19 (as sheet flow) could also enter Blows Creek during storm events. Once

entering the Blows Creek drainage, a variety of aquatic and terrestrial wildlife could be exposed to chemicals in surface water and sediment.

Surface water entering Blows Creek (through either the upland drainages or via surficial runoff) will pass through a fringing marsh before it enters the open water areas of Blows Creek. Water entering from the larger upland drainage channels typically passes through small open drainage channels that cross the fringing marsh. The marsh downgradient of the smaller drainages and the areas that would receive sheet flow typically does not have defined channels passing through it. Runoff discharging to these areas filters directly through the emergent vegetation before entering the main body of Blows Creek. The presence of the emergent vegetation and the low vertical gradient associated with Blows Creek are expected to rapidly decrease the surface water flow rate as it enters the marsh. The decreasing rate of surface water flow coupled with the high organic carbon content of the marsh sediments is expected to create a depositional sink where chemicals would adsorb and precipitate to sediments. Inorganic chemicals, pesticides, and/or PAHs were identified as the primary chemicals that are likely to be associated with Sites 3, 4, 5, and 19 and EPIC AOC-1. Sediments are generally expected to represent an important depositional sink for these chemicals.

Some chemicals may not be captured by the marsh, but may instead be transported to the main body of Blows Creek. Upon entering the open water area of Blows Creek, chemicals could readily disperse either upstream or downstream from the point of initial discharge. The direction of movement would depend on the direction of tidal flux at the time of release. Sediments throughout most of the main body of Blows Creek have a composition similar to that present in the fringing marsh. This is characteristic of the low energy, depositional environments that typically occur in tidally-influenced marshes such as Blows Creek. Chemicals entering the main body of Blows Creek would also be expected to adsorb and precipitate to sediment. It should be noted, however, that the entire length of Blows Creek is not depositional. Some localized areas do not contain high organic carbon content sediments. These areas appear to be regularly scoured by tidal movements. Most notably, sediments immediately downstream of the railroad bridge and adjacent to the mouth of Blows Creek (where it passes under a small bridge before entering the Southern Branch of the Elizabeth River) contain a much higher sand content than sediments in the remaining portions of Blows Creek. This localized condition results from the higher surface water flow rate and scouring that occurs as a result of the constricted flow at these locations. The coarser sandy sediments in these areas would be expected to retain few chemicals.

Ongoing deposition in the Blows Creek drainage is expected to result in the burial of sediments and their associated chemicals. Burial decreases the exposure potential for most ecological receptors. Storm events and tidal fluxes will resuspend some of the accumulated sediments. Some of the resuspended sediments may be redistributed and resettle in the Blows Creek sediment, while some will be transported out the mouth of Blows Creek and into the Southern Branch of the Elizabeth River.

There is also the potential for chemicals present in groundwater originating from Sites 3, 4, 5, and 19 and EPIC AOC-1 to discharge to Blows Creek. Available information indicates that inorganic chemicals are the primary chemicals that could be discharging to surface water from Sites 3, 4, 5, and 19. Groundwater data are not available for EPIC AOC-1 and Site 19. However, PAHs represent the primary class of compounds present in site soils at

EPIC AOC-1. Following discharge into the creek, chemicals would be distributed by the same forces described for chemicals entering Blows Creek via surficial runoff. Sediment is once again expected to represent a sink for many of the chemicals present in groundwater.

Although Sites 3, 4, 5, and 19 and EPIC AOC-1 are expected to be the dominant sources of chemicals to Blows Creek, chemicals originating from non-site-related sources also have potential to enter this drainage system. Surficial runoff from the Craddock District of Portsmouth could transport chemicals present in the surface soils and stormwater runoff from this residential area to Blows Creek and the Southern Branch of the Elizabeth River. Typical urban residential chemicals that might be found on the surface (and thus occur in Blows Creek) include pesticides, herbicides, and petroleum compounds. Based on the size of the channel (approximately 10 feet wide and 3 to 4 feet deep) entering the SJCA, the contribution from the Craddock District is expected to be small. Non-site-related chemicals originating in the sediments and surface water of the Southern Branch of the Elizabeth River could also be transported up into Blows Creek, primarily as a result of tidal flux. Finally, Annex-related activities unrelated to Sites 3, 4, 5, and 19 and AOC-1 may also be a source of chemicals to Blows Creek and the Southern Branch of the Elizabeth River. Following transport, aquatic life present in these water bodies could be exposed to these non-site-related chemicals. These other potential risks must also be considered when evaluating the relative importance of site-related ecological risk (if any) in the Blows Creek drainage. Ecological receptors and exposure routes identified for evaluation in Section 2.3.1 will be the focus of the further evaluation for Blows Creek.

2.4 Objectives of Blows Creek Investigation

This investigation's objective is to evaluate the potential adverse effects (risk) to aquatic life resulting from chemicals in the sediments of Blows Creek. The investigation will collect surface sediment from Blows Creek for chemical/physical analysis. Tissue samples also will be collected for mercury residue analysis to further evaluate the potential risks from this chemical to higher trophic-level species. Emphasis will be placed on collecting samples from areas potentially impacted by RI Sites 3, 4, and 5, to characterize chemical inputs from these potential source areas, but will also include samples that will characterize potential impacts from Site 19 and EPIC AOC 1.

Surface water was identified as a potential exposure pathway via which aquatic life could be exposed to chemicals. However, sampling activities will focus on sediment based on the transient nature of surface water and the propensity for sediments to act as a repository for many chemicals. Consistent with communications between the U.S. Navy and the USEPA, groundwater will also be used to screen the potential for chemical transport to surface water.

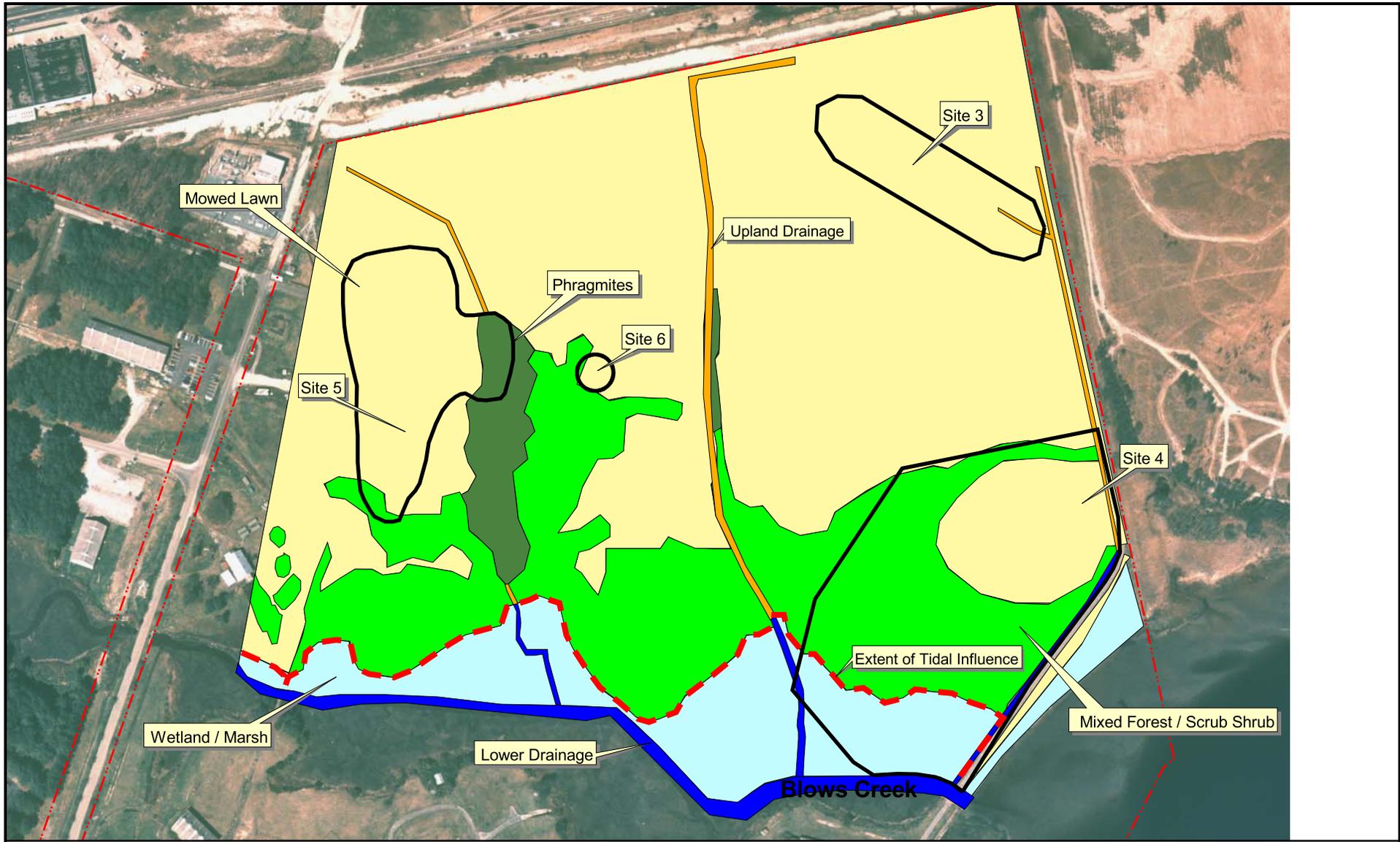
Data collected during this investigation will be used to evaluate the potential for adverse effects to Blows Creek and to complete Steps 7 and 8 of the ERA process for Sites 3, 4, and 5. The following specific objectives were identified for the Blows Creek investigation:

- Characterize chemical concentrations and the potential for adverse ecological effect in the lower (tidally influenced) reaches of the Site 3, 4, and 5 drainage swales, as they pass through the wetlands bordering Blows Creek. The area identified for investigation is

downgradient of the area sampled during the RI and is a potential depositional area, where chemicals originating from Sites 3, 4, and 5 could accumulate.

- Characterize chemical concentrations and the potential for adverse ecological effect throughout the main body of Blows Creek. Emphasis will be placed on collecting samples from areas that could be impacted by adjacent sites or AOCs and on depositional areas, where chemicals have the greatest potential to accumulate in sediment.
- Further characterize the area of elevated mercury concentration (> 6 mg/kg) detected in the drainage swale adjacent to Site 4 during the RI (CH2M HILL 2003). Mercury was identified at this location during the SERA and Step 3 ERA as a potential risk to both benthic invertebrates and avian piscivores.
- Characterize chemical concentrations in Blows Creek originating from naturally occurring concentrations (inorganic chemicals) and non-site-related sources.

The U.S. Navy and the USEPA are currently discussing the collection of subsurface sediment samples. These samples would be collected during a second phase of the site investigation.



LEGEND

- | | |
|----------------------------|--|
| Activity Boundary | Mowed Lawn |
| IR Sites | Phragmites |
| Mixed Forest / Scrub Shrub | Tidal Wetland / Marsh (Predominantly Smooth Cordgrass, Black Rush, and Phragmites) |
| Lower Drainage | Extent of Daily Tidal Influence Adjacent to Sites 3, 4, 5, & 6 |
| Upland Drainage | |



0 200 400 Feet

Figure 8-2
 Sites 3,4, and 5/6 Habitat Composition
 St. Juliens Creek Annex
 Chesapeake, Virginia

SECTION 3

Approach to Blows Creek Investigation

A phased investigation will be conducted to collect the additional data needed to characterize potential ecological risks in Blows Creek. Two main phases of investigation are planned. The Phase I investigation primarily involves the collection of surface sediment samples (0 to 6 inches). These sediment samples will be collected from locations throughout Blows Creek for chemical, physical, and bioassay analyses. Data from these samples will be used to further evaluate the potential for adverse effects to benthic organisms and determine if chemicals are present in Blows Creek sediments at concentrations that could represent a potential bioaccumulative risk to predatory wildlife. The Phase I sample locations are shown in Figure 3-1.

The Phase II investigation will consist of collecting tissue residue samples from Blows Creek. This second phase will be conducted to evaluate the potential for bioaccumulative chemicals to adversely affect higher trophic-level species and will be developed based on the results of the Phase I sediment chemical analysis.

The following sections provide an overview of the Phase I and II investigations. Section 4 details each procedure to be used in both phases of the investigation.

3.1 Phase I

A total of 38 surface sediment samples (0 to 6 inches; 32 potentially site-impacted samples and 6 upgradient reference samples) will be collected from locations throughout Blows Creek to further characterize potential risks to benthic organisms. The sediment data will also be used as part of the Phase II investigation to determine if there is potential risk to wildlife from accumulation in the aquatic food web. Sediment samples were located in different segments/regions of Blows Creek based on the following specific objectives:

- Sediment samples will be collected from small potentially site-impacted drainages as they pass through the emergent marshes that border Blows Creek. The drainages identified for sampling potentially receive chemical inputs from upland site-related drainages (sampled during the Sites 3 through 6 RI) and/or via surficial runoff. These small drainages comprise viable aquatic habitat for a variety of (mostly) invertebrate species and are likely to represent “traps” for sediment particles entering the creek from upland sources.
- Sediment samples will be collected from potentially site-impacted locations throughout the main body of Blows Creek. The main body of Blows Creek is expected to support a broader diversity of aquatic species than the fringing marsh habitats and is likely to receive and integrate chemicals from multiple potential sources. Samples will be collected from areas that are most likely to be impacted by site-related sources.
- Sediment samples will be collected from upgradient locations in Blows Creek (upgradient of AOC 1) and at the mouth of Blows Creek (adjacent to the Southern Branch of the Elizabeth River) to evaluate the influence of non-site-related sources on

chemical concentrations (and potential risks) in Blows Creek. Sediments upgradient of AOC 1 may be impacted by chemical inputs from the Craddock District, while sediments at the mouth of Blows Creek are likely to be impacted both by site-related chemical sources and by the Southern Branch of the Elizabeth River.

Figure 3-1 shows the location of all 38 surface sediment samples identified for collection from Blows Creek. Table 3-1 provides a detailed description and rationale for the collection of each sample.

At the time the sediment samples are collected, adequate material will be obtained from each location to conduct both chemical/physical and bioassay analyses. However, the sediment samples will be analyzed using a phased approach. All sediment collected from a sample location first will be placed into a sample container and homogenized. A split of this homogenized sample will be sent to the analytical laboratory for rapid turnaround chemical analysis. The remaining portion of the homogenized sample will be maintained in cold storage for subsequent bioassay analyses.

Sediment samples from all locations will undergo chemical and physical analysis. The objective of these analyses is to determine if site-related chemicals are present in sediment at concentrations that could present a potential risk to ecological receptors. All sediment samples will be analyzed for Target Compound List (TCL), TCL semivolatile organic compounds (SVOCs), TCL pesticides, PCBs, Target Analyte List (TAL) metals, and explosives. Selected sediment samples collected downgradient of Sites 3, 4, and 5 and one upgradient sample also will be analyzed for dioxins/furans. Acid-volatile sulfide/simultaneously extractable metals (AVS/SEM), grain size (sieve), and total organic carbon (TOC) will be analyzed on all sediment samples. Conductivity, pH, salinity, dissolved oxygen (DO), temperature, and turbidity will be measured in overlying water and general conditions (e.g., tidal height/stage and the presence/absence of organisms) will be recorded at the time of sampling.

The unvalidated sediment chemical analytical data will be compared to literature-based sediment screening values immediately following receipt from the laboratory. The literature-based sediment screening values used for the Sites 3, 4, 5 and 6 Steps 1 through 3 ERA (CH2M HILL 2003) will be used to screen these data. The objective of this screening is not to identify all sediment sample locations having exceedences, but instead to select a subset of samples for bioassay testing that maximizes the information provided by the bioassays. The following general guidelines will be used (following screening) to select up to 20 potentially site-impacted sediment samples for bioassay analysis:

- Only sediment samples containing COPCs identified from the screening ERA as being site-related and present at detected concentrations that represent a potential risk (hazard quotient > 1) to benthic organisms (USEPA Region III sediment flora/fauna) will be selected for bioassay testing. Selection of samples for bioassay testing will focus on samples with chemicals detected at concentrations exceeding screening values, and not on nondetected chemicals or chemicals without screening values.

TABLE 3-1
Sediment Sample Location Objectives
Blows Creek BERA Work Plan

Location Number	Analysis ¹	Sampling Location Description	Sample Location	Rationale
1	2	Main channel; adjacent to farm fields and parking lot in Craddock District to characterize chemicals potentially originating from offsite	Main channel	Characterize non site-related chemical concentrations originating from Craddock District; multiple samples collected to develop robust database that supports statistical analysis
2	2	Main channel; located where stream enters SJCA near gravel patrol road; mid-channel, to characterize chemicals potentially originating from offsite	Main channel	
3	2	Main channel; immediately south of base boundary to characterize chemicals potentially originating from offsite; main channel	Main channel	
4	2, 3	Main channel; 150' south of base boundary to characterize chemicals potentially originating from offsite; main channel	Main channel	
5	2	Main channel; on north side of road near to AOC 1; to characterize chemicals potentially originating from offsite	Main channel	<p>Determine if surficial runoff of chemicals from the adjacent AOC 1 represents a potential risk to ecological receptors in the main body of Blows Creek</p> <p>Determine if surficial runoff of chemicals from the adjacent AOC 1 represents a potential risk to ecological receptors in the main body of Blows Creek</p> <p>Determine the downgradient extent of any risks to ecological receptors in the main body of Blows Creek as a result of chemicals originating from AOC 1</p> <p>Determine if tributary represents pathway by which chemicals from AOC 1 enter Blows Creek system and if chemicals in the tributary represent a potential risk to ecological receptors; multiple samples taken in tributary to characterize possible concentration gradient</p> <p>Determine the downgradient extent of any risks to ecological receptors in the main body of Blows Creek as a result of chemicals originating from AOC 1</p> <p>Determine if surficial runoff of chemicals from the adjacent Site 5 represents a potential risk to ecological receptors in the main body of Blows Creek</p> <p>Determine if drainage represents pathway by which chemicals from Site 5 enter Blows Creek system and if chemicals in the drainage represents a potential risk to ecological receptors</p>
6	2	Main channel; on north side of road near to AOC 1; to characterize chemicals potentially originating from offsite	Main channel	
7	2	Main channel; on south side of road adjacent to AOC 1; main channel	Main channel	
8	2	Main channel; adjacent to AOC 1; main channel	Main channel	
9	2	Main channel; downstream of AOC 1	Main channel	
10	2	Tributary; to the east of AOC 1	Tributary	
11	2	Tributary; to the east of AOC 1	Tributary	
12	2	Main channel; downstream of AOC 1	Main channel	
13	2	Main channel; depositional area on south side of channel with potentially increasing Site 5 influence	Main channel	
14	2, 3	Drainage; south of Site 5, approximately 10' from main channel of Blows Creek	Drainage	

TABLE 3-1
Sediment Sample Location Objectives
Blows Creek BERA Work Plan

Location Number	Analysis ¹	Sampling Location Description	Sample Location	Rationale
15	2	Main channel; south of Site 5	Main channel	Determine if surficial runoff and discharge (via the site-related drainage) of chemicals from the adjacent Site 5 represents a potential risk to ecological receptors in the main body of Blows Creek
16	2, 3	Drainage; south of Site 5, approximately 12' from main channel of Blows Creek	Drainage	Determine if drainage represents pathway by which chemicals from Site 5 enter Blows Creek system and if chemicals within this drainage represent a potential risk to ecological receptors
17	2	Drainage; south of Site 5, approximately 10' from main channel of Blows Creek	Drainage	Determine if drainage represents pathway by which chemicals from Site 5 enter Blows Creek system and if chemicals in the drainage represent a potential risk to ecological receptors
18	2, 3	Tributary; south of Site 5; at a point where drainage enters the wetland	Tributary	Determine if tributary represents pathway by which chemicals from Site 5 enter Blows Creek system and if chemicals in the tributary represent a potential risk to ecological receptors; multiple samples taken in tributary to characterize possible concentration gradient
19	2	Tributary; south of drainage for Site 5	Tributary	
20	2, 3	Tributary; south of drainage for Site 5; approximately 15 feet from main channel of Blows Creek	Tributary	
21	2	Main channel; downstream of Site 5	Main channel	Determine if surficial runoff and discharge (via the site-related drainages/tributary) of chemicals from Site 5 represents a potential risk to ecological receptors in the main body of Blows Creek
22	2	Main channel; potentially increasing Site 3 influence	Main channel	Determine the downgradient extent of any risks to ecological receptors in the main body of Blows Creek as a result of chemicals originating from Site 5
23	2	Main channel; potentially Site 3 and/or Site 19 influence	Main channel	Determine the downgradient extent of any risks to ecological receptors in the main body of Blows Creek as a result of chemicals originating from Site 5 and risks resulting from surficial runoff and discharge (via the site-related drainage) of chemicals from Site 19
24	2	Tributary; adjacent to Site 19 and west of Site 19	Tributary	Determine if tributary represents pathway by which chemicals from Site 19 enters Blows Creek system and if chemicals in the tributary represent a potential risk to ecological receptors
25	2	Main channel; adjacent to Sites 3, 4, and 19	Main channel	Determine if surficial runoff and discharge (via the site-related drainages/tributary) of chemicals from Sites 3, 4, and/or 19 represent a potential risk to ecological receptors in the main body of Blows Creek
26	2	Southern shoreline of main channel; at discharge of terracotta pipe at Site 19	Southern shoreline of main channel	Determine if terracotta pipe from Site 19 represents a source of chemicals to Blows Creek
27	2	Southern shoreline of main channel; at concrete vault adjacent to Site 19	Southern shoreline of main channel	Determine if concrete vault adjacent to Site 19 represents a source of chemicals to Blows Creek

TABLE 3-1
Sediment Sample Location Objectives
Blows Creek BERA Work Plan

Location Number	Analysis ¹	Sampling Location Description	Sample Location	Rationale
28	2	Drainage; south of drainage for Sites 3 & 4; approximately 15 feet from main channel of Blows Creek	Drainage	Determine if drainage represents pathway by which chemicals from Sites 3 and/or 4 enter Blows Creek system and if chemicals in the branched drainage system represent a potential risk to ecological receptors; multiple samples taken to characterize possible concentration gradients in this branched drainage system
29	2	Drainage; south of drainage for Sites 3 & 4	Drainage	
30	2,3	Drainage; south of drainage for Sites 3 & 4; at a point where drainage enters the wetland	Drainage	
31	2	Drainage; south of drainage for Sites 3 & 4	Drainage	
32	2,3	Drainage; south of drainage for Sites 3 & 4; approximately 15 feet from main channel of Blows Creek	Drainage	
33	2	Adjacent to Sites 3, 4, and 19	Main channel	Determine if surficial runoff and discharge (via the site-related drainages/tributary) of chemicals from Sites 3, 4, and/or 19 represent a potential risk to ecological receptors in the main body of Blows Creek
34	2	Drainage; north side of Blows Creek adjacent to Site 4; area of disposed timbers	Drainage	Determine if drainage represents pathway by which chemicals from Site 4 enter Blows Creek system and if chemicals in the drainage represent a potential risk to ecological receptors
35	2	Southern shoreline of main channel; adjacent to Site 19	Southern shoreline of main channel	Determine if surficial runoff of chemicals from the adjacent Site 5 represents a potential risk to ecological receptors in the main body of Blows Creek
36	2	Drainage; south of drainage for Site 4; approximately 5 feet from main channel of Blows Creek; area of disposed timbers	Drainage	Determine if tributary represents pathway by which chemicals from Site 4 enter Blows Creek system and if chemicals in the tributary (most notably mercury) represent a potential risk to ecological receptors; multiple samples taken in tributary to characterize possible concentration gradient
37	2	Drainage; eastern side of Site 4	Drainage	
38	2	Drainage; eastern side of Site 4	Drainage	

Analysis:

¹Surface sediment samples for bioassay analysis were not indicated in this table because the selection of these samples will depend on a preliminary screening of the Phase I sediment chemical analytical data.

²TCL SVOCs, TCL Pest/PCBs, TAL metals, AVS/SEM, explosives, TOC, and grain size.

³Dioxins/furans

- Samples encompassing a range in the concentration of the chemical/chemical groups identified for testing will be selected in an attempt to establish a concentration/causality gradient.
- Emphasis will be placed on selecting samples that test the toxicity of key chemicals within a range of physical conditions appropriate for the test organisms (grain size, salinity, etc.) present in impacted areas of Blows Creek sediment.

Chemical analytical data from both reference and site-impacted samples will be considered along with physical data collected during the site investigation (e.g., AVS/SEM) to identify samples for bioassay analysis. It is anticipated that a conference call will occur between representatives of the USEPA Biological Technical Assistance Group (BTAG), the U.S. Navy, and CH2M HILL to communicate and discuss the samples identified for bioassay testing.

Sediment bioassays will be initiated on the split of the previously-collected sediment samples immediately following sample selection. Sediments collected from up to five of the upgradient samples also will be selected for testing to determine the potential toxicity of chemicals from non-site-related sources and to differentiate between impacts resulting from site- and non-site-related sources. The physical conditions at these sample locations (e.g., salinity, sediment grain size, and composition) will be surveyed prior to initial sample collection and the chemical composition will be reviewed prior to initiating the sediment bioassay to ensure that samples from the reference locations have a physical composition that is similar to that present in the lower reaches of Blows Creek and represent non-site-impacted sample locations. If the selected sample locations do not meet these objectives, then alternate reference sample locations will be selected.

Bioassays were selected for analysis because they directly measure the toxicity of sediments, accounting for both the bioavailability of chemicals in sediment and the toxicity of complex chemical mixtures. If the bioassays indicate sediment toxicity, the data will be further evaluated to determine if a chemical concentration gradient can be established to identify chemicals/concentrations in Blows Creek sediment that are toxic to benthic organisms.

A 28-day sediment bioassay with the amphipod *Leptocheirus plumulosus* (*L. plumulosus*) will be used for the sediment toxicity tests. *L. plumulosus* was selected for the bioassays because it is tolerant of the salinity range expected in Blows Creek, is indigenous to the Chesapeake Bay, and has direct exposure to chemicals in sediment via burrowing. Furthermore, studies have shown that *L. plumulosus* abundance in Chesapeake Bay sediments can be negatively correlated with sediment contamination (Holland et al. 1988, McGee and Fisher 1999; as cited in USEPA 2001), suggesting its appropriateness for use as a bioindicator.

The sediment bioassay results will be used in conjunction with the chemical and physical sediment data to characterize the potential for adverse effects to benthic organisms in Blows Creek sediment.

3.2 Phase II

3.2.1 Tissue Residue

Tissue residue analysis will be conducted on organisms collected from Blows Creek during the Phase II investigation. This evaluation will be conducted to further evaluate the potential for mercury, which could be originating from Sites 4 and 5, to adversely affect avian piscivores and other higher trophic-level species. The specific organisms to be collected for tissue residue analysis will be selected based on the following general guidelines:

- Organisms will be selected to be representative of those indicating potential risk in food web models. If risks are indicated for piscivorous species, for example, emphasis will be placed on collecting fish for tissue residue analysis.
- Only organisms that can be collected in adequate biomass to meet analytical needs will be selected for tissue residue analysis.
- Only resident organisms that are immobile or have limited mobility will be selected for tissue residue analysis to ensure that tissue burdens reflect (to the greatest extent possible) chemicals accumulated from Blows Creek sediments and to minimize the potential for the accumulation of chemicals from offsite areas.

Based on habitat present in Blows Creek, three candidate organism groups were identified for tissue residue analysis: fish comprising *Fundulus* spp., benthic-dwelling polychaete worms, and/or the brackish water clam *Rangia cuneata*. These organism groups represent appropriate candidates for tissue residue analysis because they:

- Are known to commonly occur in the region and in brackish, tidal, creek habitats and are likely to be present in Blows Creek.
- Represent important prey items for avian/mammalian wildlife or aquatic biota and are relevant indicators of accumulation for one or more of the predator/prey pathways modeled for Blows Creek.
- Would contain chemical body burdens (bioaccumulation) representative of Blows Creek sediments since they forage and frequently burrow in sediments.
- Are sessile or have home ranges of a few hundred feet or less, making them ideal for the evaluation of bioaccumulation within a localized area.

According to Lippson and Lippson (1997), mummichog (*Fundulus heteroclitus*) and banded killifish (*Fundulus diaphanus*) are the most relevant fish species that can be collected for tissue analysis. They are expected to be common resident species in Blows Creek.

Polychaete worm species that are expected to occur in Blows Creek include the common clam worm (*Neanthis succinea*), the red-gilled mud worm (*Marenzelleria viridis*), whip mud worm (*Polydora cornuta*), and various bloodworms (*Glycera* spp.). Finally, the clam species likely to be present in Blows Creek is the brackish water clam (*Rangia cuneata*).

An additional assessment of the viability of collecting these or other organism groups for chemical analysis will be made during the collection of the Phase I sediment samples. Information gained during this sampling effort will be considered along with the food web

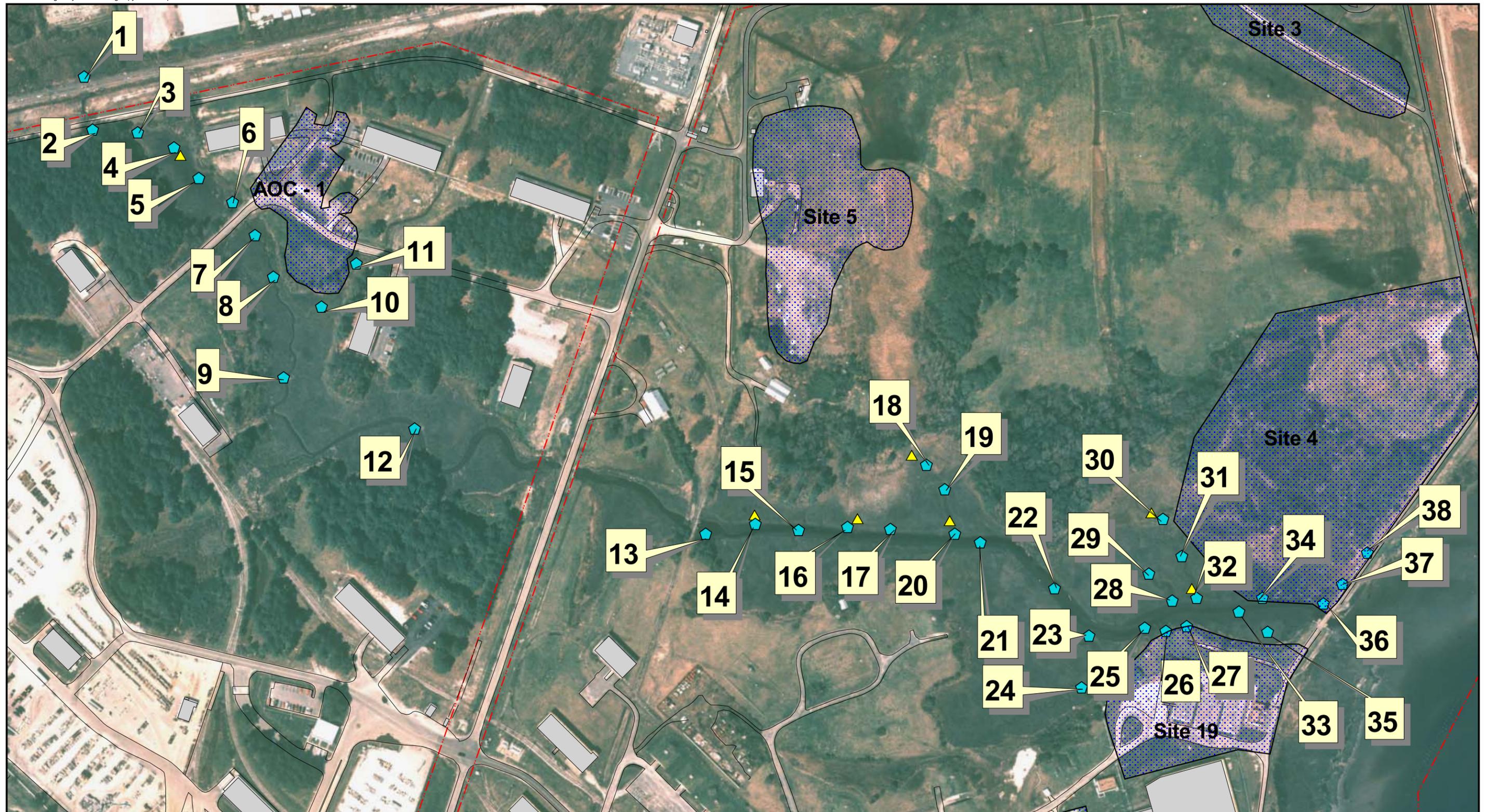
model outcomes to select one or more of these or more organism groups for tissue residue analysis, the number of tissue residue samples to be collected, and the location(s) from which the samples will be taken. It is anticipated that up to three composite samples of each organism group selected for evaluation will be collected from Blows Creek, to encompass the three primary source areas from which chemicals are likely to originate (AOC 1, Site 5, and Sites 3, 4, and 19). Composite samples will be necessary to ensure adequate biomass for chemical residue analysis. Final determination of the species, sample locations, and analytes will be made in consultation with the USEPA BTAG prior to the collection of any tissue residue samples.

It is anticipated that a conference call will occur between representatives of the USEPA BTAG, the U.S. Navy, and CH2M HILL to communicate and discuss the selection of species groups, samples identified for bioassay testing.

Tissue residue samples will be composited by organism type in appropriate lab-supplied containers and sent to the chemical analytical laboratory for whole body tissue residue analysis. Samples will be analyzed for percent lipids, percent moisture, and mercury. The food web calculations will use these data to model potential risks to higher trophic-level species.

3.2.2 Subsurface Sediment

The United States Navy and the USEPA are currently discussing the collection of subsurface sediment (greater than 6 inches) from Blows Creek and no final sampling plan has currently been developed for this media. However, no subsurface sediment samples will be collected until the Phase II investigation.



LEGEND

- ◆ Surface Sediment Samples (Full Suite Minus VOCs)
- ▲ Surface Sediment Samples (Dioxin)
- ▨ Active IR Site Boundaries

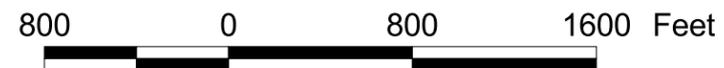


Figure 3-1
Sediment Sample Locations
BERA Work Plan
St. Juliens Creek Annex
Chesapeake, Virginia

SECTION 4

Investigation Tasks and Methodology

This section presents the tasks involved with implementing the field investigation (including sample locations) and sample analysis and data validation.

Potential sediment sample locations were first discussed during a November 2001 meeting with representatives from the USEPA BTAG, the U.S. Navy, and CH2M HILL. Each potential sample location was then surveyed during a site visit conducted in late December 2001 by representatives of the U.S. Navy and CH2M HILL. During the site visit, potential sample locations were visited and preliminary sediment grab samples were taken from each location. This site reconnaissance visit completed Step 5 of the ERA process for Sites 3, 4, and 5. Sample locations were refined/modified based on the site evaluation and following subsequent discussions with the USEPA BTAG to ensure they could be sampled as planned and met the site investigation's objectives. Tissue residue analyses were also added to this phased site investigation to further characterize potential bioaccumulative risks.

A Master Work Plan (MWP), Master Field Sampling Plan (MFSP), Master Quality Assurance Plan (MQAPP), Master Investigation-Derived Waste Management Plan (MIDWMP), and Master Health and Safety Plan (MHASP) have been prepared for SJCA (CDM 2000, updated CH2M HILL 2003). The Master Project Plans (MPP) provide the details for sampling and analysis protocols to be followed and general types of activities to be accomplished for implementing the field activities at SJCA. The preparation of site-specific plans is simplified through reference to the MPP documents. Site-specific plans are presented in the form of checklists that supplement detailed protocols and Standard Operating Procedures (SOPs) presented in the MPP documents. The checklists and SOPs are provided in Appendix A and present information specific to the BERA sampling activities. Specific procedures to be implemented will be addressed in project instructions.

4.1 Field Investigation

This task involves efforts related to fieldwork support—the field investigation, sample designation, surveying, and investigation-derived waste (IDW).

4.1.1 Fieldwork Support

Fieldwork support includes subcontractor procurement, mobilization, and demobilization. As part of the initial field mobilization to SJCA, CH2M HILL will procure an analytical laboratory, bioassay laboratory, and data validation services. The U.S. Navy will approve the subcontracted analytical laboratories. Equipment and supplies will be brought to the site when the CH2M HILL field team mobilizes for field activities. Demobilization activities will consist of general site restoration prior to the return transport of field equipment and crew. A minimal amount of IDW, consisting of decontamination fluids, will be generated.

Before beginning the fieldwork, CH2M HILL will have field meetings to discuss the work items, worker responsibilities, and to familiarize workers with the SJCA MHASP.

CH2M HILL will audit each field activity with the use of appropriate checklists (Appendix A) in addition to conforming to the MHASP.

4.1.2 Field Investigation and Sampling Activities

Field activities will involve collecting 38 sediment samples from the Blows Creek watershed. The sample locations are shown in Figure 3-1. The rationale for each sample is summarized in Table 3-1. Table 4-1 shows the required containers, preservatives, holding times, and analytical methods for samples. It is anticipated that up to three tissue samples of each selected species will be collected from Blows Creek during Phase II of the investigation. However, the number and type of samples will not be defined until the Phase I site investigation has been completed and the Phase I surface sediment data has been screened for potential risks to upper trophic-level species.

Sampling will proceed from downstream to upstream locations to reduce the amount of turbidity and potential cross-contamination of downstream samples. Sampling activities will not occur after periods of heavy rainfall. The following sections present a detailed discussion of sampling protocols and procedures, the selected sample locations and methodology, and analyses to be conducted on each sample.

4.1.2.1 Sediment Sampling

Thirty-eight surface sediment samples will be collected from the Blows Creek watershed. Figure 3-1 shows the location of each sample while Table 3-1 summarizes the rationale for each sample.

Sediment samples will be collected using a stainless-steel device such as a hand auger or Ekman dredge sampler and transferred into a stainless-steel bowl to be transferred into the sample containers. Organic material (i.e., grass roots, etc.) will be removed prior to placement into the sample containers. The remaining sample volume will be homogenized in the stainless-steel bowl using a stainless-steel trowel. One part of the total volume will be used for chemical and physical analyses while the remainder will be used for bioassay toxicity testing.

All sediment samples will be analyzed for TCL SVOCs, TCL pesticides, PCBs, TAL metals, and explosives (Table 3-1). Selected sediment samples collected downgradient of Sites 3, 4, and 5 (Samples 14, 16, 18, 20, 30, and 32) will be analyzed for dioxins/furans based on these sites' potential to be source areas for these compounds. Dioxins/furans will also be analyzed in one of the upgradient samples (Sample 4), to determine if there are non-site-related sources of these compounds. In addition to chemical analyses, all sediment samples will be analyzed for AVS/SEM, grain size (sieve), and TOC. Conductivity, pH, salinity, DO, temperature, and turbidity will be measured in overlying water and general conditions will be recorded including tidal height/stage and the presence/absence of organisms. The bioassay laboratory will monitor confounding factors such as elevated ammonia levels and sulfides in test containers (Appendix B).

TABLE 4-1

Analytical Methods, Required Containers, Preservatives, and Holding Times for Samples
Blows Creek BERA Work Plan

Sediment Samples					
Analysis	Method	Sample Container	Preservative	Holding Time	Volume of Sample
TCL SVOCs	CLP OLM03	One 8-oz glass bottle with Teflon-lined cap	Cool to 4C	14 days	Fill completely
TCL Pesticides/PCBs	CLP OLM03	Two 4-oz glass bottles with Teflon-lined cap	Cool to 4C	14 days	Fill completely
Explosives	SW 846 8330	One 4-oz glass bottle with Teflon-lined cap	Cool to 4C	14 days	Fill completely
Dioxin/Furans	SW 846 8290	One 4-oz glass bottle with Teflon-lined cap	Cool to 4C	14 days	Fill completely
TAL Metals/Cyanide	CLP ILM04	One 4-oz glass bottle with Teflon-lined cap	Cool to 4C	6 months; 28 days for mercury; 14 days for cyanide	Fill to shoulder
AVS	USEPA 1991	One 8-oz glass bottle with Teflon-lined cap	Cool to 4C	14 days	Fill completely (no air space)
SEM	SW 846 6010B and 7471A				
TOC and pH	415.1 and 9045	One 8-oz glass bottle with Teflon-lined cap	Cool to 4C	28 days	Fill completely
Grain size	ASTM D422	One 8-oz glass bottle with Teflon-lined cap	None	Not applicable	Fill completely
Toxicity Bioassay	28-d, Chronic <i>L. plumulosus</i> (USEPA, 2001)	Two 1-gal polyethylene bottles	None	8 weeks	Fill completely
Aqueous (QC Samples)					
TCL SVOCs	CLP OLM03	Two 1-liter amber bottles with Teflon-lined cap	Cool to 4C	7 days to extraction, 40 days to analysis	Fill to shoulder
TCL Pesticides/PCBs	CLP OLM03	Two 1-liter amber bottles with Teflon-lined cap	Cool to 4C	7 days to extraction, 40 days to analysis	Fill to shoulder
Explosives	SW 846 8330	Two 1-liter amber bottles with Teflon-lined cap	Cool to 4C	14 days to extraction, 40 days to analysis	Fill to shoulder
Dioxin/Furans	SW 846 8290	Two 1-liter amber bottles with Teflon-lined cap	Cool to 4C	7 days to extraction, 40 days to analysis	Fill to shoulder
TAL Metals	CLP ILM04	1-liter polyethylene bottle	HNO ₃ to pH <2; Cool to 4C	6 months; 28 days for mercury	Fill to shoulder
Cyanide	CLP ILM04	1-liter polyethylene bottle	NaOH to pH <2; Cool to 4C	14 days	Fill to shoulder
Hardness	130.2	1-liter polyethylene bottle	HNO ₃ to pH <2; Cool to 4C	6 months	Fill to shoulder

A 28-day sediment bioassay with the amphipod *Leptocheirus plumulosus* (*L. plumulosus*) will be used to test a subset of the total samples. The decision process for selecting these samples is presented in Section 3. The sediment bioassay will be conducted in a manner consistent with protocols presented in USEPA 2001 and U.S. Army Corps of Engineers 1996.

Appendix B provides the toxicity-testing protocol and quality control (QC) procedure of the laboratory most likely to perform the sediment bioassay. Sediment samples from all locations that are regularly covered by water will be tested with *L. plumulosus*. The endpoints of the 28-day bioassay are survival, growth, and reproduction. These endpoints are ecologically relevant and support the assessment endpoint of benthic community viability identified in the Site 3, 4, and 5 Step 1 through 3 ERA.

4.1.2.2 Tissue Sampling

As previously discussed, the number, type, and area of each tissue residue sample collection will be based on results of the Phase I sediment data for mercury and on observations about the feasibility of organism collection. It is anticipated that up to three samples of each selected organism type will be collected. Individual organisms (fish, worms, or clams) are expected to be smaller than the minimum required biomass for chemical analysis and it will be necessary to composite multiple individuals into each sample to provide sufficient tissue biomass for analysis. The area of collection of each sample may vary depending on the number and/or size of organisms found and it is possible that sufficient biomass of these organisms will not be obtainable at all sample locations. Best professional judgement by the sampling team will be considered to determine when a reasonable effort has been expended and the sampling at a location is concluded. One field duplicate sample will be collected during sampling, providing sufficient biomass is available. A duplicate sample is defined as an additional sample of tissue collected from the same sample location.

Collection Techniques. The collection techniques used will depend on the specific organisms to be collected. As discussed in the preceding sections, mummichog (*Fundulus* spp.) and benthic invertebrates (polychaete worms or clams) are considered the most likely candidate species for tissue residue analysis. The following section presents techniques that will be used for the collection of these species. These collection techniques may be modified as necessary based on the final species identified for tissue residue analysis.

Fish. The primary methods for collecting *Fundulus* spp. will be minnow traps. Other collection techniques may be used if minnow traps do not yield sufficient biomass for tissue residue analysis. These techniques are briefly described below.

- **Minnow Traps**—Wire-framed minnow traps with double conical entrance openings of 1 inch will be positioned in shallow water locations of each sampling station. Several feet of twine and a numbered float will be attached to each for recovery and tracking purposes. Each trap will be baited with crab meat, fish, or cat food. Traps will remain deployed for up to 3 days at each sampling location and will be checked once daily, as practical, for captured fish. Should the traps yield few to no samples, they will be relocated to a more viable collection location. Minnow traps will be recovered earlier if a sufficient number of samples is collected prior to 3 days.
- **Cast Nets**—Cast nets are designed to be hand thrown over schools of fish, which are trapped as the netting falls rapidly through the water column and closes as it is retrieved

with a hand line. Typical construction includes a monofilament netting with an approximately 5-foot radius and lead weighted perimeter. Mesh sizes that may be used during this sampling event include, but are not limited to, 0.5-inch, 0.75-inch, and 1-inch stretched mesh. The cast net will be used in shallow and deeper water areas where there are few obstructions to tangle the net. Cast nets will not be used if minnow traps are effective.

- **Seines**—Seines are useful for capturing shoreline fishes, and will be used in areas where shoreline and substrate conditions (e.g., depth) are safe for field personnel. A 50-foot seine will be used; however, actual extended length may vary based on field conditions. Field personnel will haul in each end of the net, taking care to avoid fish escape by pulling the weighted bottom end forward until it is on the beach. This method will not be used if minnow traps are effective.

Benthic Invertebrates (Polychaetes and Clams). The primary method for collecting polychaete worms or clams will involve sieving sediment grab samples. Surficial sediments (the approximate top 6 inches) will be collected from the selected locations using an Ekman dredge or Ponar sampler. These sediments will be placed into a sieving device with a 0.5-mm mesh size opening (e.g., a mesh bag or a bucket with a mesh-lined bottom). The samples in the sieving device will be rinsed of the majority of particulate and debris by dunking or rinsing with site water. The entirety of these sieved samples (organisms with remaining debris and/or large sediment particles) will be transferred into appropriately sized sample containers for preparation prior to shipment (Section 2.3.2).

Sample Preparation and Analyses. Once the organisms are collected, they will be prepared for shipment to the subcontracted laboratory for chemical analysis. The analytical laboratory will process all tissue samples prior to analysis. Fish and polychaete worms samples will be processed as whole body samples, while the clams will require separation of the tissue from the shell prior to analysis. These preparation and analysis steps are described below.

Fundulus species collected from the minnow traps will first be separated from any other fish species collected by the trap. Non-*Fundulus* species will be released following identification. Each individual *Fundulus* collected in a sample will be sexed, measured, and weighted and the general condition of each fish will be noted. Each fish will then be transferred into a decontaminated container to comprise the composite sample. The container for each composite sample will then be labeled and placed on wet ice until it can be transferred to a freezer for holding. Once all samples are collected, they will be shipped as specified by the laboratory and analytical method (e.g., in coolers of wet ice, or containers with dry ice).

After sieving, the polychaete worm or clam samples will be transferred to a pan or dish, where they will be isolated from unwanted debris and other aquatic organisms. Each individual of the desired species collected within a sample will be measured and weighed and transferred into a decontaminated container to comprise the composite sample. The container for each composite sample will then be labeled and placed on wet ice until it can be transferred to a freezer for holding. Once all samples are collected, they will be shipped according to the specification of the laboratory and analytical method (e.g., in coolers of wet ice, or containers with dry ice).

4.1.3 Sample Designation

Sampling locations and sampled media collected during the investigation will be assigned unique designations to allow the sampling information and analytical data to be entered into the Geographic Information System (GIS) and Data Management system for SJCA.

Each sample will be designated by an alphanumeric code that identifies the site and matrix sampled and contains a sequential sample number. Quality assurance/quality control (QA/QC) samples will have a unique sample designation. The first two letters indicate the Installation (St. Juliens Creek Annex) and the next two indicate the unique site (Blows Creek watershed). The two letters following the dash indicate the type of sample taken and the last two digits indicate the sample number. Surface sediment samples will be designated in a similar manner beginning with SJBC-SD01; sample dates and duplicate qualifiers will follow. The guide for sample identification is documented in the MPP. A summary of the sample identification scheme is presented in Table 4-2.

A listing of the sample identification numbers will be maintained by the field team leader, who will be responsible for enforcing the use of the standardized numbering system during all sampling activities.

4.1.4 Surveying

The 38 sample locations will be surveyed using a hand-held global positioning system (GPS) unit as samples are collected. The sample points will be incorporated into the GIS database.

4.1.5 Investigation-Derived Waste Management

A minimal amount of IDW, consisting of decontamination fluids and personal protective equipment (PPE), will be generated during this sampling program. Sampling equipment will be decontaminated prior to sampling and between samples. Since the amount of IDW fluids generated will be minimal (less than 2 gallons per day), the detergents and solvents will be disposed of by placing the fluid on the ground adjacent to the decontamination line.

4.2 Sample Analysis and Data Validation

CH2M HILL will track sample analyses and obtain results from the laboratory. Following the initial data evaluation (that will be used to identify sediment samples for bioassay and tissue residue analyses to be conducted) the analytical data generated during the investigation field program will be validated. This data validation will be conducted by an independent data validation subcontractor according to USEPA standard procedures. These validated data will be used to complete Step 6 of the ERA process for Blows Creek. A detailed discussion of quality control procedures for field investigations at SJCA is presented in the MWP and in the MQAPP.

TABLE 4-2
 Summary of Sample ID Scheme
 Blows Creek BERA Work Plan

First Segment		Second Segment		Third Segment
Installation	Site Number	Sample Type	Sample Location	Sample Date; Qualifier
AA	AA	AA	NN	NNAA
<u>Installation:</u> SJ = St. Juliens Creek Annex	<u>Site Number:</u> BC = Blows Creek	<u>Sample Type:</u> SD = Sediment SW = Surface Water	<u>Sample Location:</u> Sequential Location Number	<u>Sample Date:</u> NN- Last 2 digits of year <u>A = Quarter of the Year:</u> A = 1st Quarter B = 2nd Quarter C = 3rd Quarter D = 4th Quarter <u>Qualifier:</u> P - Duplicate

Notes: "A"= alphabetic "N"= numeric

Numbering format for QA/QC Samples:	
AA-NNNNNN	
<u>AA = QA/QC type:</u> TB = Trip Blank EB = Equipment Blank FB = Field Blank	NNNNN = DDMMYY (example = 082902)

Notes: Equipment blanks will not be taken after the first sample collection each day.

4.2.1 Sample Analysis

An expedited 14-day turnaround time will be used for chemical analysis of the Phase I sediment samples. A standard 28-day turnaround time will be used for all other chemical and all physical analytical samples. All analyses will be conducted at a contracted laboratory that fulfills all requirements of the U.S. Navy's QA/QC Program Manual and USEPA's Contract Laboratory Program (CLP). A signed certificate of analysis will be provided with each laboratory data package, along with a certificate of compliance certifying that all work was performed in accordance with the applicable federal, state, and local regulations. All analyses will be performed following the highest level of Navy guidance. Bioassays will be conducted in a manner consistent with USEPA guidelines (USEPA 1994).

4.2.2 Field Quality Control Procedures

QC duplicate samples and blanks are used to provide a measure of the internal consistency of the samples and to provide an estimate of the components of variance and the bias in the analytical process. QA procedures for laboratory toxicity tests will follow those described by the USEPA (USEPA 1994). Data will be collected to meet high-level data quality objectives (DQOs) as described in this document.

Three types of blanks can be generated during sampling activities: field blanks, equipment rinsate blanks, and temperature blanks. American Society for Testing Materials (ASTM) Type II water will be used for blanks. A summary of the sample identification scheme is presented in Table 4-2. The QC samples to be collected during the investigation are summarized in Table 4-3 for each medium and are described below.

One field blank will be collected per week to determine if there is any influence from ambient conditions in the sampling area location imparted to the sample. The field blank will be collected at one location where there is most likely to be ambient air contamination. If windy or dusty conditions are present during sample collection, field blanks will be collected daily.

Equipment blanks give an indication of the efficiency of decontamination procedures. One equipment blank will be collected per day for all non-disposable sampling equipment (hand auger, trowel, bowl, etc.), however; they will not be taken after the first sample collected each day.

A temperature blank will be included in each cooler containing samples for CLP analyses so that the laboratory can record the temperature without disturbing the samples. The temperature blank will be labeled, but will not be given a sample number nor will be listed as a sample on the Chain or Custody (COC) form.

Field duplicate samples are typically collected at a frequency of 1 per 10 field samples per matrix. The duplicates will be collected from randomly selected locations. The duplicate sample will be split evenly into two sample containers and submitted for analysis as two independent samples.

TABLE 4-3
 General Requirements for QC Sample Collection
 Blows Creek BERA Work Plan

QC Samples	QC Specified Collection Frequency
Field Duplicate	One per 10 samples (not counting other QC samples) per matrix or one duplicate per day and matrix, whichever is more frequent
Trip Blank	One per cooler containing samples collected for VOC analysis
Equipment (Rinsate) Blank	One per day per matrix
Field Blank	One per sampling event
Temperature Blank	One per cooler
Matrix Spike/Matrix Spike Duplicate	One per matrix for each group of up to 20 samples (including all QC samples) sent to a single laboratory

Matrix spike/matrix spike duplicate (MS/MSD) samples will be collected at a frequency of 1 for every 20 field soil samples collected. Analytical results of these samples indicate the impact the matrix (sediment) has on extracting the analyte for analysis. MS/MSD samples give an indication of the laboratory's analysis accuracy and precision within the sample matrix. Data validators will use these results to evaluate the accuracy of the analytical data.

4.2.3 Data Validation

CH2M HILL subcontractors approved by the Navy will validate analytical results. Data validators will use USEPA Region III guidance. Data that should be qualified will be appropriately flagged. Results for QA/QC samples will be reviewed and the data will be qualified further, if necessary. Finally, the dataset as a whole will be examined for consistency, anomalous results, reasonableness, and utility.

SECTION 5

Data Evaluation

The following sections describe the approach that will be used to evaluate the bioassay and chemical analytical data for use in completing Steps 7 and 8 of the ERA.

5.1 Evaluation of Bioassay Outcomes

A 28-day *L. plumulosus* laboratory sediment bioassay will be conducted with sediment samples collected from Blows Creek. These laboratory sediment tests will be used to evaluate potential toxicity resulting from chemicals in the sediment. Potential toxicity from the downgradient and reference samples will be determined by comparing response endpoints of organisms exposed to site samples to those exposed to laboratory control sediments. The endpoints to be tested include survival (percent mean survivors), growth (mean dry weight gain per day per adult survivor), and reproduction (number of offspring or neonates per adult). Laboratory control sediments are essentially free of contaminants and no toxic effects are expected from this treatment. In addition to being used to determine potential toxicity, the laboratory control sediments are used to ensure that the test protocols and/or the condition of the test organisms are not the cause of any observed adverse effect. According to test protocol, test acceptability is met if the control exhibits survival of greater than or equal to 80 percent and measurable reproduction and growth. In addition, no single replicate in the control can exhibit less than 60-percent survival in order to meet test acceptability requirements.

Each endpoint will be quantified, summarized, and analyzed by the contracted laboratory performing the toxicity tests. The endpoints for sediments associated with Sites 3, 4, and 5 will be analyzed to detect statistically significant differences from the control treatment. The contracted laboratory will evaluate the endpoint response data (e.g., growth, survival, and reproduction) to determine homogeneity of sample variances and normality of distribution. Datasets that are both homogeneous and exhibit a normal distribution will be evaluated using parametric statistical models (e.g., two-sample t-test). Those data that do not meet both criteria will be transformed (e.g., arcsine-square root) and retested for adherence to the conditions required for parametric testing. Otherwise, appropriate nonparametric analysis procedures (e.g., Kruskal-Wallis) will be used. The parametric or nonparametric procedures will be used to determine statistically significant differences ($\alpha=0.05$) in organism response (e.g., survival, reproduction, and growth) for each test treatment compared to controls.

A decision will then be made as whether or not the sediment samples exhibit toxicity. Survival, growth, and reproduction will be weighted equally when determining sediment toxicity. Table 5-1 shows the potential toxicity testing outcomes and decision process based on considerations of survival, growth, and reproduction:

TABLE 5-1
Determination of Toxicity—All Sediment Samples Compared to Control

Significant Effect ^a				Significant Effect?
Survival	Reproduction	Growth		
+	+	+		Y
+	+	-		Y
+	-	-		Y
-	+			Y
-	-	+		Y
-	-	-		N

A “+” indicates potential for adverse effect; “-“ indicates no potential for adverse effect

If any Blows Creek sediment samples indicate toxicity based on comparison to controls, the affected parameter (survival, growth, reproduction) from the potentially site-impacted sediment sample(s) will then be compared to the same parameter in the reference sediment sample. This comparison will be used to help differentiate between potential for adverse effects from facility-related sources with those potentially unrelated to the facility (Sample 1). The same statistical protocol described above will be used for this analysis. Sediment samples indicating a significantly greater adverse effect as compared to reference samples, will be determined to be “potentially impacted” by site-related sources. Factors besides chemicals (e.g., TOC, grain size) that could influence the bioassay outcome, will also be considered when interpreting the adverse effects indicated by the bioassay. Sediment samples that do not show a statistically significant difference in toxicity when compared to the reference sample will be determined to be not impacted.

5.2 Evaluation of Chemical Analytical and Physical Data

Chemical analytical and physical data collected during the Blows Creek investigation will be evaluated using the same approach as for the Steps 1 through 3 ERA. Consistent with the ERA, chemicals detected in sediment will be compared to direct exposure screening values, to evaluate potential risks from direct exposure to chemicals in sediment (benthic organisms), and will be modeled to evaluate potential risks associated with indirect exposure to mercury via accumulation in the food web. The same models, toxicity values, and aquatic receptors used in the Steps 1 through 3 ERA also will be used for the evaluation of potential for adverse effects with the sediment data collected from Blows Creek.

5.2.1 Weight of Evidence Analysis

The bioassay results will be used in conjunction with the sediment chemical analytical and physical data as part of a weight-of-evidence approach to evaluate the overall potential for adverse effects to benthic invertebrates. Table 5-2 shows the potential bioassay and chemistry outcomes and the decision process based on considerations of their possible outcomes:

TABLE 5-2
Integration of Bioassay and Chemistry Outcomes—All Sediment Sample Locations

Evaluation Outcome ^a			Conclusion
Bioassay Outcome	Chemistry Outcome		
+	+		Potential for adverse effect to benthic organisms
+	-		Outcome may be related to physical or chemical factor and more detailed consideration is warranted
-	+		Chemicals in sediment are not adversely affecting benthic organisms
-	-		No potential for adverse effect to benthic organisms

A “+” indicates potential for adverse effect; “-“ indicates no potential for adverse effect

If both the bioassay and chemistry outcomes indicate the potential for adverse effects at a sample location, then it will be concluded there is the potential for adverse effects to benthic organisms. Conversely, if both the bioassay and chemistry outcomes do not indicate the potential for adverse effects at a sample location, then it will be concluded there is no potential for adverse effects to benthic organisms at that location. These outcome combinations are the most straightforward to interpret. The other possible outcomes require more detailed consideration. If the bioassay outcome indicates the potential for adverse effect but no chemicals are detected in sediment at concentrations that would indicate the potential for adverse effect, then it is likely that a non-chemical factor such as sediment composition (e.g., grain size, sediment composition) and/or a laboratory artifact (e.g., sulfide, ammonia) caused the observed bioassay organism response. The possibility that chemicals are acting synergistically or additively will also be considered when interpreting this outcome combination. If it is determined that chemicals could be acting synergistically or additively, then it will be concluded there is the potential for adverse effects to benthic organisms at that site. If the bioassay outcome does not indicate the potential for adverse effect but the chemistry outcome suggests the potential for adverse effect, it will be concluded there is no potential for adverse effect to benthic organisms at that location. This outcome can occur when chemicals in sediment are not bioavailable or when the literature-based screening toxicity values (used for the comparison to chemical data) are protective of organisms that are more sensitive to chemicals than those used in the bioassay. Possible factors leading to this disparity of conclusion will be considered when evaluating this outcome combination.

5.2.2 Evaluation of Chemicals Potentially Causing Bioassay Organism Response

The primary objective of the Blows Creek investigation is to determine if there is a potential risk to ecological receptors from site-related contaminants. However, if the bioassay and chemistry data indicate toxicity, a more detailed evaluation will be conducted to determine if there is a relationship between bioassay organism response and a specific chemical or group of chemicals in sediment. Non-chemical factors that could influence the outcome of the bioassay (e.g., TOC, grain size) also will be considered. The objective of this evaluation is to identify the possible cause(s) of the observed toxicity, which would further facilitate the interpretation/evaluation of the risk outcomes. It should be recognized, however, that there

will be substantial variability in the chemical and physical composition of each sediment sample. Because the bioassay integrates all chemical and physical factors when measuring the toxicity of a sample, it may not be possible to establish a relationship between a single chemical or physical factor and the bioassay organism's response. If, however, the bioassay results indicate toxicity, the data will be reviewed using multiple regressions or other appropriate statistical analyses to determine if a relationship can be established to explain the observed outcome. Spatial patterns in toxicity will be evaluated, along with the chemical data, to determine if toxicity is related to Sites 3, 4, 5, and 19 or EPIC AOC-1 or to another facility or non-facility-related source.

5.3 Evaluation of Tissue Residue Data (Phase II)

Food web models in the Sites 3, 4, 5 and 6 Steps 1 through 3 ERA (CH2M HILL 2003) used measured sediment concentrations and literature-based bioaccumulation factors to estimate potential bioaccumulative risks to higher trophic-level species. Based on the outcome of the Steps 1 through 3 evaluation, tissue residue data for mercury was collected as part of the Phase II investigation. These data will be used instead of literature-based bioaccumulation concentration estimates to recalculate potential risks to higher trophic-level species. The tissue residue data for mercury will use the same models and assumptions that were used in Step 3 of the ERA.

5.4 BERA Report

The methods, results, analyses (including statistical analyses), agreements on decision parameters from preceding phase data, and risk characterization conclusions will be reported in the BERA Report. The report will evaluate the potential risk to ecological receptor populations associated with the Blows Creek watershed. If a risk exists, the report will identify the areas showing potential impact. Based on the evaluation of the results presented in the Draft Report, a Final Report will be prepared. The report will contain the following:

- An introductory section summarizing the sites, providing past history, and discussing objectives for this work.
- A summary of field investigation activities that will describe sample collection, laboratory analysis, and analytical results.
- Development of a conceptual site model for the ERA describing plausible pathways and receptors and initial comparison to ecological screening values for completed exposure pathways.
- Conclusions and recommendations regarding further action.

SECTION 6

Project Management and Staffing

This project will be managed and staffed by CH2M HILL's Virginia Beach office. The CH2M HILL Activity Manager, Mr. William Friedmann, will assume primary responsibility for ensuring that the work is performed in a manner acceptable to LANTDIV, the Base, and government regulatory agencies. Mr. Mike Elias will provide support.

Qualified CH2M HILL staff members will perform the BERA fieldwork. CH2M HILL will notify LANTDIV and the Base as to which CH2M HILL personnel will mobilize to the sites prior to initiating field activities. Dr. Steve Petron will provide senior review for the ERA.

SECTION 7

Project Schedule

This section presents the project schedule and the due dates of deliverables. Figure 7-1 and Table 7-1 show a breakdown on project milestones, primary deliverables, and assumed intervals for governmental review. Longer periods of review will result in an extended schedule.

TABLE 7-1
Project Schedule

Milestone	Duration	Start Date	Completion Date
Submit Draft BERA Work Plan	0 days	10/02/02	10/02/02
Subcontractor Procurement	5	10/09/02	10/15/02
Work Plan Review and Comments	70 days	10/03/02	01/08/03
Comment Resolution	119 days	01/09/03	6/24/03
Submit Final BERA Work Plan	30 days	6/25/03	08/05/03
Data Collection	14 days	09/22/03	10/09/03
Data Analysis	28 days	10/10/03	11/18/03
Data Validation	15 days	11/19/03	12/09/03
Data Loading	7 days	12/10/03	12/18/03
Data Evaluation	21 days	12/19/03	01/16/04
Prepare Draft BERA Report	21 days	01/05/04	02/02/04
Submit Draft BERA Report	1 days	02/03/04	02/04/04
Regulatory Review	45 days	02/04/04	03/16/04
Response to Regulatory Comments	14 days	03/17/04	04/05/04
Comment Resolution	7 days	04/06/04	04/14/04
Submit Final BERA Report	1 days	04/15/04	04/15/04

Figure 7-1
Project Schedule
St. Juliens Creek Annex
BERA Work Plan

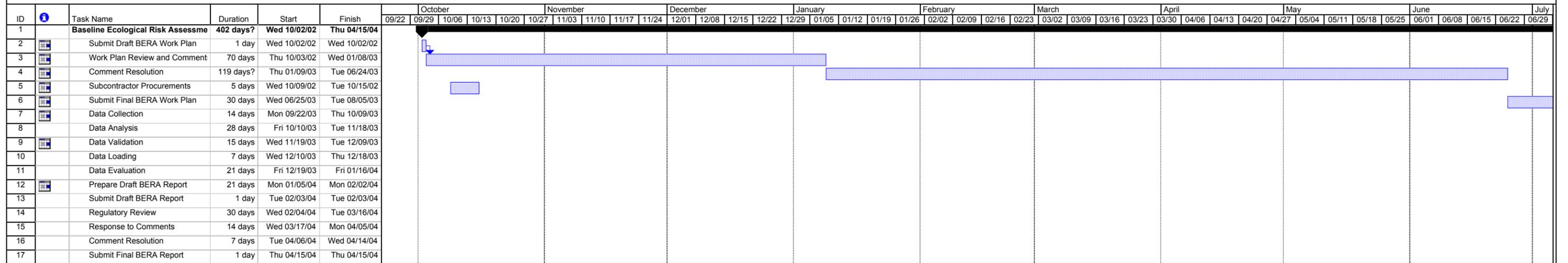
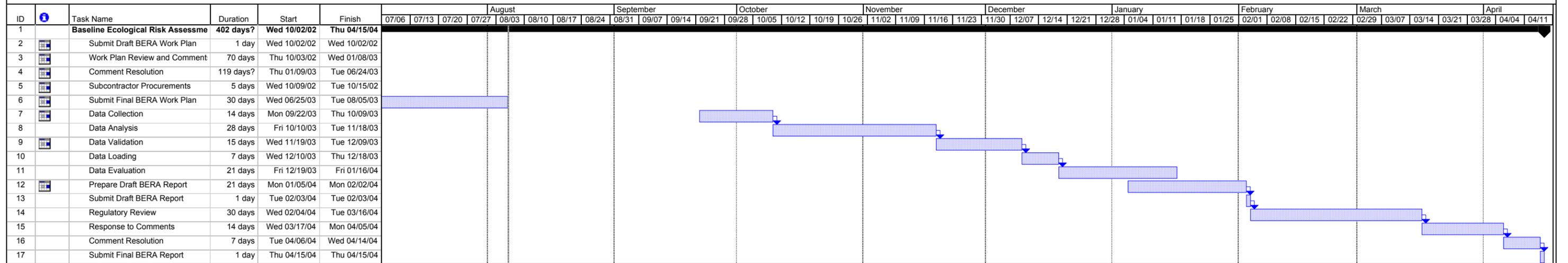


Figure 7-1
Project Schedule
St. Juliens Creek Annex
BERA Work Plan



Project: Figure 6-1
Date: Wed 08/06/03



SECTION 8

References

Chief of Naval Operations (CNO). April 1999. *Navy Policy for Conducting Ecological Risk Assessments*. Memorandum from Chief of Naval Operations to Commander, Naval Facilities Engineering Command. SER N453E/9U595355.

CH2M HILL. March 2003. *Remedial Investigation/Human Health Risk Assessment/Ecological Risk Assessment Report for Sites 3, 4, 5, and 6, St. Juliens Creek Annex, Chesapeake, Virginia*. Final.

CDM. July 2000. *Federal Master Project Plan, Naval Station Norfolk, St. Juliens Creek Annex, Chesapeake, Virginia*. Final.

Lippson, A.J. and R.L. Lippson. 1997. *Life in the Chesapeake Bay*. Second Edition. John Hopkins University Press.

USACE. 1996. *Preliminary Protocol for conducting 28-day chronic sub-lethal sediment bioassays using the estuarine amphipod Leptocheirus plumulosus (Shoemaker)*. Environmental Effects of Dredging Technical Notes, No. EEDP-01-36, US Army Engineer Waterways Experiment Station, Vicksburg, MS.

USEPA. 2001. *Methods for Assessing the Chronic Toxicity of Marine and Estuarine Sediment-associated Contaminants with the Amphipod Leptocheirus plumulosus - First Edition*. EPA/600/R-01/020.

USEPA. 1997. *Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Ecological Risk Assessments*. Interim Final. EPA/540/R-97/006.

USEPA. 1994. *Methods for Assessing the Toxicity of Sediment-Associated Contaminants with Estuarine and Marine Amphipods*. EPA600R94025.

Appendix A
Health and Safety Checklist and SOPs

St. Juliens Creek Annex – Field Performance Audit Checklist from Master Quality Assurance Project Plan

Project Responsibilities

Project No.: 138802.FI.EC Date: _____

Project Location: St. Juliens Creek Annex Signature: _____

Team Members: W. Friedmann, D. Holloway, C. Schwarz, & J. Culbreth

Yes ___ No ___ 1) Was a SAP Prepared?
Comments _____

Yes ___ No ___ 2) Was a briefing held for project participants?
Comments _____

Yes ___ No ___ 3) Were additional instructions given to project participants?
Comments _____

Yes ___ No ___ 4) Is the current approved SAP being used?
Comments _____

Sample Collection

Yes ___ No ___ 1) Is there a written list of sampling locations and descriptions?
Comments _____

Yes ___ No ___ 2) Are samples collected as stated in the FSP?

Comments _____

Yes ___ No ___ 3) Are samples collected in the type of containers specified in the FSP?

Comments _____

Yes ___ No ___ 4) Are samples preserved as specified in the FSP?

Comments _____

Yes ___ No ___ 5) Are the number, frequency, and type of samples collected as specified in the FSP?

Comments _____

Yes ___ No ___ 6) Are quality assurance checks performed as specified in the FSP?

Comments _____

Yes ___ No ___ 7) Are photographs taken and documented as specified in the FSP?

Comments _____

Document Control

Yes ___ No ___ 1) Have any accountable documents been lost?

Comments _____

Yes ___ No ___ 2) Have any accountable documents been voided?

Comments _____

Yes ___ No ___ 3) Have any accountable documents been disposed of?

Comments _____

Yes ___ No ___ 4) Are the samples identified with sample tags?

Comments _____

Yes ___ No ___

5) Are blank and duplicate samples properly identified?

Comments _____

Yes ___ No ___

6) Are samples listed on a chain-of-custody record?

Comments _____

Yes ___ No ___

7) Is chain-of-custody documented and maintained?

Comments _____

St. Juliens Creek Annex - Site-Specific Field Sampling Plan Checklist

This checklist supplements the Master Field Sampling Plan with site-specific information. Once completed for a specific project, it provides necessary field sampling information for each investigation. It is to be taken into the field with the Master FSP.

Site: St. Juliens Creek Annex – Blows Creek Baseline Ecological Risk Assessment

1. Tasks to be performed:

- | | |
|---|---|
| <input type="checkbox"/> Geophysical surveys | <input type="checkbox"/> In-situ groundwater sampling |
| <input type="checkbox"/> Soil gas surveys | <input type="checkbox"/> Aquifer testing |
| <input checked="" type="checkbox"/> Sediment Sampling | <input type="checkbox"/> Hydrogeologic measurements |
| <input type="checkbox"/> Surface soil sampling | <input type="checkbox"/> Biota sampling |
| <input type="checkbox"/> Soil boring installation | <input type="checkbox"/> Trenching |
| <input type="checkbox"/> Subsurface soil sampling | <input type="checkbox"/> Land surveying |
| <input type="checkbox"/> Monitoring well installation and development | <input type="checkbox"/> Investigation derived waste sampling |
| <input type="checkbox"/> Monitoring well abandonment | <input type="checkbox"/> Decontamination |
| <input type="checkbox"/> Groundwater sampling | <input checked="" type="checkbox"/> Other: Surface water |

2. Field measurements to be taken:

- | | |
|--|---|
| <input checked="" type="checkbox"/> temperature | <input type="checkbox"/> surveying |
| <input checked="" type="checkbox"/> pH | <input type="checkbox"/> magnetometry |
| <input checked="" type="checkbox"/> dissolved oxygen | <input checked="" type="checkbox"/> global positioning system |
| <input checked="" type="checkbox"/> turbidity | <input type="checkbox"/> soil gas parameters (list): |
| <input checked="" type="checkbox"/> specific conductance | <input type="checkbox"/> combustible gases |
| <input checked="" type="checkbox"/> organic vapor monitoring | <input type="checkbox"/> water-level measurements |
| <input type="checkbox"/> geophysical parameters (list): | <input type="checkbox"/> pumping rate |
| <input type="checkbox"/> electromagnetic induction | <input type="checkbox"/> other _____ |
| <input type="checkbox"/> ground-penetrating radar | |

3. Sampling program (nomenclature, etc.):

- As per Section 3.1 of Master FSP Other

4. Map of sediment and surface water sampling locations: See work plan Figure 3-1.

5. Table of field samples to be collected: See work plan Table 3-1.

6. Applicable SOPs (Volume 1 of Master Project Plans) or references to specific pages in Master FSP:

- Flat Bottom Boat Sampling Operations
- Field Measurement of pH and Eh
- Field Measurement of pH

- Field Measurement of Specific Conductance and Temperature
- Field Measurement of Dissolved Oxygen
- Chain-of-Custody
- Sediment Sampling
- Homogenization of Soil and Sediment Samples
- Surface Water Sampling
- VOC Sampling – Water
- Preserving Non-VOC Aqueous Samples
- Field Filtering
- Field Rinse Blank Preparation
- Decontamination of Personnel and Equipment

6. Site-specific procedures or updates to protocols established in the Master FSP:
Described in the work plan.

St. Juliens Creek Annex - Site-Specific Health and Safety Plan

This checklist must be used in conjunction with the Site-specific HASP. This checklist is intended for use by CH2M HILL employees only. All CH2M HILL employees performing tasks under this checklist must read and sign both this checklist and the Site-specific HASP and agree to abide by their provisions (see EMPLOYEE SIGNOFF attached to the Site-specific HASP).

Site: St. Juliens Creek Annex – Blows Creek Baseline Ecological Risk Assessment

Location(s): Sampling Location Maps attached (see work plan)

This document shall be maintained on site with the Site-specific HASP. It will include as attachments from the work plan a site map and the site characterization and objectives for this site. The procedures described in the Master Health and Safety Plan will be followed unless otherwise specified in this Site-Specific Health and Safety Plan.

1. HAZWOPER-Regulated Tasks

<input type="checkbox"/> Test pit and excavation	<input type="checkbox"/> Groundwater sampling
<input type="checkbox"/> Soil boring installation	<input type="checkbox"/> Aquifer testing
<input type="checkbox"/> Hollow stem boring	<input type="checkbox"/> Hydrologic measurements
<input type="checkbox"/> Geophysical surveys	<input checked="" type="checkbox"/> Surface water sampling
<input type="checkbox"/> Hand augering	<input type="checkbox"/> Biota sampling
<input type="checkbox"/> Subsurface soil sampling	<input type="checkbox"/> Investigation-derived waste (drum) sampling and disposal
<input type="checkbox"/> Surface soil sampling	<input type="checkbox"/> Observation of loading of material for offsite disposal
<input type="checkbox"/> Soil gas surveys	<input type="checkbox"/> Oversight of remediation and construction
<input checked="" type="checkbox"/> Sediment sampling	<input type="checkbox"/> Other _____
<input type="checkbox"/> Monitoring well/drive point installation	
<input type="checkbox"/> Monitoring well abandonment	

2. Hazards of Concern: (Check as many as are applicable. Refer to Section 3 of Master H&S Plan for control measures):

<input type="checkbox"/> Heat stress	<input type="checkbox"/> Confined space entry
<input type="checkbox"/> Cold stress	<input type="checkbox"/> Trenches, excavations
<input type="checkbox"/> Buried utilities, drums, tanks	<input checked="" type="checkbox"/> Protruding objects
<input type="checkbox"/> Inadequate illumination	<input type="checkbox"/> Vehicle traffic
<input type="checkbox"/> Drilling	<input type="checkbox"/> Ladders, scaffolds
<input checked="" type="checkbox"/> Heavy equipment	<input type="checkbox"/> Fire
<input checked="" type="checkbox"/> Working near water	<input checked="" type="checkbox"/> Working on water
<input type="checkbox"/> Flying debris	<input checked="" type="checkbox"/> Snakes or insects
<input type="checkbox"/> Gas cylinders	<input checked="" type="checkbox"/> Poison ivy, oak, sumac
<input type="checkbox"/> Noise	<input checked="" type="checkbox"/> Ticks
<input checked="" type="checkbox"/> Slip, trip, or fall hazards	<input type="checkbox"/> Radiological
<input checked="" type="checkbox"/> Back injury	<input type="checkbox"/> Other: _____

3. Contaminants of Concern (List if known. Refer to Site-specific HASP)

Semi volatile organic compounds, metals

4. Personnel (List CH2M HILL field team members and telephone numbers):

Field team leader(FTL)	Bill Friedmann	757-460-3734 ext. 19
Site safety coordinator(SSC)	Bill Friedmann	757-460-3734 ext. 19
Alternate FTL and SSC	Dan Holloway	757-460-3734 ext. 30
Field team members	Carrie Schwarz	757-460-3734 ext. 14
	Jamie Culbreth	757-460-3734 ext. 39

5. Contractors/Subcontractors: No subcontractor will be hired or be the responsibility of CH2M HILL.

_____ Procedures as per Master and Site-specific HASP

_____ Other _____

Name: To be added _____

Contact: To be added _____

Telephone: To be added _____

6. Level of personal protective equipment (PPE) required: D
Refer to Master HASP, Site-specific HASP, CH2M HILL SOPs, and Respiratory Protection, Section 2 of the Site Safety Notebook.

7. Air monitoring instruments to be used (refer to Master HSP for action levels):

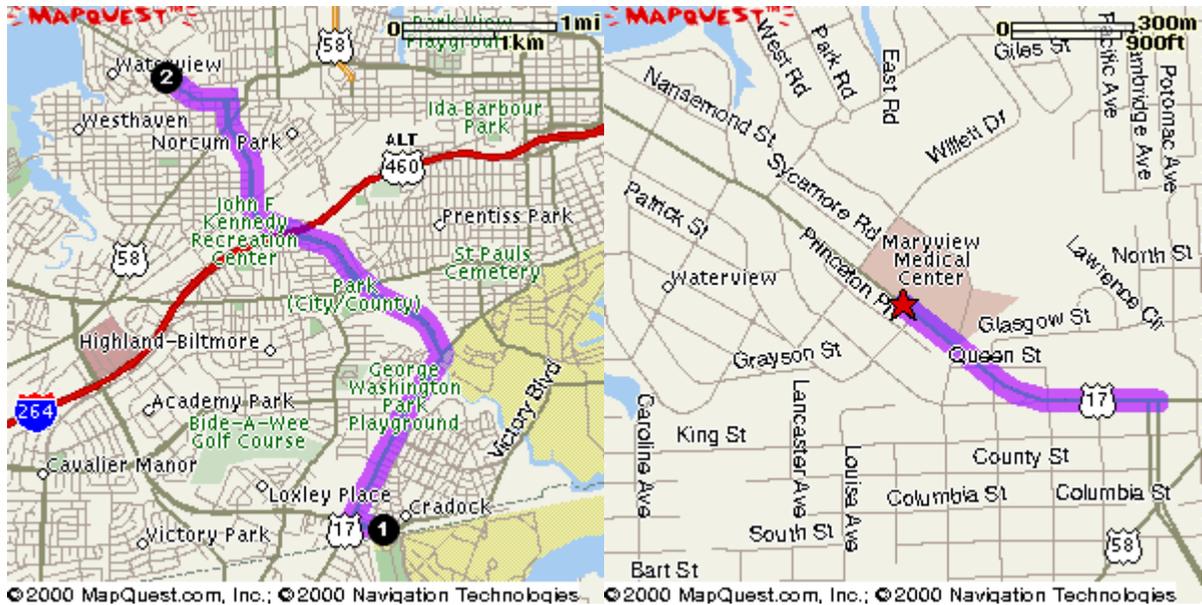
<u> X </u> OVM 10.6	_____ FID
_____ CGI	_____ Dust monitor
_____ O ₂	_____ PID

8. Decontamination procedures:

_____ As per Section 7 of Master HASP

_____ Other

9. List any other deviations or variations from the Master HASP: None
10. Emergency Response (Check that all names and numbers are correct in site-specific HASP and attach corrected page to this checklist)
11. Map to hospital (See site-specific HASP)



Driving Directions to Maryview Medical Center

	Direction	Miles
1	Turn LEFT onto VICTORY BLVD/VA-239 W.	0.0
2	Turn RIGHT onto US-17/GEORGE WASHINTON HIGHWAY	0.2
3	Turn LEFT onto US-17 Alternate/FREDRICK BLVD.	1.2
4	Turn LEFT onto HIGH ST.	2.3
5	Turn RIGHT into MARYVIEW MEDICAL CENTER	0.4

12. Emergency Contacts (Check that all names and numbers are correct in site-specific HASP)

Field Measurement of Specific Conductance and Temperature

I. Purpose and Scope

The purpose of this procedure is to provide a general guideline for field measurement of specific conductivity and temperature of groundwater samples. The following general discussion applies to most commonly used meters but may differ between specific brands. The operator's manual should be consulted for specific calibration and operating procedures.

II. Equipment and Materials

- Conductivity meter and electrode
- Distilled water in squirt bottle
- Standard potassium chloride (KCl) solution (0.01 N)

III. Procedures and Guidelines

A. Technical:

Detection limit = 1 $\mu\text{mho/cm}$ @ 25°C; range = 0.1 to 100,000 $\mu\text{mho/cm}$

B. Calibration:

Calibrate prior to initial daily use with standard solution. The standards should have different orders of conductance. Clean probe according to manufacturer's recommendations. Duplicates should be run once every 10 samples. Calibration procedure:

1. With mode switch in OFF position, check meter zero. If not zeroed, set with zero adjust.
2. Plug probe into meter.

3. Turn mode switch to red line and turn red line knob until needle aligns with red line on dial. If they cannot be aligned, change the batteries.
4. Immerse probe in 0.01 N standard KCl solution. Do not allow the probe to touch the sample container.
5. Set the mode control to TEMPERATURE. Record the temperature on the bottom scale of the meter in degrees C.
6. Turn the mode switch to appropriate conductivity scale (i.e., x100, x10, or x1). Use a scale that will give a midrange output on the meter.
7. Wait for the needle to stabilize. Multiply reading by scale setting and record the conductivity.
8. If the conductivity meter does not perform an automatic temperature adjustment, the conductivity may be adjusted to 25°C using the formula:

$$G_{25} = G_T / [1 + 0.02 (T - 25)]$$

Where:

G_{25} = conductivity at 25°C, $\mu\text{mho/cm}$

T = temperature of sample, degrees C

G_T = conductivity of sample at temperature T, $\mu\text{mho/cm}$

The table below lists the values of conductivity that the calibration solution would have if the distilled water were totally nonconductive; however, even water of high purity will possess a small amount of conductivity.

Temperature °C	Conductivity ($\mu\text{mho/cm}$)
15	1,141.5
16	1,167.5
17	1,193.6
18	1,219.9
19	1,246.4
20	1,273.0
21	1,299.7
22	1,326.6
23	1,353.6
24	1,380.8
25	1,408.1
26	1,436.5
27	1,463.2

28	1,490.9
29	1,518.7
30	1,546.7

9. Rinse the probe with deionized water.

C. Sample Measurement:

Pour the sample into a small beaker and place the probe in the sample. Note and record the reading. Rinse the probe with deionized water when done.

IV. Attachments

- Conductivity meter calibration sheet

V. Key Checks and Preventive Maintenance

- Check battery.
- Calibrate meter.
- Clean probe with deionized water when done.
- When reading results, note sensitivity settings.
- Refer to operations manual for recommended maintenance.
- Check batteries, and have a replacement set on hand.

CONDUCTIVITY METER CALIBRATION SHEET

<u>Date</u>	<u>Time</u>	<u>Analyst Initials</u>	<u>Instrument Readings</u>		<u>Comments</u>
			<u>Uncalibrated @ EC=225</u>	<u>Calibrated @ EC=225</u>	

Decontamination of Personnel and Equipment

I. Purpose

To provide general guidelines for the decontamination of personnel, sampling equipment, and monitoring equipment used in potentially contaminated environments.

II. Scope

This is a general description of decontamination procedures.

III. Equipment and Materials

- Demonstrated analyte-free, deionized (“DI”) water (specifically, ASTM Type II water)
- Distilled water
- Potable water; must be from a municipal water supplier, otherwise an analysis must be run for appropriate volatile and semivolatile organic compounds and inorganic chemicals (e.g., Target Compound List and Target Analyte List chemicals)
- 2.5% (W/W) trisodium phosphate (“TSP”) and water solution
- Concentrated (V/V) pesticide grade methanol (DO NOT USE ACETONE)
- 10% (V/V) nitric acid (HNO₃) and water solution (only ultrapure grade HNO₃ is to be used)
- Large plastic pails or tubs for TSP and water, scrub brushes, squirt bottles for TSP, methanol and water, plastic bags and sheets
- DOT approved 55-gallon drum for disposal of waste
- Phthalate-free gloves
- Decontamination pad and steam cleaner/high pressure cleaner for large equipment

IV. Procedures and Guidelines

A. PERSONNEL DECONTAMINATION

To be performed after completion of tasks whenever potential for contamination exists, and upon leaving the exclusion zone.

1. Wash boots in TSP solution, then rinse with water. If disposable latex booties are worn over boots in the work area, rinse with TSP solution, remove, and discard into DOT approved 55-gallon drum.
2. Wash outer gloves in TSP solution, rinse, remove, and discard into DOT approved 55-gallon drum.
3. Remove disposable coveralls ("Tyveks") and discard into approved 55-gallon drum.
4. Remove respirator (if worn).
5. Remove inner gloves and discard.
6. At the end of the work day, shower entire body, including hair, either at the work site or at home.
7. Sanitize respirator if worn.

B. SAMPLING EQUIPMENT DECONTAMINATION—GROUNDWATER SAMPLING PUMPS

Sampling pumps are decontaminated after each use as follows.

1. Don phthalate-free gloves.
2. Spread plastic on the ground to keep hoses from touching the ground
3. Turn off pump after sampling. Remove pump from well and place pump in decontamination tube, making sure that tubing does not touch the ground
4. Turn pump back on and pump 1 gallon of TSP solution through the sampling pump.
5. Rinse with 1 gallon of 10% methanol solution pumped through the pump. (DO NOT USE ACETONE).
6. Rinse with 10% HNO₃ solution pumped through the pump, when sampling for inorganics (carbon steel split spoons will be rinsed with a 1% solution).
7. Rinse with 1 gallon of tap water.
8. Rinse with 1 gallon of deionized water.
9. Keep decontaminated pump in decontamination tube or remove and wrap in aluminum foil or clean plastic sheeting.

10. Collect all rinsate and dispose of in a DOT approved 55-gallon drum.

C. SAMPLING EQUIPMENT DECONTAMINATION—OTHER EQUIPMENT

Reusable sampling equipment is decontaminated after each use as follows.

1. Don phthalate-free gloves.
2. Prior to entering the potentially contaminated zone, wrap soil contact points in aluminum foil (shiny side out).
3. Rinse and scrub with potable water.
4. Wash all equipment surfaces that contacted the potentially contaminated soil/water with TSP solution.
5. Rinse with potable water.
6. Rinse with 10% HNO₃ solution when sampling for inorganics (carbon steel split spoons will be rinsed with a 1% solution).
7. Rinse with distilled or potable water and methanol solution (DO NOT USE ACETONE).
8. Air dry.
9. Rinse with deionized water.
10. Completely air dry and wrap exposed areas with aluminum foil (shiny side out) for transport and handling if equipment will not be used immediately.
11. Collect all rinsate and dispose of in a DOT approved 55-gallon drum.

D. HEALTH AND SAFETY MONITORING EQUIPMENT DECONTAMINATION

1. Before use, wrap soil contact points in plastic to reduce need for subsequent cleaning.
2. Wipe all surfaces that had possible contact with contaminated materials with a paper towel wet with TSP solution, then a towel wet with methanol solution, and finally three times with a towel wet with distilled water. Dispose of all used paper towels in a DOT approved 55-gallon drum.

E. SAMPLE CONTAINER DECONTAMINATION

The outsides of sample bottles or containers filled in the field may need to be decontaminated before being packed for shipment or handled by personnel without hand protection. The procedure is:

1. Wipe container with a paper towel dampened with TSP solution or

immerse in the solution AFTER THE CONTAINERS HAVE BEEN SEALED. Repeat the above steps using potable water.

2. Dispose of all used paper towels in a DOT approved 55-gallon drum.

F. HEAVY EQUIPMENT AND TOOLS

Heavy equipment such as drilling rigs, drilling rods/tools, and the backhoe will be decontaminated upon arrival at the site and between locations as follows:

1. Set up a decontamination pad in area designated by the Navy
2. Steam clean heavy equipment until no visible signs of dirt are observed. This may require wire or stiff brushes to dislodge dirt from some areas.

V. Attachments

None.

VI. Key Checks and Items

- Clean with solutions of TSP, methanol, nitric acid, and distilled water.
- Do not use acetone for decontamination.
- Drum all contaminated rinsate and materials.
- Decontaminate filled sample bottles before relinquishing them to anyone.

Field Measurement of Dissolved Oxygen

I. Purpose

To provide general guidelines for the calibration and use of the Dissolved Oxygen (DO) meter.

II. Scope

The following general discussion applies to more commonly used meters but may differ between specific brands. The operator's manual should be consulted for specific calibration and operation procedures.

III. Equipment and Materials

- Operations manual
- A DO probe and readout/control unit with batteries
- Electrolyte solution (KCl dissolved in deionized water) and probe membrane

IV. Procedures and Guidelines

A. Calibration

Calibrate prior to initial daily use before any readings are taken. Clean probe according to manufacturer's recommendations.

1. Prepare DO probe according to manufacturer's recommended procedures using electrolyte solution.
2. In the off position, set the pointer to zero using the screw in the center of the meter panel.
3. Turn function switch to red line and adjust using red line knob until the meter needle aligns with red mark at the 31 degrees C position.
4. Turn function switch to zero and adjust to zero using the zero control knob.
5. Attach prepared probe and adjust retaining ring finger tight.
6. Allow 15 minutes for optimum probe stabilization (when meter is off or during disconnection of the probe).
7. For YSI meters, place probe in hollow stopper that is supplied for use with the YSI Calibration Chamber.

8. Place approximately 1/2 inch of deionized water into a 4-ounce, wide mouth screw cap bottle. Keep this bottle capped and with the DO meter.
9. Just before use, shake the bottle to saturate the water with air.
10. Remove cap, place probe in bottle keeping an air-tight seal around the rubber stopper. Swirl water around in the bottle while waiting for conditions to reach equilibrium.
11. Shield chamber from sun and wind to avoid temperature fluctuations during calibration.
12. Turn function switch to temperature and record temperature reading. Determine calibration factor for that temperature and altitude correction factor from tables supplied by manufacturer.
13. Multiply the calibration factor by the correction factor to get a corrected calibration value.
14. Turn function switch to appropriate ppm range and adjust the calibrate knob until the meter reads the corrected calibration value. Wait two minutes to verify calibration value. Re-adjust as necessary.

B. Procedure

1. Before going out into the field:
 - a) Check batteries
 - b) Obtain fresh electrolyte solution
 - c) Prepare DO probe
2. Calibrate meter using calibration procedure.
3. Place probe in water to be measured. The probe should be moved through the water at 1 ft/sec or use a probe with a built-in stirrer.
4. Allow sufficient time for probe to stabilize to water temperature and DO. Record DO meter reading.

V. Attachments

DO Meter Calibration Sheet.

VI. Key Checks and Items

- Battery check
- Calibration

VII. Preventive Maintenance

- Refer to operation manual for recommended maintenance.
- Check batteries, have replacement set on hand.

**DO METER
CALIBRATION SHEET**

Date	Time	Analyst's Signature	Temp (C)	Alt. (ft)	Predict (ppm O₂)	Actual (ppm O₂)	Comment
-------------	-------------	--------------------------------	---------------------	----------------------	--	---------------------------------------	----------------

Field Filtering

I. Purpose

To provide a general guideline for the field filtering of water samples for dissolved metals analysis.

II. Scope

This is a general discussion of the standard method of field filtering techniques. Operating manuals should be consulted regarding specific procedures.

III. Equipment and Materials

- Geotech Filtering apparatus or equivalent
- Pump
- nitric acid (HNO₃) solution - high grade - reagent grade not acceptable
- Glass fiber prefilters
- Vacuum source
- 45 µm cellulose acetate filters
- inline filters

IV. Procedures and Guidelines

A. REAGENT PREPARATION

1. 10% HNO₃ solution: Add about 900 ml of ASTM Type II water to a 1 liter Erlenmeyer flask. Using a graduated cylinder, ASTM Type II, add 100 ml concentrated HNO₃ to the DI water while stirring.

B. PROCEDURE

1. Attach a vacuum source (pump, syringe, etc.) or a Q.E.D. online filter or equivalent to the receiver assembly.
2. Flush the entire filter system with 10% HNO₃ solution. Open assembly, discard rinsate, and reassemble unit.
3. Flush the entire filter system with 60 ml ASTM Type II water. Open assembly, discard rinsate and reassemble unit (not required when using Q.E.D. online filter).
4. Filter sample and transfer to polyethylene bottle (with preservative) for shipment.

5. Discard filter assembly and prefilter.

V. Attachments

None.

VI. Key Checks and Items

- 10% HNO₃ solution for cleaning
- All water must be ASTM Type II
- Prefilter with glass fiber filters if sample is turbid
- Record lot number of nitric acid and water
- Note monitoring wells with high concentrations of suspended solids in field notebooks
- The equipment blank collected with the sample is called a filtration blank and is collected through the filter.

Flat Bottom Boat Transport and Sampling Operations

I. Purpose

Flat bottom boat sampling operations are a non-standard practice of RCRA/CERCLA investigations. The objective of these operations is to access those sample locations inaccessible to larger, deeper draft, motorized water craft. This document outlines safe practices for the transportation, launching, and recovery of a flat-bottom boat. Along with procedures aimed at safe and accurate flat bottom boat-based sampling operations.

II. Scope

The provisions of this SOP apply to all program and project personnel engaged directly in technical boating operations, whether planning or executing those operations. These provisions apply whenever technical boating equipment or activities are used or included in project operations.

III. Responsibilities

Project Manager - The Project Manager is responsible for ensuring that project-specific plans for boating operations and federal and state boating safety regulations are in accordance with these procedures, where applicable, or that other approved procedures are developed. The project manager should ensure that the Field Team Leader is familiar and comfortable with trailer-based flat bottom boat operation.

Field Team Leader - The Field Team Leader is responsible for ensuring that these boating procedures are implemented in the field, and for ensuring that personnel performing these activities have been briefed and trained to execute these procedures. The Field Team leader will be responsible for the transportation, launching, and recovery of the flat bottom boat.

Sampling Personnel - It is the responsibility of the sampling personnel to follow these procedures or to follow documented, project-specific procedures as directed by the Field Team Leader and/or the Project Manager. The sampling personnel are responsible for the proper sampling procedures, proper operation of the boat and adherence to waterborne health and safety procedures.

IV. Procedures

The following procedures outline the planning and execution of flat bottom boat sampling operations:

1. All operations involving technical boating will be directed by qualified and experienced boater as the team leader.
2. All persons participating in boating operations will be directed by the Team Leader.
3. All persons participating in boating operations will have been trained by the Team Leader or provide proof of experience in operating such water craft.
4. All water craft shall operate on a "line of sight" rule. No water craft will go out of sight of each other.
5. All personnel shall wear their Personnel Floatation Devices at all times while they are on the water.
6. The boating team will include at least one person qualified in First Aid/CPR for nonstandard conditions (for example: fire rescue, air/land/sea rescue).
7. All personnel shall wear bright colors (for example: hunter orange, yellow, etc.) to enhance their visibility to one another.
8. All personnel shall collect one sample at a time, and return that sample to the "mother ship," the dock, or other location as determined by site conditions and situation.

Team Leader has final authority on operations with regards to weather and water conditions.

The following procedures outline the transportation of the flat bottom boat using a single axle "v-bunk" trailer. This action should be performed by staff comfortable with trailer operation, including backing:

1. Perform visual inspection of trailer, checking tire pressure, safety chain integrity, smooth operation of trailer coupler, and for loose bolts in the trailer frame. Also check for license plate, and proper boat and trailer registration in vehicle glove compartment.
2. Open trailer coupler by removing safety pin and lifting latch to the up and open position. Attach to ball of towing vehicle, engage coupling mechanism and replace safety pin. Give trailer tongue a few hard tugs, make sure trailer stays snugly connected to ball and there is minimal play between parts.
3. Attach safety chains to towing vehicle, chains should be crisscrossed if possible and should not drag on the ground. Do not connect the safety chains in the same location on the hitch.

Homogenization of Soil and Sediment Samples

I. Purpose

The homogenization of soil and sediment samples is performed to minimize any bias of sample representativeness introduced by the natural stratification of constituents within the sample.

II. Scope

Standard techniques for soil and sediment homogenization and equipment are provided in this SOP. These procedures do not apply to aliquots collected for TCL VOCs or field GC screening; samples for these analyses should NOT be homogenized.

III. Equipment and Materials

Sample containers, stainless steel spoons or spatulas, and stainless steel pans.

IV. Procedures and Guidelines

Soil and sediment samples to be analyzed for semivolatiles, pesticides, PCBs, metals, cyanide, or field XRF screening should be homogenized in the field. After a sample is taken, a stainless steel spatula should be used to remove the sample from the split spoon or other sampling device. The sampler should not use fingers to do this, as gloves may introduce organic interferences into the sample.

Samples for VOCs should be taken immediately upon opening the spoon and should not be homogenized.

Prior to homogenizing the soil or sediment sample, any rocks, twigs, leaves, or other debris should be removed from the sample. The sample should be placed in a decontaminated stainless steel pan and thoroughly mixed using a stainless steel spoon. The soil or sediment material in the pan should be scraped from the sides, corners, and bottom, rolled into the middle of the pan, and initially mixed. The sample should then be quartered and moved to the four corners of the pan. Each quarter of the sample should be mixed individually, and then rolled to the center of the pan and mixed with the entire sample again.

All stainless steel spoons, spatulas, and pans must be decontaminated following procedures specified in SOP Decontamination of Personnel and Equipment prior to homogenizing the sample. A composite equipment rinse blank of homogenization equipment should be taken each day it is used.

Field Rinse Blank Preparation

I. Purpose

To prepare a blank to determine adequacy of decon procedures and whether any cross-contamination is occurring during sampling.

II. Scope

The general protocols for preparing the rinse blank are outlined. The actual equipment to be rinsed will depend on the requirements of the specific sampling procedure.

III. Equipment and Materials

- Blank liquid (use ASTM Type II grade water)
- Sample bottles as appropriate
- Gloves
- Preservatives as appropriate

IV. Procedures and Guidelines

- A. Decontaminate all sampling equipment that has come in contact with sample according to SOP Decontamination of Personnel and Equipment.
- B. To collect the sample for volatiles analysis, pour blank water over one piece of equipment and into 40-ml vials until there is a positive meniscus and seal vials. Note the sample number and associated piece of equipment in the field notebook.

For non-volatiles, one aliquot is to be used for equipment. For example, if a pan and trowel are used, place trowel in pan and pour blank fluid in pan such that pan and trowel surfaces which contacted the sample are contacted by the blank fluid. Pour blank fluid from pan into appropriate sample bottles.

Do not let the blank fluid come in contact with any equipment that has not been decontaminated.

- C. Document and ship samples in accordance with the procedures for other samples.
- D. Collect next field sample.

V. Attachments

None.

VI. Key Checks and Items

- Wear gloves.
- Do not use any non-decontaminated equipment to prepare blank.
- Use ASTM-Type II grade water.

Sediment Sampling

I. Purpose

These general outlines describe the collection and handling of sediment samples during field operations.

II. Scope

The sediment sampling procedures generally describe the equipment and techniques needed to collect representative sediment samples. Operator's manual, if available, should be consulted for specific details.

III. Equipment and Materials

- Sample collection device (hand corer, scoop, dredge, grab sampler, or other suitable device)
- Stainless steel spoon or spatula for media transfer
- Measuring tape
- Log book
- Personal protection equipment (rubber or latex gloves, boots, hip waders, etc.)
- Materials for classifying soils, particularly the percentage of fines
- Sample jars, including jars for Total Organic Carbon and pH, as appropriate

IV. Procedures and Guidelines

1. Field personnel will start downstream and work upstream to prevent contamination of unsampled areas.
2. Make a sketch of the sample area showing important nearby river features and permanent structures that can be used to locate the sample points on a map. Whenever possible, include measured distances from such identifying features. Also include depth and width of waterway, rate of flow, type and consistency of sediment, and point and depth of sample removal (along shore, mid-channel, etc).
3. Transfer sample into appropriate sample jars with a stainless steel spoon or utensil. The sampler's fingers should never touch the sediment since gloves may introduce organic interferences into the sample. Classify the soil type of

the sample using the Unified Soil Classification System, noting particularly the percentage of silt and clay.

4. Samples for volatile organics should immediately be placed in jars. Rocks and other debris should be removed before placement in jars.
5. For channel sampling, be on the alert for submerged hazards (rocks, tree roots, drop-offs, loss silt and muck) which can make wading difficult.
6. Sample sediment for TOC and pH also, to give context to organic and inorganic data during the risk assessment.
7. Follow the site safety plan designed for the specific nature of the site's sampling activities and locations.
8. Decontaminate all sampling implements and protective clothing according to prescribed procedures.

V. Attachments

None.

VI. Key Checks and Items

- Start downstream, work upstream.
- Log exact locations using permanent features.
- Beware of hidden hazards.

Packaging and Shipping Procedures

I. Low-Concentration Samples

- A. Prepare coolers for shipment:
 - Tape drains shut.
 - Affix “This Side Up” labels on all four sides and “Fragile” labels on at least two sides of each cooler.
 - Place mailing label with laboratory address on top of coolers.
 - Fill bottom of coolers with about 3 inches of vermiculite.
- B. Arrange decontaminated sample containers in groups by sample number. Consolidate VOC samples into one cooler to minimize the need for trip blanks.
- C. Affix appropriate adhesive sample labels to each container. Protect with clear label protection tape.
- D. Seal each sample bottle within a separate ziplock plastic bag or bubble wrap, if available. Tape the bag around bottle. Sample label should be visible through the bag.
- E. Arrange sample bottles in coolers so that they do not touch.
- F. If ice is required to preserve the samples, cubes should be repackaged in zip-lock bags and placed on and around the containers.
- G. Fill remaining spaces with vermiculite.
- H. Complete and sign chain-of-custody form (or obtain signature) and indicate the time and date it was relinquished to Federal Express or the courier.
- I. Separate copies of forms. Seal proper copies (traffic reports, packing lists) along with a return address label within a large zip-lock bag and tape to inside lid of cooler.
- J. Close lid and latch.
- K. Carefully peel custody seals from backings and place intact over lid openings (right front and left back). Cover seals with clear protection tape.
- L. Tape cooler shut on both ends, making several complete revolutions with strapping tape. **Do not** cover custody seals.

- M. Relinquish to Federal Express or to a courier arranged with the laboratory. Place airbill receipt inside the mailing envelope and send to the sample documentation coordinator along with the other documentation.

II. Medium- and High-Concentration Samples:

Medium- and high-concentration samples are packaged using the same techniques used to package low-concentration samples, with several additional restrictions. First, a special airbill including a Shipper's Certification for Restricted Articles is required. Second, "Flammable Liquid N.O.S." or "Flammable Solid N.O.S." (as appropriate) labels must be placed on at least two sides of the cooler. Third, sample containers are packaged in metal cans with lids before being placed in the cooler, as indicated below:

- Place approximately ½ inch of vermiculite in the bottom of the can.
- Position the sample jar in the zip-loc bag so that the sample tags can be read through the plastic bag.
- Place the jar in the can and fill the remaining volume with vermiculite.
- Close the can and secure the lid with metal clips.
- Write the traffic report number on the lid.
- Place "This Side Up" and "Flammable Liquid N.O.S." or "Flammable Solid N.O.S." (as appropriate) labels on the can.
- Place the cans in the cooler.
- For medium concentration samples, ship samples with ice or "blue ice" inside the coolers. (Double bag ice in zip-lock plastic bags.)

III. Special Instructions for Shipping Medium and High Concentration Samples by Federal Express

- A. Label cooler as hazardous shipment:
- Write shipper's address on outside of cooler. If address is stenciled on, just write "shipper" above it.
 - Write or affix sticker saying "This Side Up" on two adjacent sides.
 - Write or affix sticker saying "ORM-E" with box around it on two adjacent sides. Below ORM-E, write NA#9188.
 - Label cooler with "Hazardous Substance, N.O.S." and "liquid" or "solid," as applicable.

- B. Complete the special shipping bill for restricted articles.
- Under Proper Shipping Name, write “Hazardous Substance, N.O.S.” and “liquid” or “solid,” as applicable.
 - Under Class, write “ORM-E.
 - ”Under Identification No., write NA No. 9188.
- C. For high concentration samples, ship samples with "blue ice" only inside coolers.

Chain-of-Custody

I Purpose

The purpose of this SOP is to provide information on chain-of-custody procedures to be used under the CLEAN Program.

II Scope

This procedure describes the steps necessary for transferring samples through the use of Chain-of-Custody Records. A Chain-of-Custody Record is required, without exception, for the tracking and recording of samples collected for on-site or off-site analysis (chemical or geotechnical) during program activities (except wellhead samples taken for measurement of field parameters). Use of the Chain-of-Custody Record Form creates an accurate written record that can be used to trace the possession and handling of the sample from the moment of its collection through analysis. This procedure identifies the necessary custody records and describes their completion. This procedure does not take precedence over region specific or site-specific requirements for chain-of-custody.

III Definitions

Chain-of-Custody Record Form - A Chain-of-Custody Record Form is a printed two-part form that accompanies a sample or group of samples as custody of the sample(s) is transferred from one custodian to another custodian. One copy of the form must be retained in the project file.

Custodian - The person responsible for the custody of samples at a particular time, until custody is transferred to another person (and so documented), who then becomes custodian. A sample is under one's custody if:

- It is in one's actual possession.
- It is in one's view, after being in one's physical possession.
- It was in one's physical possession and then he/she locked it up to prevent tampering.
- It is in a designated and identified secure area.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the point and time that it was collected.

IV Responsibilities

Project Manager - The Project Manager is responsible for ensuring that project-specific plans are in accordance with these procedures, where applicable, or that other, approved procedures are developed. The Project Manager is responsible for development of documentation of procedures which deviate from those presented herein. The Project Manager is responsible for ensuring that chain-of-custody procedures are implemented. The Project Manager also is responsible for determining that custody procedures have been met by the analytical laboratory.

Field Team Leader - The Field Team Leader is responsible for determining that chain-of-custody procedures are implemented up to and including release to the shipper or laboratory. It is the responsibility of the Field Team Leader to ensure that these procedures are implemented in the field and to ensure that personnel performing sampling activities have been briefed and trained to execute these procedures.

Sample Personnel - It is the responsibility of the field sampling personnel to initiate chain-of-custody procedures, and maintain custody of samples until they are relinquished to another custodian, the sample shipper, or to a common carrier.

V Procedures

The term "chain-of-custody" refers to procedures which ensure that evidence presented in a court of law is valid. The chain-of-custody procedures track the evidence from the time and place it is first obtained to the courtroom, as well as providing security for the evidence as it is moved and/or passed from the custody of one individual to another.

Chain-of-custody procedures, recordkeeping, and documentation are an important part of the management control of samples. Regulatory agencies must be able to provide the chain-of-possession and custody of any samples that are offered for evidence, or that form the basis of analytical test results introduced as evidence. Written procedures must be available and followed whenever evidence samples are collected, transferred, stored, analyzed, or destroyed.

V.1 Sample Identification

The method of identification of a sample depends on the type of measurement or analysis performed. When in-situ measurements are made, the data are recorded directly in bound logbooks or other field data records with identifying information.

Information which shall be recorded in the field logbook, when in-situ measurements or samples for laboratory analysis are collected, includes:

- Field Sampler(s);
- CTO Number;
- Project Sample Number;
- Sample location or sampling station number;
- Date and time of sample collection and/or measurement;

- Field observations;
- Equipment used to collect samples and measurements; and,
- Calibration data for equipment used.

Measurements and observations shall be recorded using waterproof ink.

V.1.1 Sample Label

Samples, other than in-situ measurements, are removed and transported from the sample location to a laboratory or other location for analysis. Before removal, however, a sample is often divided into portions, depending upon the analyses to be performed. Each portion is preserved in accordance with the Sampling and Analysis Plan. Each sample container is identified by a sample label (see Attachment A). Sample labels are provided, along with sample containers, by the analytical laboratory. The information recorded on the sample label includes:

- Project - Contract Task Order (CTO) Number.
- Station Location - The unique sample number identifying this sample.
- Date - A six-digit number indicating the day, month, and year of sample collection (e.g., 12/21/85).
- Time - A four-digit number indicating the 24-hour time of collection (for example: 0954 is 9:54 a.m., and 1629 is 4:29 p.m.).
- Medium - Water, soil, sediment, sludge, waste, etc.
- Sample Type - Grab or composite.
- Preservation - Type and quantity of preservation added.
- Analysis - VOA, BNAs, PCBs, pesticides, metals, cyanide, other.
- Sampled By - Printed name of the sampler.
- Remarks - Any pertinent additional information.

Using only the work assignment number of the sample label maintains the anonymity of sites. This may be necessary, even to the extent of preventing the laboratory performing the analysis from knowing the identify of the site (e.g., if the laboratory is part of an organization that has performed previous work on the site).

V.2 Chain-of-Custody Procedures

After collection, separation, identification, and preservation, the sample is maintained under chain-of-custody procedures until it is in the custody of the analytical laboratory and has been stored or disposed.

V.2.1 Field Custody Procedures

- Samples are collected as described in the site Sampling and Analysis Plan. Care must be taken to record precisely the sample location and to ensure that the sample number on the label matches the Chain-of-Custody Record exactly.

- The person undertaking the actual sampling in the field is responsible for the care and custody of the samples collected until they are properly transferred or dispatched.
- When photographs are taken of the sampling as part of the documentation procedure, the name of the photographer, date, time, site location, and site description are entered sequentially in the site logbook as photos are taken. Once developed, the photographic prints shall be serially numbered, corresponding to the logbook descriptions; photographs will be stored in the project files. It is good practice to identify sample locations in photographs by including an easily read sign with the appropriate sample/location number.
- Sample labels shall be completed for each sample, using waterproof ink unless prohibited by weather conditions, e.g., a logbook notation would explain that a pencil was used to fill out the sample label if the pen would not function in freezing weather.

V.2.2 Transfer of Custody and Shipment

Samples are accompanied by a Chain-of-Custody Record Form. A Chain-of-Custody Record Form example is shown in Attachment B. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the Record. This Record documents sample custody transfer from the sampler, often through another person, to the analyst in the laboratory. The Chain-of-Custody Record is filled out as given below.

- Enter header information (CTO number, samplers, and project name).
- Enter sample specific information (sample number, media, sample analysis required and analytical method grab or composite, number and type of sample containers, and date/time sample was collected).
- Sign, date, and enter the time under "Relinquished by" entry.
- Have the person receiving the sample sign the "Received by" entry. If shipping samples by a common carrier, print the carrier to be used in this space (i.e., Federal Express).
- If a carrier is used, enter the airbill number under "Remarks," in the bottom right corner;
- Place the original (top, signed copy) of the Chain-of-Custody Record Form in a plastic zipper-type bag or other appropriate sample shipping package. Retain the copy with field records.
- Sign and date the custody seal, a 1- by 3-inch white paper label with black lettering and an adhesive backing. Attachment C is an example of a custody seal. The custody seal is part of the chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field. Custody seals shall be provided by the analytical laboratory.

- Place the seal across the shipping container opening so that it would be broken if the container were to be opened.
- Complete other carrier-required shipping papers.

The custody record is completed using waterproof ink. Any corrections are made by drawing a line through and initialing and dating the change, then entering the correct information. Erasures are not permitted.

Common carriers will usually not accept responsibility for handling Chain-of-Custody Record Forms; this necessitates packing the record in the shipping container (enclosed with other documentation in a plastic zipper-type bag). As long as custody forms are sealed inside the shipping container and the custody seals are intact, commercial carriers are not required to sign the custody form.

The laboratory representative who accepts the incoming sample shipment signs and dates the Chain-of-Custody Record, completing the sample transfer process. It is then the laboratory's responsibility to maintain internal logbooks and custody records throughout sample preparation and analysis.

VI Quality Assurance Records

Once samples have been packaged and shipped, the Chain-of-Custody copy and airbill receipt become part of the quality assurance record.

VII Attachments

Sample Label
Chain of Custody Form
Custody Seal

VIII References

USEPA. *User's Guide to the Contract Laboratory Program*. Office of Emergency and Remedial Response, Washington, D.C. (EPA/540/P-91/002), January 1991.

Attachment A
Example Sample Label

CDM Federal Programs Corporation
13135 Lee Jackson Memorial Highway Fairfax, VA 22033

NAVY C.L.E.A.N. - ST. JULIEN'S CREEK ANNEX
SITE: 02 SAMPLE ID: SJS02-GW1S-002
DATE: _____ TIME: _____ SAMPLER: LC/TS
ANALYSIS: Low Con. VOA PRES.: HCl pH <2, Cool 4 C

CDM Federal Programs Corporation
13135 Lee Jackson Memorial Highway Fairfax, VA 22033

NAVY C.L.E.A.N. - ST. JULIEN'S CREEK ANNEX
SITE: 02 SAMPLE ID: SJS02-GW1S-002
DATE: _____ TIME: _____ SAMPLER: LC/TS
ANALYSIS: Low Con. VOA PRES.: HCl pH <2, Cool 4 C

CDM Federal Programs Corporation
13135 Lee Jackson Memorial Highway Fairfax, VA 22033

NAVY C.L.E.A.N. - ST. JULIEN'S CREEK ANNEX
SITE: 02 SAMPLE ID: SJS02-GW1S-002
DATE: _____ TIME: _____ SAMPLER: LC/TS
ANALYSIS: TCL SVOAs/Pest/PCBs PRES.: Cool 4 C

CDM Federal Programs Corporation
13135 Lee Jackson Memorial Highway Fairfax, VA 22033

NAVY C.L.E.A.N. - ST. JULIEN'S CREEK ANNEX
SITE: 02 SAMPLE ID: SJS02-GW1S-002
DATE: _____ TIME: _____ SAMPLER: LC/TS
ANALYSIS: Nitramines PRES.: Cool 4 C

CDM Federal Programs Corporation
13135 Lee Jackson Memorial Highway Fairfax, VA 22033

NAVY C.L.E.A.N. - ST. JULIEN'S CREEK ANNEX
SITE: 02 SAMPLE ID: SJS02-GW1S-002
DATE: _____ TIME: _____ SAMPLER: LC/TS
ANALYSIS: TAL Metals (unfiltered) PRES.: HNO₃ pH <2, Cool 4 C

CDM Federal Programs Corporation
13135 Lee Jackson Memorial Highway Fairfax, VA 22033

NAVY C.L.E.A.N. - ST. JULIEN'S CREEK ANNEX
SITE: 02 SAMPLE ID: SJS02-GW1S-002
DATE: _____ TIME: _____ SAMPLER: LC/TS
ANALYSIS: TAL Cyanide PRES.: NaOH pH >12, Cool 4 C

CDM Federal Programs Corporation
13135 Lee Jackson Memorial Highway Fairfax, VA 22033

NAVY C.L.E.A.N. - ST. JULIEN'S CREEK ANNEX
SITE: 02 SAMPLE ID: SJS02-GW1S-002
DATE: _____ TIME: _____ SAMPLER: LC/TS
ANALYSIS: Low Con. VOA PRES.: HCl pH <2, Cool 4 C

CDM Federal Programs Corporation
13135 Lee Jackson Memorial Highway Fairfax, VA 22033

NAVY C.L.E.A.N. - ST. JULIEN'S CREEK ANNEX
SITE: 02 SAMPLE ID: SJS02-GW1S-002
DATE: _____ TIME: _____ SAMPLER: LC/TS
ANALYSIS: TCL SVOAs/Pest/PCBs PRES.: Cool 4 C

CDM Federal Programs Corporation
13135 Lee Jackson Memorial Highway Fairfax, VA 22033

NAVY C.L.E.A.N. - ST. JULIEN'S CREEK ANNEX
SITE: 02 SAMPLE ID: SJS02-GW1S-002
DATE: _____ TIME: _____ SAMPLER: LC/TS
ANALYSIS: TCL SVOAs/Pest/PCBs PRES.: Cool 4 C

CDM Federal Programs Corporation
13135 Lee Jackson Memorial Highway Fairfax, VA 22033

NAVY C.L.E.A.N. - ST. JULIEN'S CREEK ANNEX
SITE: 02 SAMPLE ID: SJS02-GW1S-002
DATE: _____ TIME: _____ SAMPLER: LC/TS
ANALYSIS: Nitramines PRES.: Cool 4 C

CDM Federal Programs Corporation
13135 Lee Jackson Memorial Highway Fairfax, VA 22033

NAVY C.L.E.A.N. - ST. JULIEN'S CREEK ANNEX
SITE: 02 SAMPLE ID: SJS02-GW1S-002F
DATE: _____ TIME: _____ SAMPLER: LC/TS
ANALYSIS: TAL Metals (filtered) PRES.: HNO₃ pH <2, Cool 4 C

CDM Federal Programs Corporation
13135 Lee Jackson Memorial Highway Fairfax, VA 22033

NAVY C.L.E.A.N. - ST. JULIEN'S CREEK ANNEX
SITE: 02 SAMPLE ID: SJS02-GW1S-002
DATE: _____ TIME: _____ SAMPLER: LC/TS
ANALYSIS: Total Phosphorous PRES.: H₂SO₄ pH _____, Cool 4 C

Attachment B
Example Chain-of-Custody Record

Attachment C
Example Custody Seal

ENVIRONMENTAL SAMPLING SUPPLY

SAMPLE # _____ DATE _____

CUSTOMER _____

SIGNATURE _____

9501 San Leandro Street, Oakland, California 94603
(510) 562-4988 (800) 233-8425

SAMPLE LABEL

CUSTODY SEAL

Person Collecting Sample _____ (signature) _____ Sample No. _____

Date Collected _____ Time Collected _____

SAMPLE CUSTODY SEAL

Field Measurement of pH and Eh

I. Purpose and Scope

The purpose of this procedure is to provide a guideline for field measurement of pH and Eh.

II. Equipment and Materials

- pH buffer solution for pH 4, 7, and 10
- Deionized water in squirt bottle
- pH/Eh meter, calibration sheet, and instructions
- pH and redox electrodes
- Beakers
- Glassware that has been washed with soap and water, rinsed twice with hot water, and rinsed twice with deionized water
- 4 M KCl saturated with Ag/AgCl solution, electrode filling solution.

III. Procedures and Guidelines

A. Calibration

Calibrate unit prior to initial daily use. There are no calibration procedures for the redox electrode. Calibrate with at least two solutions. Clean the probe according to the manufacturer's recommendations. Duplicate samples should be run once every 10 samples. The order of calibration solutions will be based on the instrument manufacturer's recommendation.

1. Place electrode in pH 7 buffer solution.
2. Allow meter to stabilize, and then turn calibration dial until a reading of 7.0 is obtained.
3. Rinse electrode with deionized water and place it in a pH 4 or pH 10 buffer solution.
4. Allow meter to stabilize again and then turn slope adjustment dial until a reading of 4.0 is obtained for the pH 4 buffer solution or 10.0 for the pH 10 buffer solution.

5. Rinse electrode with deionized water and place in pH 7 buffer. If meter reading is not 7.0, repeat sequence.

B. Procedure

1. Before going out into the field:
 - a. Check batteries.
 - b. Do a quick calibration at pH 7 and 4 to check electrode.
 - c. Obtain fresh calibration solutions.
 - d. Fill electrodes.
2. Calibrate meter using calibration procedure.
3. Pour the sample into a clean beaker.
4. Rinse electrode with deionized water between samples.
5. Immerse electrode in solution. Make sure the white KCl junction on the side of the electrode is in the solution. The level of electrode solution should be one inch above sample to be measured.
6. Recheck calibration with pH 7 buffer solution after every five samples.

C. General

1. When calibrating the meter, use pH buffers 4 and 7 for samples with pH <8, and buffers 7 and 10 for samples with pH >8. If meter will not read pH 4 or 10, something may be wrong with the electrode.
2. Measurement of pH is temperature dependent. Therefore, buffers temperatures should be within about 2 degrees C of sample temperatures. For refrigerated or cool samples, use refrigerated buffers to calibrate the pH meter.
3. Weak organic and inorganic salts and oil and grease interfere with pH measurements. If oil and grease are visible, note it on the data sheet. Clean electrode with soap and water and rinse with distilled water. Then recalibrate meter.
4. Following field measurements, report problems and compare with previous data. Clean dirt off meter and inside case and store electrode in pH 4 buffer.
5. Accuracy and precision are dependent on the instrument used; refer to manufacturer's manual. Expected accuracy and precision are +/- 0.1 pH unit.
6. The redox electrode should be checked prior to beginning site work and when anomalous readings suggest that the probe is malfunctioning. The procedure for checking the redox electrode is as below:

- a. Prepare solution A (0.1 M potassium ferrocyanide and 0.005 M potassium ferricyanide): weigh out 4.22 g reagent-grade $K_4Fe(CN)_6 \cdot 3H_2O$ and 1.65 g reagent-grade $K_3Fe(CN)_6$. Place in a 100 ml volumetric flask. Add about 50 ml distilled water and swirl to dissolve solids. Dilute to volume with distilled water.
- b. Prepare solution B (0.01 M potassium ferrocyanide, 0.05 M potassium ferricyanide, and 0.36 M potassium fluoride): Weigh out 0.42 g reagent-grade $K_4Fe(CN)_6 \cdot 3H_2O$ 1.65 g reagent-grade $K_3Fe(CN)_6$, and 3.39 g reagent-grade $KF \cdot 2H_2O$. Place in a 100 ml volumetric flask. Add 50 ml distilled water, and swirl to dissolve solids. Dilute to volume with distilled water.
- c. Transfer solution A to a 150 ml beaker. Place electrode in the solution and wait until the reading stabilizes. The potential should be about 234 mV.
- d. Rinse electrode and repeat the measurement with solution B. The potential should be about 66 mV greater in solution B than in solution A.

IV. Key Checks and Preventive Maintenance

- Check batteries, have a replacement set on hand.
- Calibrate meter.
- Refer to operation manual for recommended maintenance.

Preserving Non-VOC Aqueous Samples

I. Purpose

To provide general guidelines for preserving aqueous samples.

II. Scope

Standard aqueous sample preservation procedures for non-VOC samples are provided.

III. Equipment and Materials

- Disposable eye droppers
- Clean beakers for transfer of small portions of chemical preservative
- pH paper strips (Range 0 to 14)
- Chemical preservatives, as appropriate
- Personal protection, as appropriate
- Clean out door or vented indoor area

IV. Procedures and Guidelines

1. Remove caps from sample containers to be chemically preserved in designated area. Add appropriate amount of chemical preservative to opened container. To determine the approximate amount of preservative which will be required, preserve a sample of potable water and calculate the volume of preservative required.
2. After adding the appropriate preservatives to the sample containers, cap containers tightly. Invert sample container a few times to mix.
3. After preserving all the sample containers and mixing, open the container and check the pH of the sample by pouring out a small quantity of the sample to a clean receptacle and dipping a pH indicating strip into the sample. Add more preservative to the sample to adjust the pH, if necessary repeating steps 1 and 2. When three times the amount of preservative used to preserve a sample of potable water has been added, record the pH and notify the sample manager that the sample could not be preserved.
4. Wrap, package, and ice samples according to the SOP Packaging and Shipping Procedures.

V. Attachments

None.

VI. Key Check Items

Surface Water Sampling

I. Purpose and Scope

This procedure presents the techniques used in collecting surface water samples. Materials, equipment, and procedures may vary; refer to the Field Sampling Plan and operators manuals for specific details.

II. Materials and Equipment

Materials and equipment vary depending on type of sampling; the Field Sampling Plan should be consulted for project-specific details.

- Open tube sampler
- Dip sampler
- Weighted bottle sampler
- Hand pump
- Kemmerer or Van Dorn sampler
- Depth-integrating sampler
- Sample containers
- Meters for specific conductance, temperature, pH, and dissolved oxygen

III. Procedures and Guidelines

Before surface water samples are taken, all sampler assemblies and sample containers are cleaned and decontaminated as described in SOP Decontamination of Personnel and Equipment. Methods for surface water sample collection are described below.

A. Manual Sampling

Surface water samples are collected manually by submerging a clean glass, stainless steel, or Teflon container into the water body. Samples may be collected at depth with a covered bottle that can be removed with a tripline. The most common sampler types are beakers, sealable bottles and jars, pond samplers, and weighted bottle samplers. Pond samplers have a fixed or telescoping pole attached to the sample container. Weighted bottle samplers are lowered below water surface, where the attached bottle is opened, allowed to fill, and pulled out of the water. When retrieved, the bottle is tightly capped and removed from the sampler assembly. Specific types of weighted bottle samplers include dissolved oxygen, Kemmerer, or Van Dorn, and are acceptable in most instances.

A sample is taken with the following specific steps:

1. The location and desired depth for water sampling are selected.
2. The sample site is approached from downstream in a manner that avoids disturbance of bottom sediments as much as possible. The sample bottle is gently submerged with the mouth pointed upstream and the bottle tilted slightly downstream. Bubbles and floating materials should be prevented from entering the bottle.
3. For weighted bottle samplers, the assembly is slowly lowered to the desired depth. The bottle stopper is unseated with a sharp tug and the bottle is allowed to fill until bubbles stop rising to the surface.
4. When the bottle is full, it is gently removed from the water. If sample transfer is required, it should be performed at this time.
5. Measure dissolved oxygen, specific conductance, temperature, and pH at the sampling location.

STANDARD OPERATING PROCEDURE

VOC Sampling--Water

I. Purpose

To provide general guidelines for sampling aqueous volatile organic compounds.

II. SCOPE

Standard techniques for collecting representative samples are summarized. Site specific details are discussed in the FSP.

III. EQUIPMENT AND MATERIALS

- Sample vials, clean latex or surgical gloves, pH meter
- Hydrochloric acid (HCl) for preservation
- pH meter or pH indicating paper
- Surgical or latex gloves

IV. PROCEDURES AND GUIDELINES

1. Sample VOCs before sampling other analyte groups.
2. When sampling for VOCs, especially residential wells, evaluate the area around the sampling point for possible sources of air contamination by VOCs. Products that may give off VOCs and possibly contaminate a sample include perfumes and cosmetics, skin applied pharmaceuticals, automotive products (gasoline, starting fluid, windshield deicers, carburetor cleaners, etc.) and household paint products (paint strippers, thinners, turpentine, etc.).
3. To check the amount of hydrochloric acid (HCl) that needs to be added at each location, fill a test vial (40 ml) with the water to be sampled, add one drop of hydrochloric acid (HCl), gently mix, and check the pH. Repeat this cycle (if necessary) until you reach a pH of 2, counting the number of drops of HCl required. DISCARD THE TEST VIAL and add an equal number of drops of HCl to each of the sample vials. proceed to sample.
4. Keep the caps off the sample vials for as short a time as possible.

5. Wear clean latex or surgical gloves.
6. Fill the sample vial immediately, allowing the water stream to strike the inner wall of the vial to minimize formation of air bubbles. **DO NOT RINSE THE SAMPLE VIALS BEFORE FILLING.**
7. Fill the sample vial with a minimum of turbulence, until the water forms a positive meniscus at the brim.
8. Replace the cap by gently setting it on the water meniscus. Tighten firmly, but **DO NOT OVERTIGHTEN.**
9. Invert the vial and tap it lightly. If you see air bubbles in the sample, do not add more sample. Use another vial to collect another sample. Repeat if necessary until you obtain a proper sample.

V. ATTACHMENTS

None.

VI. KEY CHECKS AND ITEMS

- Check for possible sources of contamination.
- Check pH.
- Fill slowly, with as little turbulence as possible.
- Check for air bubbles.

VOC_AQ01.DOC

Appendix B
Bioassay SOPs

ENVIROSYSTEMS, INCORPORATED
STANDARD OPERATION PROCEDURE

SOP Number: QA-1325
Revision Number: 1
Page: 1 of 14

TITLE: Ammonia by Lachat

Approved By:

QA Officer: _____ Date: _____

President: _____ Date: _____

Revision History:

Revision Number	Changes	Authorization	Date
1	Review and Update	Sue Dionne	5/00

TITLE: Ammonia by Lachat

1.0 Purpose and Applicability

This Standard Operation Procedure describes the determination of ammonia in wastewater and groundwater samples using the Lachat analysis system. It is based on the Berthelot Reaction.

2.0 Definitions - NA

3.0 Applicable Documents/References

Standard Methods for the Examination of Water and Wastewater. 1995. 19th Edition. Nitrogen (Ammonia) 4500 NH₃ G, Automated Phenolate Method.

ESI SOP 1328. Ammonia Distillation.

Quik Chem Automated Ion Analyzer, Methods Manual. Lachat Instruments 6645 West Mill Road, Milwaukee, WI 53218.

Quik Chem Automated Ion Analyzer, Training Manual. Lachat Instruments 6645 West Mill Road, Milwaukee, WI 53218.

Quik Chem Automated Ion Analyzer, Hardware Installation and System Operation Manual. Lachat Instruments 6645 West Mill Road, Milwaukee, WI 53218.

4.0 Materials and Apparatus

4.1 Lachat Instruments

RAS Autosampler
Reagent Pump
Quik Chem 8000 System Unit
Computer

Printer
Heating Unit
630nm Filter

TITLE: Ammonia by Lachat

Ammonia Manifold

4.2 Reagents

Milli-Q[®] water
DI rinse water
Concentrated Sulfuric Acid
Sodium Phenolate
Sodium Hypochlorite
 Sodium EDTA Buffer
 Sodium Nitroprusside
Stock Standard
Seven Working Standards
 Check Standard

4.3 Parts Equipment List

Pump tubing - Green - Green
 Orange - Orange
 Grey - Grey
 Black - Black
 Red - Red
Culture Tubes -15 mL and 12.75 mm
Lachat Repair and Extra Parts Kit
Micro Loop
Waste Container
Ruler (measuring in cm)
Cutting Utensil
Stir Bar
Drying oven set at 110EC " 1EC

5.0 Methods/Procedures

5.1 Reagents

Use Milli-Q[®] water for the preparation of all solutions. To prevent gas bubbles, Milli-Q[®] water may be autoclaved for 20 minutes at slow vent speed.

TITLE: Ammonia by Lachat

Store all reagents in the dark at 4°C; expiration of one month after mixing.

5.1.1. Sodium Phenolate

Caution: Read MSDS. Phenols can cause severe burns and are rapidly absorbed into the body through the skin. Will melt many types of plastic.

In a pre-weighed 1L volumetric flask, add approximately 800mL of Milli-Q[®] water. Add **83g crystalline phenol (C₆H₅OH)**. While stirring, slowly add **32g sodium hydroxide (NaOH)**. Cool, dilute to volume, invert to mix thoroughly. Do not degas.

5.1.2. Sodium Hypochlorite

In a 500ml volumetric flask, place **250mL Regular Clorox Bleach** [5.25% sodium hypochlorite (NaOCl), The Clorox Company, Oakland, CA] and dilute to volume with Milli-Q[®] water. Invert to mix.

5.1.3. Buffer

In a pre-weighed 2L volumetric flask, place approximately 900mL of Milli-Q[®] water. Add **100g disodium ethylenediamine tetraacetate (Na₂EDTA)** and **11.0g sodium hydroxide (NaOH)**. Add a stir bar, mix well until all solids are dissolved, cool and dilute to volume with Milli-Q[®] water. Invert to mix.

5.1.4. Sodium Nitroprusside

In a pre-weighed 1L volumetric flask, dissolve **3.50g sodium nitroprusside (Sodium Nitroferricyanide [Na₂Fe(CN)₅NOC₂H₂O])** and dilute to volume with Milli-Q[®] water.

5.1.5. Standard 1- Stock Standard 10000.0mg N/L as NH₃

In a 1L volumetric flask dissolve **38.19g ammonium chloride**

TITLE: Ammonia by Lachat

(NH₄CL), (previously dried for two hours in a drying oven set to 110°C ± 1°C) in approximately 800 mL Milli-Q[®] water. Add 1mL **sulfuric acid**. Dilute to volume with Milli-Q[®] and invert to mix. Store in the dark at 4°C. If no sulfuric acid is used, the standard expires in 48 hours.

5.1.6. Standard 2 - **Intermediate Stock Standard 200.0mg N/L as NH₃**

In a 1L volumetric Flask, add **20.0mL Stock Standard** (Standard 1). Add 1mL **sulfuric acid**, and dilute to volume with Milli-Q[®] water and invert to mix. Store in the dark at 4°C. If no sulfuric acid is used, the standard expires in 48 hours.

5.1.7 Seven Working Standards:

To seven 250mL volumetric flasks add, respectively, exactly the amounts shown under the final concentration of the **Intermediate Stock Standard** (Standard 2) in approximately 200 mL Milli-Q[®] water. Add 0.5 mL of concentrated **sulfuric acid**, dilute to volume and invert to mix. Store in the dark at 4°C. If no sulfuric acid is used, the standard expires in 48 hours.

Final Concentration (MgN/L)	20.0	8.00	2.00	1.00	0.400	0.200	0.100
Standard 2 to add (mL)	25.0	10.0	2.50	1.250	0.500	0.250	0.125

5.1.8. Check Standard

Use the extra sample from ERA WP test standards with known quantities and limits.

5.2 Apparatus Set Up

5.2.1 Timing

TITLE: Ammonia by Lachat

System IV GAIN: 175* 1. AE instrument, top scale response = 1.0 abs.

Sample throughput:	90 samples/h; 40 s/sample
Pump Speed:	35
Cycle Period:	40s
Inject to Start of peak period:	25s
Inject to End of peak period:	63s
Warm up time:	15 - 30 min.

5.2.2 The Ammonia Manifold - Please see the Hardware Installation and System Operation Manual for complete details regarding operations of the various apparatus. Replace any lines that have become flattened or worn from the pump.

5.2.2.1. The **Sample Line** is pumped from the **autosampler needle** to **port 6** on the injection valve using a **Green - Green** line with its ends cut to exactly 2cm.

5.2.2.2. The **Micro Loop** connects **ports 1** and **4** on the injection valve.

5.2.2.3. The **Carrier Line** is pumped from the **rinse water** container to **port 2** on the injection valve using a **Grey - Grey** line.

5.2.2.4. The **Manifold Line** connects from **port 3** on the injection valve to the tee fitting labeled **from valve** on the left side.

5.2.2.5. The **Waste Line** is connected to **port 5** on the injection valve and to the **waste container** under the counter using a **Green - Green** line.

5.2.2.6. The **Sodium EDTA Buffer Line** is connected from the **Buffer** container to the tee fitting labeled **Buffer** on the right side using a **Red - Red** line.

5.2.2.7. The **Sodium Phenolate** line is connected from the

TITLE: Ammonia by Lachat

Phenolate container to the tee fitting labeled ⇒**Phenolate** on the left side using an **Orange - Orange** line.

5.2.2.8. The **Sodium Hypochlorite** line is connected from the **Hypochlorite** container to the tee fitting labeled ⇒**Hypochlorite** on the left side using a **Black - Black** line.

5.2.2.9. The **Sodium Nitroprusside** line is connected from the **Nitroprusside** container to the tee fitting labeled ⇒**Nitroprusside** on the left side using an **Orange - Orange** line.

5.2.2.10. The **Heater In** line comes from the left of the heater and up through the **hole** in the manifold board connecting to the top of the tee fitting labeled ⇒**Nitroprusside**.

5.2.2.11. The **Heater Out** line comes from the right of the heater (after use it becomes slightly brown in color) and up through the **hole** in the manifold board connecting to the left of the fitting labeled **to flow cell** ⇒ **630nm**.

5.2.2.12. The fitting labeled **to flow cell** ⇒ **630nm** connects to the **flow cell** line to the right of it.

5.2.2.13. Turn the power on, the sampling needle should rise to its top position and the temperature reading will turn on with an indicator light for the flow cell. Turn on the pump and check that all is working properly. The pump speed is set to 35 for this procedure.

5.2.2.14. Place all of the reagent lines into the rinse water container and allow water to flow through the entire system.

5.2.2.15. To set the Temperature, locate the heater control on the right side of the Quick Chem 8000. Press the **Arrow in an Oval** key until "**SP for Set Point**" shows. Press the **Big Arrow** keys to raise or lower the set point to 60°C. Once the

TITLE: Ammonia by Lachat

temperature has been set, press the **Enter** key to save the set point. Press the **Arrow in an Oval** key to display the current temperature.

Caution! The pump must remain on and have water flowing through it, as the reagent line will melt if empty or allowed to sit in standing water.

5.2.2.16. Place the 630 nm Filter right-side up into the flow cell. It is stored in a desiccator and can be put into place just before starting the run. It should be placed back into the desiccator immediately after use.

5.3 System Start Up - See the manual for complete details.

5.3.1. Turn the computer and the Quick Chem unit on. Log into the Omnion FIA program. Enter the program using analyst's name and password.

5.3.2. Click on "Flow Injection Analysis" and select the Ammonia Method.

5.3.3. Click on "Tray" and select the tray to be used or modified. Enter all sample data required into the tray spreadsheet.

5.3.4. Click on "DQM" and select "Ammonia." The DQM will require updating each time a change is made in the check standard. See the Hardware Installation and System Operation Manual if the "DQM" requires updating.

5.3.5. Go to "File" and "Save Tray."

5.4 Sampling

Samples are preserved with sulfuric acid and tested within 28 days. Unpreserved samples must be run within 24 hours.

The Federal Register entry which defines standard EPA NPDES and

TITLE: Ammonia by Lachat

NIPDWR methods states that “Manual distillation is NOT required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.” Certain samples may be required to be distilled, speak to the lab manager if there are any questions as to which samples will be distilled. See ESI SOP 1328 for the distillation procedure.

5.4.1. Standards -The Ammonia procedure uses S1 - S9 with 15 mL culture tubes. Pour each standard into its appropriate culture tube and place it into the autosampler rack as labeled in the tray. S9, containing check standard, is listed in the DQM.

5.4.2. Samples - Mix each sample and pour it into a 12x75 mm culture tube, placing it into the autosampler rack as it is listed in the tray on the computer.

5.4.3 To start a run, click on “Run Tray.”

5.4.3.1. Prompt “Method.” Enter “Ammonia”.

5.4.3.2. Prompt “Tray.” Enter the tray to be run.

5.4.3.3. Prompt “Data File” .

5.4.3.4. Enter any comments that may be useful.

5.4.3.5. Click on the “Run” box. If the data file already exists it will state twice “This data file and/or its associated runtime report already exist. Choose ‘OK’ to overwrite or ‘Cancel’ to enter another data file name.”

The autosampler will move the rack to the appropriate location and run the samples.

5.4.4. Dilute any samples with results that are above 20 mgN/L. Add them to the tray with the dilution adjusted in the column labeled “Manual Dilution”, and save the tray. **Note:** If the tray is not re-

TITLE: Ammonia by Lachat

saved before the added sample is reached, the computer will not run the new sample. The computer will beep when the run is finished.

5.5 Shut-Down Procedure

- 5.5.1. When the tray is finished and the Quality Control elements are verified as within limits, the lines may be removed from the reagents and placed in rinse water.
- 5.5.2. Remove the filter and place it into the desiccator.
- 5.5.3. Re-set the Temperature to room. Press the **Arrow in an Oval** key until "**SP for Set Point**" shows. Press the **Big Arrow** keys to lower the set point to approximately 20°C. Once the temperature has been set, press the **Enter** key to save the set point. Press the **Arrow in an Oval** key to display the current temperature.
- 5.5.4. When the temperature has reached below 30°C the lines may be removed from the rinse water and dried by allowing the pump to run until there is no water left in the reagent lines.
- 5.5.5. Exit the program and log off.

6.0 Quality Control Requirements

- 6.1 Standards Curve - The standards required for the ammonia curve are: 20 mg N/L, 8.0 mg N/L, 2.0 mg N/L, 1.0 mg N/L, 0.4 mg N/L, 0.2 mg N/L, 0.1 mg N/L, and 0.0 mg N/L.
- 6.2 Other required Standards - Blanks and duplicates shall be run at 10% of total samples. Check standards will be run every ten (10) injections.
- 6.3 If standards are found to be out of limits in the initial run, the "Peak Base Width" and the "Threshold" may need adjusting.
 - 6.3.1 Go to "Method" in the top bar and to "Graphical Events

TITLE: Ammonia by Lachat

Programming.”

6.3.2. To change the “Peak Base Width,” click on it and the screen will show the graph. Choose the best fit on the largest peak available. Click once at the beginning of the peak and once at the end of the peak.

6.3.3. To change the “Threshold,” click on it and the screen will show the graph. Choose the best fit on the flattest blank standard available. Click once at the beginning of the peak and once at the end of the peak.

6.3.4 If repeated peak corrections do not bring the standards in line, see section 8.1 and run the curve again.

6.4 Continuing calibration check standards will be evaluated to determine acceptability of the data. To achieve this, a known standard will be analyzed with each batch of 20 samples. Results of the calibration data will be maintained with the raw data set.

6.5 Precision will be assessed through duplicate analysis. One sample in 20 will be evaluated in duplicate to determine the relative percent difference between the two analyses. Relative percent difference values will be compared to historic data sets to determine acceptability.

7.0 Calculations/Reporting

7.1 Custom Report - Please see the Hardware Installation and System Operation Manual for complete information.

7.1.1. If a run has just finished, the data file it is stored in is already open. If not, go to the “Data” button at the top of the screen and open the file to be reported.

7.1.2. Go to the button labeled “Custom” at the top of the screen.

7.1.3. Check that the report has been updated to reflect current standards

TITLE: Ammonia by Lachat

data by double clicking in each box on each page. Make any necessary changes. Each time the standards are renewed the "Header" box must have the new "Date Made" information entered.

7.1.4. Preview each page to adjust the data location.

7.1.4.1 If there are overflow messages on page one in the "Multi-Channel Table" box, close the preview. Change the Range box which lists the number of cups that will be printed on that page. Enter the number of the last cup readable from the preview. Change the "Multi-Channel Table" box on page two to start with the next cup and end with the last cup run.

7.1.4.2 On page four the "Graph Chanel 1" box shows the portion of the graph which has the curve for the run included in the preview. If the standardization curve is not shown, exit the preview and double click on the "Graph Chanel 1" box. Change the "X Range (sec):" and "Y Range (Fv-s):" to the required ranges for the correct portion of the curve to show.

7.1.5. Print the report by clicking on the printer icon.

7.2. Export Data

7.2.1. Go to "File" and "Export Data."

7.2.2. Turn on all required columns for the final report.

7.2.3. Chose the "Ammonia" data folder and name the file.

7.2.4. Open "Corel Quattro Pro 8."

7.2.5. Go to "File" and "Open." In the "File Type" find the "ASCII Text [".txt"]" type file and open the file to be modified. It will be found in C:\omnion\data\ammonia\ "File name."

7.2.6. Go to "Tools", "Data Tools", "Quick Columns" and "Parse." The source must be "File" and the file name.

TITLE: Ammonia by Lachat

7.2.7. Sort the data and record any required notes about the run. If any dilutions have been run, report the dilution and calculate the final result. Include the file name, date and initials at the top of the report.

8.0 Corrective Actions

8.1 Precision errors - If baseline drifts, peaks are too wide, or other problems with precision arise, clean the manifold by the following procedure:

8.1.1. Place all reagent lines in deionized water and pump to clear reagents (2 - 5 minutes).

8.1.2. Place reagent lines and carrier in 1 M hydrochloric acid (one part concentrated HCL added to 11 parts of deionized water) and pump for several minutes.

8.1.3. Rinse all lines in deionized water until thoroughly cleaned.

8.1.4. Resume pumping reagents.

8.2. If samples are colored or are suspected to show a background absorbance, subtract the absorbance from the result by:

8.2.1. Calibrate the system in the normal manner.

8.2.2. Disable the check standard or DQM features and analyze the samples.

8.2.3. Place reagent and carrier lines in DI water and allow the baseline to stabilize.

8.2.4. Inject samples again without recalibrating.

8.2.5. Subtract the "background" concentration from the original concentration to give the corrected concentration. Corrected

TITLE: Ammonia by Lachat

Concentration=Original Concentration - Background Concentration

- 8.3. Calcium and magnesium ions may precipitate if present in sufficient concentrations. Tartrate or EDTA is added to the sample in-line in order to prevent this problem.
- 8.4.1 If the base line is sloping, curved or shows interference, see the troubleshooting portion of the Hardware Installation and System Operation Manual.

9.0 Health and Safety

Samples that have been preserved with sulfuric acid should be handled with gloves, safety glasses and proper shoes. A well-ventilated work area is recommended. As with all samples, gloves and safety glasses should be worn when handling undiluted effluents. Read all appropriate MSDS reports. Waste containing Phenols must be treated as hazardous.

10.0 Responsibilities

It is the lab manager's responsibility to insure technicians performing this procedure are properly trained and the training is documented in the technician's training file. Specialized Lachat training is required for this procedure. The technician is responsible for following the procedures outlined in this SOP.

ENVIROSYSTEMS, INCORPORATED
STANDARD OPERATION PROCEDURE

SOP Number: QA-1339
Date Issued: 06/01
Revision Number: 0
Page: 1 of 5

TITLE: Collection of Sediment Pore Water Samples

Approved By:

QA Officer: _____ Date: _____

President: _____ Date: _____

Revision History:

Revision Number	Changes	Revised By	Date

TITLE: Collection of Sediment Pore Water Samples

1.0 Purpose and Applicability

Conduct of various sediment assays may require collection of pore water samples to document water chemistry. Pore water samples may be collected using two (2) basic techniques, a destructive sampling protocol which generates the required sample but renders the sediment unusable for subsequent testing and a non-destructive protocol in which the sample is collected using techniques that result in minimal disturbance to the sediment. The latter technique may be used during the conduct of an assay.

2.0 Definitions

Pore Water- water located in the spaces between grains of sediment/soil.

3.0 Applicable Documents/References

NA

4.0 Materials and Apparatus

Centrifuge
Centrifuge bottles
Aquarium air stones or fritted glass
Plastic air line
Syringes, various volumes, or appropriate device for creating vacuum

5.0 Methods/Procedures

5.1 Destructive Sampling Technique

The destructive sampling technique involves collection of pore water from a sediment sample using centrifugation. A representative sample is placed in a

TITLE: Collection of Sediment Pore Water Samples

centrifuge bottle and centrifuged to separate the pore water from the sediment. Use of this technique to collect pore water samples during an assay requires inclusion of a sufficient number of surrogate test vessels in the study design to accommodate the anticipated number of pore water collections.

5.1.1 Sample Collection - Whole Sample

Samples received at the laboratory will be inspected to determine that the sample has not been artificially compacted. If the observations of the sample in the sample container indicate significant water above the sample surface, the client will be notified and the condition of the sample reviewed to determine if the overlying water is pore water forced out of the sample during transport or if it was surface water. If there is no evidence of excessive surface water, a representative sample will be collected. The sample will be transferred to a centrifuge bottle and placed in a centrifuge. NOTE - review centrifuge protocol regarding sample volumes, speeds and balancing. The sample will be centrifuged until the supernatant is clear. The supernatant is decanted into a clean bottle, preserved as required, for subsequent analysis.

5.1.2 Sample Collection - During Assay

Pore water samples collected during an assay using the destructive methodology are obtained from surrogate test vessels. Overlying water in the test vessel is carefully decanted, minimizing resuspension of the sediment. After removal of the overlying water, the remaining sediment is transferred to a centrifuge bottle and then placed in a centrifuge. The sample is centrifuged and the supernatant, expelled pore water, is transferred to an appropriate bottle, preserved as required, for subsequent analysis.

Note, the volume of pore water that will be extracted from a sample is dependant upon the amount of pore water in the sample, sample grain size distribution, length of time for centrifuging and centrifuge speed.

TITLE: Collection of Sediment Pore Water Samples

5.2 Non-Destructive Sample Collection

A non-destructive sample collection technique may be used to collect pore water samples in cases where sample volume is limited, in field applications where a centrifuge is not available or during an assay.

5.2.1 Sample Collection - During Assay

At the start of the assay, during sample addition, an air stone or other fritted glass element is placed in the test chamber. An extraction line, plastic or Teflon®, is attached to the air stone or fritted element. The length of the line should be sufficient so that the end is above the top of the test chamber. Test sediment is added to the test chamber. Insure that the sediment completely covers the air stone. When required, a syringe is attached to the end of the sampling line and an appropriate volume of pore water is extracted from the test sediments by applying a vacuum sampling line. The syringe may be replaced with a small vacuum pump. The sample is then transferred to a clean bottle for subsequent analysis. If there is evidence of sediment in the sample, transfer the sample to a centrifuge bottle and centrifuge to remove solid material, or if appropriate for the analytes, the particulate material may be removed from the sample by filtration.

5.2.2 Sample Collection - Field Event

Pore water samples may be collected during field activities using air stones and a syringe or vacuum pump. An air stone is attached to a sample collection line and inserted into the sediment being sampled. After insertion to the appropriate depth, a sample may be collected by drawing a vacuum using either the syringe or vacuum pump. The sample is then transferred to a clean bottle for subsequent analysis. If there is evidence of sediment in the sample, transfer the sample to a centrifuge bottle and centrifuge to remove solid material, or if appropriate for the analytes, the particulate material may be removed from the sample by filtration.

Note - the size the air stone or fritted element used in the sample collection process will, in part, determine sample collection rates.

TITLE: Collection of Sediment Pore Water Samples

6.0 Quality Control Requirements

NA

7.0 Calculations/Reporting

Provide specifics related to methodologies used for sample collection plus information on centrifuge time and speeds. If filtration was used, provide specifics related to filter type and pore size.

8.0 Corrective Actions

NA

9.0 Health and Safety

- 9.1 As with all samples, gloves and safety glasses should be worn when handling sediment samples and chemicals.
- 9.2 At the end of the process excess sample material and material used in the collection process will be disposed of appropriately.
- 9.3 Sample disposal will be conducted using procedures to minimize potential pollution of surface and ground waters plus soil and sediments.

10.0 Responsibilities

It is the lab managers responsibility to insure technicians performing this procedure are properly trained and the training is documented in the technician's training file. The technician is responsible for following the procedures outlined in this SOP.

ENVIROSYSTEMS, INCORPORATED
STANDARD OPERATION PROCEDURE

SOP Number: QA-1341
Date Issued: 03/02
Revision Number: 0
Page: 1 of 5

TITLE: Sulfide Analysis by Titration

Approved By:

QA Officer: _____ Date: _____

President: _____ Date: _____

Revision History:

Revision Number	Changes	Revised By	Date
0	Preparation of SOP	S. Dionne	03/02

TITLE: Sulfide Analysis by Titration

1.0 Purpose and Applicability

Sulfide often is present in groundwater, especially in hot springs. Its common presence in wastewaters comes partly from the decomposition of organic matter, sometimes from industrial wastes, but mostly from the bacterial reduction of sulfate. Hydrogen sulfide escaping into the air from sulfide-containing wastewater causes odor nuisances. Dissolved H₂S is toxic to fish and other aquatic organisms.

This SOP uses a back-titration method to determine sulfide levels in aqueous samples.

2.0 Definitions - NA

3.0 Applicable Documents/References

APHA. *Standard Methods for the Examination of Water and Wastewater*. 20th Edition. 1998. 4500-S²⁻ F.

4.0 Materials and Apparatus

Hydrochloric Acid, HCl, 6N
Standard Iodine solution, 0.025N (KI, iodine)
Standard sodium thiosulfate solution, 0.025N (Na₂S₂O₃ · 5H₂O, NaOH -6N or solid)
Standard Bi-iodate solution (KH(IO₃)₂)
Starch solution (soluble starch, salicylic acid)
Sulfide standard (1.00 mg S²⁻ /1.00 mL) - buy prepared
 Intermediate Standard : Dilute 10 mL stock to 1 L water. Prepare fresh daily.
 Standardize 1mL ≈ 0.01 mg S²⁻
 Working standards: Dilute 50 mL intermediate to 500 mL with 0.01N NaOH.
 Prepare fresh daily. 1.00 mL ≈ 0.001 mg S²⁻
1000 mL volumetric flasks

TITLE: Sulfide Analysis by Titration

Erlenmeyer flasks
Stir plate / stir bars
Buret / stand

5.0 Methods/Procedures

5.1 Reagents:

- 5.1.1 Standard Iodine solution, 0.025*N*: Dissolve 20 - 25 g KI in 10 -20 mL water and add 3.2 g iodine. After iodine has dissolved, dilute to 1000 mL and standardize against 0.025*N* Na₂S₂O₃, using starch solution as indicator.
- 5.1.2 Standard thiosulfate solution, 0.025*N*: Dissolve 6.205 g Na₂S₂O₃ · 5H₂O in distilled water. Add 1.5 mL 6*N* NaOH or 0.4 g solid NaOH and dilute to 1000 mL. Standardize with bi-iodate solution.
- 5.1.3 Standard Potassium bi-iodate solution, 0.0021 *M*: Dissolve 812.4 mg KH(IO₃)₂ in distilled water and dilute to 1000 mL.
- 5.1.4 Standardization: Dissolve approximately 2 g KI, free from iodate, in an Erlenmeyer flask with 100 to 150 mL distilled water. Add 1 mL 6*N* H₂SO₄ or a few drops of conc. H₂SO₄ and 20.00 mL standard bi-iodate solution. Dilute to 200 mL and titrate liberated iodine with thiosulfate titrant, adding starch toward end of titration, when a pale straw-yellow color is reached. When the solutions are of equal strength, 20.00 mL 0.025*M* Na₂S₂O₃ should be required. If not, adjust the Na₂S₂O₃ solution to 0.025*M*.
- 5.1.5 Starch solution: Dissolve 2 g laboratory grade soluble starch and 0.2 g salicylic acid, as a preservative, in 100 mL hot distilled water.

5.2 Procedure: (Iodometric Method)

- 5.2.1 Measure from a buret into a 500 mL Erlenmeyer flask an amount of iodine solution estimated to be an excess over the amount of sulfide present. Add distilled water, if necessary, to bring volume to about 20

TITLE: Sulfide Analysis by Titration

mL.

5.2.2 Add 2 mL 6N HCl.

5.2.3 Pipet 200 mL sample into flask, discharging sample under solution surface. If iodine color disappears, add more iodine until color remains.

5.2.4 Back-titrate with $\text{Na}_2\text{S}_2\text{O}_3$ solution, adding a few drops of starch solution as end point is approached, and continuing until blue color disappears.

5.3 The iodometric method suffers interference from reducing substances that react with iodine, including thiosulfate, sulfite, and various organic compounds, both solid and dissolved. Eliminate interference by first precipitating ZnS, removing the supernatant, and replacing it with distilled water. Use the same procedure, even when not needed for removal of interferences, to concentrate sulfide.

5.3.1 Zinc acetate solution: Dissolve 220 g $\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}$ in 870 mL water; this makes 1 L solution.

5.3.2 Sodium hydroxide solution, NaOH, 6N.

5.3.3 Put 0.20 mL (4 drops) zinc acetate solution and 0.10 mL (2 drops) 6N NaOH into a 100-mL glass bottle, fill with sample, and add 0.1 mL (2 drops) 6N NaOH solution. Stopper with no air bubbles under stopper and mix by rotating back and forth vigorously. Add enough NaOH to raise the pH above 9. Let precipitate settle for 30 min. The treated sample is relatively stable and can be held for several hours. However, if much iron is present, oxidation may be fairly rapid.

5.3.4 Collect precipitate (ppt) on a glass fiber filter. Return filter with ppt to original bottle and add about 100 mL water. Add iodine solution and HCl and titrate as in step 5.2.4.

6.0 Quality Control Requirements

TITLE: Sulfide Analysis by Titration

- 6.1 Precision: The precision of the end point varies with the sample. In clean waters it should be determinable within 1 drop, which is equivalent to 0.1 mg/L in a 200 mL sample.
- 6.2 Sulfide standard must be within 10% of known concentration.
- 6.3 Duplicates shall be run in 5% of samples, or one per batch of less than 20. Results shall be within 0.1 mg/L in a 200 mL sample.
-

7.0 Calculations/Reporting

1 mL 0.025*N* iodine solution reacts with 0.4 mg S²⁻:

$$\text{mg S}^{2-} / \text{L} = \frac{[(A \times B) - (C \times D)] \times 16,000}{\text{mL sample}}$$

where: A = mL iodine solution
B = normality of iodine solution
C = mL Na₂S₂O₃ solution
D = normality of Na₂S₂O₃ solution.

ENVIROSYSTEMS, INCORPORATED
STANDARD OPERATION PROCEDURE

SOP Number: QA-1448
 Revision Number: 6
 Date Issued: 04/97
 Page: 1 of 14

TITLE: Chronic Toxicity of Sediments to the Amphipod, *Leptocheirus plumulosus*

Approved By:

QA Officer: _____ Date: _____

President: _____ Date: _____

Revision History:

Revision Number	Changes	Authorization	Date
1	Update and Review	K. A. Simon	3/99
2	Review and Update	Sue Dionne	5/00
3	Review and Update (NELAP)	K. A. Simon	7/01
4	Update based to include EPA protocol	K. A. Simon	10/01
5	Review and Update, Addition of NELAP Requirements	S. Dionne	03/02
6	Modifications as per CH2M Hill project requirements	K. A. Simon	01/03
7	Modification to Section 11 as per request of CCH2M Hill	K. A. Simon	02/03

TITLE: Chronic Toxicity of Sediments to the Amphipod, *Leptocheirus plumulosus*

1.0 Purpose and Applicability

This Standard Operating Procedure describes methods for assessing the chronic toxicity of marine sediments to the amphipod *Leptocheirus plumulosus* using a 28-day exposure period based on methods developed by the U.S. EPA (2001) and U. S. Army Corps of Engineers (1996).

The study involves exposing neonate amphipods to marine and estuarine sediments for a period of 28 days. At the end of the exposure period the amphipods are recovered, sexed, and their growth determined based on dry weight. The number of juveniles produced are also recorded.

This protocol is suitable for evaluation of samples to assess impacts of contaminated sediments under Comprehensive Environmental Response, Compensation and Liability Act (Superfund), Clean Water Act, Marine Protection, Resources and Sanctuary Act (Dredging), National Environmental Policy Act, Resource Conservation and Recovery Act, plus additional federal programs.

This protocol has been modified to meet project specific requirements. Specifically, the frequency of the measurement for ammonia and sulfide in overlying and pore waters has been increased.

2.0 Definitions

Overlying Water: the water placed over sediment in a test chamber during a test.

Reference Sediment: a whole sediment near an area of concern used to assess sediment conditions exclusive of material(s) of interest. The reference sediment may be used as an indicator of localized sediment conditions exclusive of the specific pollutant input of concern. Such sediment would be collected near the site of concern and would represent the ambient conditions resulting from any non site related localized pollutant inputs as well as global pollutant input. This is the manner in which reference sediment is used in dredge material evaluations.

Reference-Toxicity Test: a test conducted with reagent-grade reference chemical to assess the sensitivity of the test organisms. Deviations outside an established normal range may indicate a change in the sensitivity of the test organism population. Reference-toxicity tests are most often performed in the absence of sediment.

TITLE: Chronic Toxicity of Sediments to the Amphipod, *Leptocheirus plumulosus*

Pore water: Water located in spaces between grains of sediment.

Sediment: particulate material that usually lies below water. Formulated particulate material that is intended to lie below water in a test.

Whole Sediment: sediment and associated pore water which have had minimal manipulation. The term bulk sediment has been used synonymously with whole sediment.

3.0 Applicable Documents/References

ASTM. 2001. *Guide for Conducting 10 Day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods*. Method E 1367-99. ASTM Annual Book of Standards, Volume 11.04.

U.S. EPA. 2001. *Methods for Assessing the Chronic Toxicity of Marine and Estuarine Sediment-associated Contaminants with the Amphipod, Leptocheirus plumulosus*. March 2001. EPA 600/R-01/120.

U.S. Army Corps of Engineers. 1996. *Preliminary Protocol for Conducting 28-day Chronic Sublethal Bioassays Using the Estuarine Amphipod Leptocheirus plumulosus*. Technical Note EEDP-01-36, March 1996.

4.0 Materials and Apparatus

Test Organisms, Amphipod, *Leptocheirus plumulosus*
Test Chambers, 1 L beakers drilled with screened overflow
Incubator or water bath capable of maintaining 25±3°C
Thermometer, Temperature Data Logger
pH Meter
Salinometer
D.O. Meter
Tetra-Min®
Dried Alfalfa
Wheat Grass Powder

TITLE: Chronic Toxicity of Sediments to the Amphipod, *Leptocheirus plumulosus*

Neo-Novum Shrimp Maturation Feed (Argent Chemical Laboratories)
1 mm, 0.6 mm, 0.5 mm and 0.25 mm stainless steel sieves
Blender or mortar & pestle to grind food stocks
Oven, 70°C, and desiccator
Balance, 0.01 mg sensitivity
Lachat Autoanalyzer – Ammonia Analysis
SOP 1325 - Ammonia Analysis using Lachat Autoanalyzer
SOP 1339 - Collection of Pore Water Samples
SOP 1341 - Sulfide Analysis by Titration

5.0 Methods/Procedures

5.1 Test Material

- 5.1.1 The Test material will be clearly identified by the client. Data related to sample collection time, date and location must be provided on a chain of custody for each sample. Each sample container must be clearly identified with the identification corresponding to the chain of custody. If discrepancies exist between the chain of custody and sample container or, the chain of custody is not complete, the client will be notified as soon as possible.
- 5.1.2 Upon receipt, the sample will receive a sample number and be logged into the sample inventory as described in SOP QA-1103. Test material will be stored at 2-4°C, or as specified by the client. The sample shall never be stored at temperatures below 0°C.
- 5.1.3 Maximum holding time shall be 8 weeks. Holding times for samples known to be containing volatile compounds that are of regulatory concern should be <2 weeks.

5.2 Test Species

- 5.2.1 Amphipods will be neonates ≤24 hours old or selected from those that pass through a 600 µm sieve and be retained by a 250 µm screen.

TITLE: Chronic Toxicity of Sediments to the Amphipod, *Leptocheirus plumulosus*

5.2.2 Identification of the test animals shall be verified by the supplier or by using appropriate taxonomic keys.

5.3 Pretest Observations and Procedures

5.3.1 Pretest observation data concerning the source, handling procedures, receipt date, disease treatment (if any), health, feeding, and mortality of animals used in the test will be recorded and reported.

5.3.2 The mean dry weight of the amphipods will be determined prior to the start of the assay. Weights will be determined for a minimum of 25 organisms.

5.4 Exposure Conditions

5.4.1 Amphipods will be maintained in 1 liter beakers containing approximately 175 mL sediment and approximately 725 mL of overlying water.

5.4.2 Dilution water will be natural seawater, collected from the Hampton / Seabrook Estuary, adjusted, using deionized water, to a salinity of either 5 or 20‰ with daily limits set at $\leq 3\%$. The 28-day average salinity will fall within $\leq 2\%$ of the specified level. The appropriate range will be based on a measure of initial pore water salinity. Samples with an initial pore water salinity of 1 to 10‰ will be tested at 5‰ while samples having >10 to 35‰ will be tested at 20‰. Water will be passed through a ≤ 5 micron filter. Water temperature will be $25 \pm 2^\circ\text{C}$.

5.4.3 Prior to use, test sediments will be sieved, 1.0 mm, to remove larger particles and other living organisms. Sieving will be accomplished using as little water as possible. The salinity of the water used shall equal that used for overlying water. After sieving is complete the sample is allowed to settle. After settling, the overlying water is decanted and the sample slurry is added to the test chambers.

5.4.4 Control sediment will be a natural marine sediment that has been

TITLE: Chronic Toxicity of Sediments to the Amphipod, *Leptocheirus plumulosus*

sieved, 1.0 mm, to remove larger particles and other living organisms. Laboratory control sediment will be collected from a site in the Hampton/Seabrook Estuary. The estuary, located on the New Hampshire seacoast, is rural in nature and receives no direct industrial inputs. The general area is characterized by over 10,000 areas of salt marsh with tidal creeks. Two small rivers provide freshwater influx to the system. The area is utilized for recreational purposes and mooring local fishing vessels. Sediments from the laboratory control sediment site were evaluated to determine acceptability prior to initial use.

5.4.5 Amphipods will be tested using a static renewal procedure which provides for two volume additions three times per week on a Monday, Wednesday, Friday schedule.

5.4.6 Dissolved oxygen concentration shall be maintained $\geq 60\%$ saturation, relative to temperature and salinity, during the test. Dilution water may be aerated to assure that dissolved oxygen concentrations are above 60% saturation prior to use. If aeration is required for test chambers, it will be supplied to all test chambers at a rate of approximately 100 bubbles per minute. The tip of the air line shall be approximately 2 cm above the sediment's surface to minimize sediment resuspension. Placement and type of air lines will be the same for controls and treatments.

5.4.7 Photoperiod will be automatically controlled and adjusted to 16 hours light, 8 hours dark. Light intensity will be 500 to 1000 Lux. Light source will be wide spectrum fluorescent bulbs.

5.5 Study Conduct

5.5.1 Amphipods will be exposed to the test sediments for a total of 28 days. Amphipods will be added to the test vessels after sediments have settled for 24 hours.

5.5.2 Each treatment group, sample, and control will initially consist of 100 organisms equally divided between 5 replicates. The animals will be randomly assigned to the test vessels. The control will also utilize 100 organisms distributed between 5 replicates.

TITLE: Chronic Toxicity of Sediments to the Amphipod, *Leptocheirus plumulosus*

5.5.3 The number of surviving adults and juveniles will be determined and recorded at the end of the exposure period.

5.5.4. Dissolved oxygen, temperature, pH, specific conductivity, and salinity will be measured in the overlying water in one replicate from each treatment at the start and end of the assay and prior to water renewals during the assay. Instruments will be checked and calibrated as specified in Section 9 of ESI's Quality Assurance Manual.

Ammonia and sulfide will be measured in the overlying water on days 0 (start), 7, 14, 21 and 28 (end) of the assay in each treatment. Ammonia analysis will be conducted following guidance specified in ESI SOP 1325. Sulfide will be measured using the titration, ASTM Method 4500-S²-F, ESI SOP 1341 (comparable to EPA 376.2).

Ammonia, sulfide, pH, temperature and salinity will be measured in the pore water from one replicate of each treatment on days 0, 7, 14, 21 and 28 of the assay. Pore water samples will be collected utilizing procedures outlined in ESI SOP number 1339, "Collection of Sediment Pore Water Samples."

If the unionized ammonia in the pore water at the start of the assay exceeds 0.8 mg/L the client will be notified and the data reviewed. If the unionized ammonia is deemed to be sufficient high to result in acute toxicity the sediments may be "washed" according to Army Corps of Engineers protocol to reduce ammonia levels.

Temperature will be measured in one surrogate test chamber or water bath on an hourly basis.

5.5.5 To insure an adequate level of food, test amphipods will be fed a prepared feed on a Monday/Wednesday/Friday basis. Feeding rates during the first two weeks will be 1 mg per initial amphipod (total of 20 mg/replicate). During the second two weeks of the assay the rate is increased to 2 mg/amphipod (total of 40 mg/replicate).

TITLE: Chronic Toxicity of Sediments to the Amphipod, *Leptocheirus plumulosus*

5.5.6 Food for the amphipods will be based on guidance provided in U.S. Army Corps of Engineers (1996) guide for conducting the 28-day exposure assay. Food will be prepared as follows:

Mix in a blender and pass through a 0.5 mm screen.

48.5 grams Tetra-Min®

24.0 grams dried Alfalfa

24.0 grams Wheat Grass Powder

4.5 grams Neo-Novum Shrimp Maturation Feed

5.6 Study Termination

5.6.1 At the end of the exposure period the sediments will be removed from the chamber and sieved through 600 and 250 μm mesh screens stacked together. Amphipods retained by the 600 μm screen will be classified as adults while those retained by the 250 μm screen are classified as juveniles.

5.6.2 Amphipods will be collected and counted. Adult male and female amphipods will be enumerated separately. Juveniles will be enumerated separately from the adult amphipods.

5.6.3 Male and female adult amphipods will be transferred to separate tarred weigh boats and dried at 70°C for 24 hours. Amphipods will be allowed to cool in a desiccator and weighed to the nearest 0.01 mg.

6.0 Quality Control Requirements

6.1 A 'water only' reference toxicant assay will be conducted for each batch of test organisms.

6.2 After the organisms have been recovered from the sediments a representative number of the recovered organisms, 10% of the test chambers, will be recounted to determine the accuracy of the initial count.

6.3 Interferences

TITLE: Chronic Toxicity of Sediments to the Amphipod, *Leptocheirus plumulosus*

- 6.3.1 Salinity less than 5‰ may result in reduced ability for the amphipods to reproduce.
- 6.3.2 Water quality (dissolved oxygen, salinity, temperature) parameters outside established limits may impact the outcome of the assay. In cases where these values exceed protocol limits overall water quality data will be evaluated to determine if there was a likelihood of a significant negative impact. In cases where decreases in dissolved oxygen are noted the monitoring frequency will be increased. The increased monitoring will provide additional data to make a determination regarding use of aeration.
- 6.3.3 Variation in total organic carbon (TOC) content of the sediments may impact the outcome of the assay. Published data suggest that TOC values <1% and >7% may impact survival. In cases where TOC values are outside these ranges a reference sediment with similar TOC levels should be included in the design.
- 6.3.4 Pore water salinity, ammonia and sulfides may impact the outcome of an assay. Data from available literature indicates that pore water salinity <1‰ may impact the outcome of an assay. Salinity values between 1 and 35‰ have had no impact on the outcome of an assay. There is no available data related to pore water salinity values of >35‰. Pore water ammonia levels of >16 mg/L may have lethal and sublethal impacts on *L. plumulosus*. The presence of hydrogen sulfide in test sediments may have an impact on the outcome of an assay. Currently, no data is available to determine levels of hydrogen sulfide that may result in acute or sublethal impacts. *L. plumulosus*'s burrowing activity and circulation of water into the burrows will reduce or eliminate exposure to pore water hydrogen sulfide. Monitoring pore water ammonia and sulfides at the start and end of the assay will document existing conditions. Additional monitoring during an assay would provide data for analysis of trends in these two parameters.

6.4 Detection Limits – Not Applicable for Assay

6.5 Precision and accuracy of meters used in the assays to measure water

TITLE: Chronic Toxicity of Sediments to the Amphipod, *Leptocheirus plumulosus*

quality will be documented as described in Section 9 of ESI's Quality Assurance Manual. Precision and accuracy of ammonia and sulfide analyses will be documented by analysis of duplicate and known, ammonia, and prepared, sulfide, samples.

7.0 Calculations/Reporting

7.1 Statistical Analysis of Data

7.1.1 Survival data generated will be evaluated using analysis of variance techniques. Data will be evaluated using the TOXSTAT, or similar, program. Prior to conducting the ANOVA, the data set will be evaluated to determine its normality and homogeneity of variances. Data sets that are normally distributed and homogeneous will be evaluated using parametric statistics.

7.1.2 The number of juvenile amphipods produced per original amphipod and per surviving female will be determined. A separate determination of numbers of juveniles per female is essential to allow for compensation in cases where there are no or few females in a sample. These values will be analyzed using analysis of variance techniques.

7.1.3 Dry weights of the surviving adults and females will be determined along with the mean growth per day per surviving male and surviving female. These values will be analyzed using analysis of variance techniques.

7.2 Reporting

All survival and tissue body burden data will be summarized and presented in tabular format. The final report will provide the following: Introduction, methods and materials, results of reference toxicant assay, and summary of results. Copies of all bench sheets, raw data and statistical output will be provided in an appendix included with the report.

TITLE: Chronic Toxicity of Sediments to the Amphipod, *Leptocheirus plumulosus*

8.0 Corrective Actions

8.1 Acceptability Criteria

8.1.1 The amphipod assay will be considered acceptable if environmental parameters (temperature, dissolved oxygen, salinity, and pH) fall within the ranges specified. Mean survival in the control sediments after 28 days exposure will be $\geq 80\%$ with no single replicate have $< 60\%$ survival. In addition, there will be evidence of juvenile reproduction and growth in the control treatment.

8.1.2 Criteria specified in Section 11 will be met.

8.2 If survival fails to meet the minimum value specified by the protocol the client will be notified and the test restarted.

8.3 If water quality values fall outside study limits the laboratory manager, using sound scientific practice, will determine if the study requires repeating or the data is allowed to be accepted. The client will be notified, the results reviewed and a final determination made as to the acceptability of the data.

8.4 Corrective Actions

8.4.1 In the event that an element of the assay falls outside acceptable limits or there is a change in the protocol a Corrective Action Report must be initiated and completed.

9.0 Health and Safety

9.1 As with all samples, gloves and safety glasses should be worn when handling sediment samples, effluents and chemicals.

9.2 At the end of an assay excess sample material and material used in the assay will be disposed of properly. Material may be returned to the client, air dried and placed in an appropriate container for disposal at an

TITLE: Chronic Toxicity of Sediments to the Amphipod, *Leptocheirus plumulosus*

approved disposal facility or, in the case where the material is non-hazardous, the material may be disposed in an appropriate waste container.

- 9.3 Assays and sample disposal will be conducted using procedures to minimize potential pollution of surface and ground waters plus soil and sediments.

10.0 Responsibilities

- 10.1 It is the Lab Manager's responsibility to ensure analysts performing this procedure are properly trained and the training is documented in their training file. The analyst is responsible for following the procedures outlined in this SOP.
- 10.2 Prior to any staff member working unsupervised on a testing procedure, they must be certified by the Laboratory Manager. Certification will include reading this and associated SOP's, review of the primary literature and participation in similar procedures under the direct supervision of a trained staff member. Certification will be based upon a review of the persons' demonstrated abilities.

TITLE: Chronic Toxicity of Sediments to the Amphipod, *Leptocheirus plumulosus*

11.0 Summary of Test Conditions

1. Test Mode: Static Renewal
2. Test Duration: 28 Days
3. Renewal Schedule: Two volume additions three time per week
4. Temperature: $25 \pm 3^{\circ}\text{C}$; 28-day average $25 \pm 2^{\circ}\text{C}$
5. Photoperiod: 16 hr light/ 8 hr dark
6. Light Source: Wide - spectrum fluorescent
7. Light Intensity: 500 to 1000 Lux
8. Salinity: 5 or $20\text{‰} \pm 3\text{‰}$; use 5‰ if pore water salinity is 1 to 10‰ and 20‰ if pore water salinity is >10 to 35‰ .
9. Test Chamber: 1000 mL beakers
10. Solution Volume: 725 mL overlying water (approximately)
11. Sediment Depth: 175 mL
12. Organisms/Chamber: 20
13. Replicates/Treatment: 5, minimum; 5 for control
14. Treatments: Site Sediment, and Control Sediment
15. Age of Organisms: Neonate amphipods, ≤ 24 hours or retained between $250 \mu\text{m}$ and $600 \mu\text{m}$ screens
16. Feeding Regime: 1 mg prepared feed per amphipod for first two weeks, 2 mg per amphipod for last two weeks
17. Dilution Water: Natural Seawater - salinity adjusted
18. Aeration: As required to maintain D.O. $\geq 60\%$ saturation. Rate set so a not to resuspend sediments
19. Endpoint: Survival, growth (weight), reproduction
20. Acceptability: Control survival of $\geq 80\%$ with reproduction and growth in control treatment. No single replicate with less than 60% survival
21. Support Chemistry: Measurement of D.O., salinity, ammonia, sulfide and pH in pore water from one replicate of each treatment on days 0 and 28. Measurement of ammonia and sulfide in pore water on days 7, 14, and 21. Measurement of D.O., salinity, pH in overlying water prior to water change in 1 replicate. Hourly

TITLE: Chronic Toxicity of Sediments to the Amphipod, *Leptocheirus plumulosus*

- temperature in surrogate vessel. Measurement of ammonia and sulfide in overlying water, prior to renewal in one replicate of each treatment on days 0, 7, 14, 21 and 28 of the assay.
22. Pore Water Ammonia <60 mg/L total ammonia, <0.8 mg/L unionized ammonia.
 23. Pore Water pH Normal range of 7.0 to 9.0. If values fall outside of range notify client before preceding.