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Post-Removal Action Verification Field Sampling Plan

for

Site 8 - Nitroglycerin Plant Office Indian Head Division Naval Surface Warfare Center

Indian Head, Maryland

Northern Division
Naval Facilities Engineering Command
Contract Number N62472-90-D-1298
Contract Task Order 0099

April 1994

Halliburton NUS
CORPORATION

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POST-REMOVAL ACTION
VERIFICATION FIELD SAMPLING PLAN
SITE 8 - NITROGLYCERIN PLANT OFFICE
INDIAN HEAD DIVISION, NAVAL SURFACE WARFARE CENTER
INDIAN HEAD, MARYLAND

COMPREHENSIVE LONG-TERM
ENVIRONMENTAL ACTION NAVY (CLEAN) CONTRACT

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Environmental Branch Code 18
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Submitted by:
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1.0 INTRODUCTION

The Northern Division of the Naval Facilities Engineering Command has issued Contract Task Order Number 0099 (CTO 99) to Halliburton NUS Corporation (Halliburton NUS), under the Comprehensive Long-Term Environmental Action Navy (CLEAN) Contract No. N62472-90-D-1298.

CTO 99 is for environmental work to support interim removal action at Site 8 - Nitroglycerin Plant Office (Site 8), at the Indian Head Division, Naval Surface Warfare Center, (IHDIVNAVSURFWARCEN or NSWC) in Indian Head, Maryland. IHDIVNAVSURFWARCEN is part of the Engineering Field Activity, Chesapeake (EFA Chesapeake). This Post-Removal Action Verification Field Sampling Plan presents a plan to perform sampling and analysis work to confirm the removal of mercury contaminated sediment and soil at Site 8.

1.1 PROJECT BACKGROUND

The NSWC contains approximately 3,423 acres and is located approximately 25 miles southwest of Washington, D.C., in the northwestern section of Charles County, Maryland. It is located on a peninsula bounded by the Potomac River to the northwest and Mattawoman Creek to the south and southeast. As shown on Figure 1-1, Site 8 is located on the southeast side of the NSWC; water from Site 8 discharges into Mattawoman Creek.

Site 8 - Nitroglycerin Plant Office consists of the following: Building 766 - Former Nitroglycerin Plant Office Laboratory, a 3-inch drain pipe from Building 766 to a manhole (Manhole A), a concrete pipe that conveys water from Manhole A under railroad tracks to a stream, approximately 1,300 feet of stream, and a pond/ marsh area. The pond/swamp area is approximately 700 feet long and 200 feet wide; it includes both a marsh and open water area. The pond outlets through a culvert under Noble Road and into Mattawoman Creek.

Building 766 was constructed in 1953. From 1953 to 1981, mercury used in laboratory tests in Building 766 was inadvertently disposed through drains and discharged into Site 8. Since 1981, the drains have been sealed and the practice discontinued. Environmental investigative work began at Site 8 after mercury contamination was discovered in 1981. In 1984, a contractor inadvertently broke the drain pipe from Building 766 to Manhole A; mercury was observed leaking into the soil. The drain pipe, Manhole A, and approximately 200 drums (55-gallon capacity) of mercury-contaminated soil were excavated and removed from the site. After the excavation was completed, the drain pipe, manhole, and soil were replaced; the

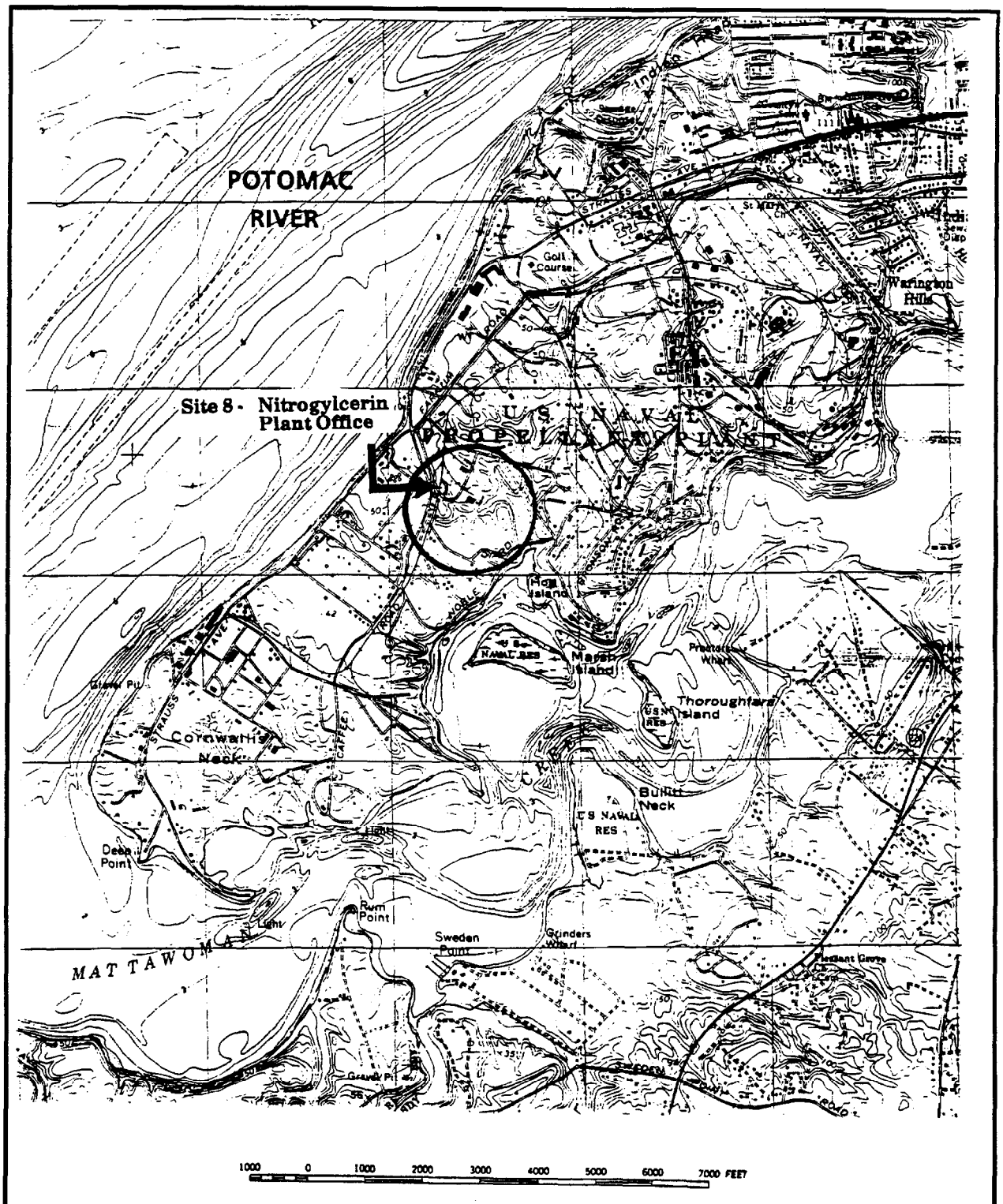

FIGURE 1-1

SITE LOCATION MAP
SITE 8 - NITROCLYCERIN PLANT OFFICE

HALLIBURTON NUS
Environmental Corporation

Source: 7.5 Minute USGS Quad Indian Head, MD-VA 1966 Photo Revised 1978

removal and subsequent replacement action was performed with oversight from the enforcement staff of the state of Maryland. The previous environmental investigative work at Site 8 has indicated that sediments in the upper section of the stream are contaminated with mercury; because of this contamination, a removal action has been proposed for the mercury-contaminated section of the stream.

1.2 OBJECTIVE AND SAMPLING STRATEGY

The objective of the work described in this Post-Removal Action Field Sampling Plan is to obtain sufficient data to confirm that mercury-contaminated sediment/soils at concentrations above the action level of 10 mg/kg at Site 8 have been removed. The data will be used to justify that no further removal action is required and/or delineate the area which requires additional removal.

If it is determined from the post-removal sampling analytical results that there are areas which need to be removed, then after the additional removal action another sampling event will occur to confirm the mercury-contaminated sediment/soils have been removed to meet the set remediation goal of less than 10 mg/kg total mercury in the sediment/soils.

1.3 PLAN FORMAT

Section 1.0 of this Post-Removal Action Field Sampling Plan contains an introduction, background information, and sampling strategy. Sections 2.0 and 3.0 describe the field operations environmental sampling procedures that will be used to implement the field work. Section 4.0 contains the Quality Assurance/Quality Control Information. The Site Management Plan is provided in Section 5.0. Additional support information is included in the appendices.

2.0 FIELD OPERATIONS

Field sampling operations to be performed at Site 8 as part of the post removal action post verification sampling will consist of collecting sediment/soil samples to verify the removal of mercury contamination above 10 mg/kg. The initial field sampling program will consist of collecting 21 sediment/soil samples plus 6 QA/QC samples for laboratory analysis. If mercury concentrations are detected in the initial samples at levels greater than 10 mg/kg, additional extraction will be performed and additional post removal verification samples collected and analyzed. One aqueous sample may also be collected to verify removal action at the pipe.

2.1 MOBILIZATION/DEMOBILIZATION

Halliburton NUS will mobilize to Site 8 before the removal action contractor begins excavation work. All Halliburton NUS field personnel will review this Post-Removal Sampling Plan including the Health and Safety Plan (HASP) contained in the appendices. In addition, a field team orientation meeting will be held with the removal action contractor to familiarize personnel with the scope of the field activities.

The Halliburton NUS Field Operations Leader (FOL) will coordinate the field activities upon arrival at NSWC. The FOL will also obtain equipment and make any equipment purchases required to conduct the field activities. It is anticipated that the equipment required for the field activities will be obtained from the Halliburton NUS warehouse in Pittsburgh and driven to the site. After field activities are completed, the FOL will demobilize the equipment and drive back to Pittsburgh.

2.2 SEDIMENT/SOIL SAMPLING

Samples taken to verify the removal of mercury-contaminated sediment/soils shall be surface samples from the edge of the excavation. The initial sample collection will consist of twenty-one field samples; three samples shall be taken in seven transect along the length of the removal effort. One sample shall be taken of the bottom of the excavation and one sample shall be taken from either side of the excavation. These samples will be able to verify the horizontal and vertical extent of removal. The approximate locations of the locations for the samples are as shown in Figure 2-1. Six QA/QC samples will also be collected.

Table 2-1 provides a summary of the location and samples to be collected.

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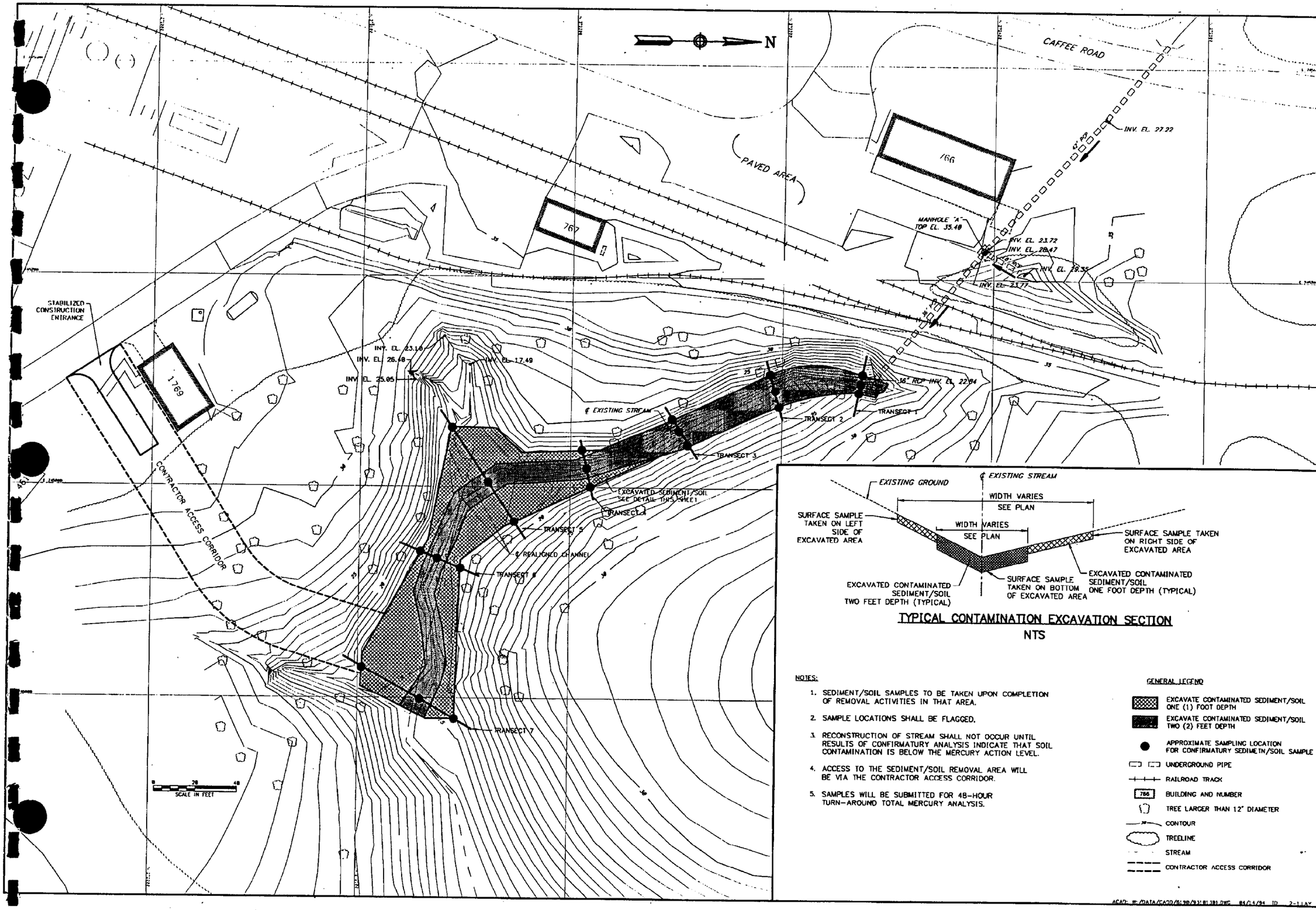

N
CAFFEE ROAD
PAVED AREA
766
767
769
INV. EL. 27.22
MANHOLE "A"
TOP EL. 35.48
INV. EL. 23.72
INV. EL. 28.47
INV. EL. 29.35
INV. EL. 23.77
STABILIZED CONSTRUCTION ENTRANCE
INV. EL. 23.18
INV. EL. 26.48
INV. EL. 25.05
INV. EL. 17.49
16" RCP INV. EL. 22.94
℄ EXISTING STREAM
TRANSECT 1
TRANSECT 2
TRANSECT 3
EXCAVATED SEDIMENT/SOIL SEE DETAIL THIS SHEET
TRANSECT 4
TRANSECT 5
℄ REALIGNED CHANNEL
TRANSECT 6
TRANSECT 7
CONTRACTOR ACCESS CORRIDOR
SCALE IN FEET
EXISTING GROUND
℄ EXISTING STREAM
WIDTH VARIES
SEE PLAN
SURFACE SAMPLE TAKEN ON LEFT SIDE OF EXCAVATED AREA
SURFACE SAMPLE TAKEN ON RIGHT SIDE OF EXCAVATED AREA
EXCAVATED CONTAMINATED SEDIMENT/SOIL TWO FEET DEPTH (TYPICAL)
SURFACE SAMPLE TAKEN ON BOTTOM OF EXCAVATED AREA
EXCAVATED CONTAMINATED SEDIMENT/SOIL ONE FOOT DEPTH (TYPICAL)
TYPICAL CONTAMINATION EXCAVATION SECTION
NTS
NOTES:
1. SEDIMENT/SOIL SAMPLES TO BE TAKEN UPON COMPLETION OF REMOVAL ACTIVITIES IN THAT AREA.
2. SAMPLE LOCATIONS SHALL BE FLAGGED.
3. RECONSTRUCTION OF STREAM SHALL NOT OCCUR UNTIL RESULTS OF CONFIRMATORY ANALYSIS INDICATE THAT SOIL CONTAMINATION IS BELOW THE MERCURY ACTION LEVEL.
4. ACCESS TO THE SEDIMENT/SOIL REMOVAL AREA WILL BE VIA THE CONTRACTOR ACCESS CORRIDOR.
5. SAMPLES WILL BE SUBMITTED FOR 48-HOUR TURN-AROUND TOTAL MERCURY ANALYSIS.
GENERAL LEGEND
EXCAVATE CONTAMINATED SEDIMENT/SOIL ONE (1) FOOT DEPTH
EXCAVATE CONTAMINATED SEDIMENT/SOIL TWO (2) FEET DEPTH
APPROXIMATE SAMPLING LOCATION FOR CONFIRMATORY SEDIMETN/SOIL SAMPLE
UNDERGROUND PIPE
RAILROAD TRACK
766 BUILDING AND NUMBER
TREE LARGER THAN 12" DIAMETER
CONTOUR
TREELINE
STREAM
CONTRACTOR ACCESS CORRIDOR
REVISIONS
DESCRIPTION
DATE
APPROVED
ENGINEER FIELD ACTIVITY CHESAPEAKE
NORFOLK, VA
INDIAN HEAD DIVISION,
NAVAL SURFACE WARFARE CENTER
REMOVAL OF CONTAMINATED SOIL
POST REMOVAL ACTION VERIFICATION SAMPLING AND ANALYSIS PLAN
CODE I.D. NO. 80091
DRAWING SIZE: D
CONST. CONT. NO. N62477-93-C-0164
SPEC. 21-93-0164
NAVFAC DRAWING NO.
SHEET 1 OF 1
2-1

TABLE 2-1

INITIAL SEDIMENT/SOIL SAMPLING LOCATIONS
INDIAN HEAD, MARYLAND

Location	Location Description	Sampling Summary	Field Samples	Total No. of Samples
Transect 1	The area just below the 36" RCP outlet.	Collect 1 sample from each side of the excavated area and 1 in the middle. Collect 1 sample event rinsate blank to verify decon procedures.	3	4
Transect 2	The area in the upper part of the stream. (Approximately 50' downstream of transect 1)	Collect 1 sample from each side of the excavated area and 1 in the middle.	3	3
Transect 3	The area in the upper middle of the stream, just before where the excavation starts to include 1-foot removal. (Approximately 50' downstream of transect 2)	Collect 1 sample from each side of the excavated area and 1 in the middle. Provide one QA/QC duplicate sample.	3	4
Transect 4	The area in the middle of the stream. (Approximately 50' downstream of transect 3)	Collect 1 sample from each side of the excavated area and 1 in the middle. Collect 1 sample event rinsate blank to verify decon procedures.	3	4
Transect 5	The lower middle of the stream where the tributary stream connects. (Approximately 50' downstream of transect 4)	Collect 1 sample from each side of the excavated area and 1 in the middle. Provide one QA/QC duplicate sample.	3	4
Transect 6	The lower section of the stream where the excavation narrows. (Approximately 50' downstream of transect 5)	Collect 1 sample from each side of the excavated area and 1 in the middle. Collect 1 sample event rinsate blank to verify decon procedures.	3	4
Transect 7	The downstream edge of the excavation. (Approximately 50' downstream of transect 6)	Collect 1 sample from each side of the excavated area and 1 in the middle. Provide one QA/QC duplicate sample.	3	4
TOTAL			21	27

The surface sediment/soil samples will be collected after initial excavation activities are completed in that area. The samples are to be submitted to the laboratory and analyzed for total mercury on a 48-hour turnaround basis. If the samples are determined to contain mercury in quantities above the remediation goal of 10 mg/Kg total mercury, then additional excavation will be performed to remove the contaminated sediment/soils. The extent of the additional removal will be determined in the field by examining the surrounding elevations and grades. After the second round of removal action (excavation) additional sediment/soil samples will be collected from the edge of the new excavation and the process repeated. If the samples submitted for total mercury analysis are below the remediation goal then the removal action can stop in that area, the removal action contractor can perform the stream reconstruction activities.

During field sampling, a lithologic description will be made of each sample and a complete sample log of each location will be maintained by the FOL. At a minimum, the Halliburton NUS sediment/soil field sample log will contain the following information:

- Sample number
- Soil density or cohesiveness
- Soil color and moisture
- Apparent depositional history (recent vs. residual)
- Relative organic content
- Miscellaneous observations

2.3 VERIFICATION OF PIPE CLEANING

The 36-inch reinforced concrete pipe (RCP) will be thoroughly cleaned by the removal action contractor. Halliburton NUS will visually inspect the pipe to verify that the pipe has been adequately cleaned. Based on actual field conditions potable water may be run through the length of the pipe and collected to assist in determining the adequacy of the pipe cleaning. The collected water may be analyzed for total mercury.

2.4 DECONTAMINATION

The equipment involved in field sampling activities will be decontaminated prior to, during, and at completion of the sampling activities.

The following decontamination steps will be taken:

- Potable water rinse

- Alconox or liquinox detergent wash
- Portable water rinse
- Dilute nitric acid rinse
- Analyte-free water rinse
- Air dry

All decontamination fluids will be discharged onto the ground at the site. Nitric acid rinsate will be collected and neutralized prior to discharge.

2.5 WASTE HANDLING

All disposal equipment, Tyveks and other investigative derived waste (IDW) will be collected by Halliburton NUS and placed in plastic garbage bags. All bagged IDW will be disposed of by the Navy.

2.6 RECORD KEEPING

In addition to chain-of-custody records provided in Appendix A, a standard sample log sheet for sediment/ soils will be completed for sample description and documentation: This form is also contained in Appendix A.

A bound/weatherproof site logbook shall be maintained by the FOL. The requirements of the site logbook are outlined in Halliburton NUS SOP SA-6.3. This book will contain a summary of the day's activities and will reference the field notebooks when applicable. All information related to sampling or field activities will be recorded in the field notebook. This information will include, but is not limited to, sampling time, weather conditions, unusual events, field measurements, descriptions of photographs, etc.

At the completion of field activities, the FOL shall submit all field records, data, field notebooks, chain-of-custody receipts, sample log sheets, drilling logs, daily logs, etc., to the Project Manager.

2.7 HEALTH AND SAFETY

All field activities will be performed in accordance with the Site-Specific Health and Safety Plan contained in Appendix C of this FSP.

3.0 ENVIRONMENTAL SAMPLING PROCEDURES

This section describes the procedures for sediment/soil environmental sampling performed at Site 8. This chapter also describes any sample handling requirements which may be applicable.

3.1 SAMPLING METHODS

This section details the site-specific methodology to be used to obtain the environmental samples. Field sampling procedures are described in the Halliburton NUS Standard Operating Procedures Guidelines contained in Appendix B. Table 3-1 provides an analytical summary for samples collected.

3.1.1 Sediment/Soil Sampling

Sediment/soil samples will be collected from the upper section of the stream as shown in Figure 2-1. The samples collected shall be surface samples. The soil sample shall be removed from the side walls and bottom of the excavation. A volume of soil measuring approximately 3 inches by 3 inches by 1 inch deep shall be taken using a stainless steel or disposable trowel and placed in the sample jar and labelled as described in Section 3.2. The chain-of-custody procedure are described in Section 3.3 (see Halliburton NUS Standard Operating Procedure [SOP] GH-1.3 in Appendix B of this FSP) and the field log book. If stainless steel trowels are utilized during sampling, the trowels must be decontaminated between samples as described in Section 2.3.

After each sampling event (number of transects completed will depend on progress of excavation activity), a rinsate blank QA/QC sample will be taken to confirm proper decontamination procedures have been followed. This will be performed by pouring analyte-free water over the decontaminated utensils into the sample bottle. Three QA/QC duplicate samples are required and can be taken from the upper, middle, and lower sections of the excavated stream.

As the removal activities proceed, sampling efforts will be performed and submitted for 48-hour turnaround total mercury analysis. Samples shall not be taken until it is confirmed the full extent of excavation, according to the contract drawings, has been performed.

The location where the samples are taken shall be flagged and these flags are not to be disturbed until the results of the analysis indicate the remediation goal of less than 10 mg/kg has been achieved. If mercury

TABLE 3-1

ANALYTICAL PROGRAM
INDIAN HEAD, MARYLAND

Parameter	Method	Detection Limit	Sample Matrix	Estimated No. Samples	Equipment Rinsate Blanks[1]	Duplicates[2]	Total Samples
Mercury (dry weight basis)	CLP SOW ILM02.1	0.1 mg/kg (ppm)	Sediment Soil	21	3	3	27
Mercury, total	CLP SOW ILM02.1	0.2 mg/kg (ppm)	Water	1	0	0	1

(1) Equipment Rinsate Blank - Samples obtained by pouring analyte-free water over sample collection equipment after decontamination. Assesses the effectiveness of field decontamination procedure. Obtained at a frequency of 1/day/media/sampling train but only samples obtained from every other day are analyzed unless significant positive detections are recorded. Number of samples reflects the number of actual laboratory analyses performed.

(2) Duplicates - A single sample split into two portions during a single act of sampling. Assess the overall precision of the sampling and analysis program. Obtained at a frequency of 10% of the number of samples.

contamination is found additional excavation will be performed and verification sampling performed until the clean up goal is achieved.

3.1.2 36-Inch RCP Cleaning

After the pipe has been thoroughly cleaned by the removal action contractor, it will be visually inspected to verify it has been adequately cleaned. All visible sediments/material lining the pipe shall be removed. Based on the visual condition of the pipe after cleaning, a potable water rinse may be performed. The potable water rinse will consist of flowing potable water down the length of the pipe, collecting it in a clear plastic container and then inspecting it for presence of sediments. If the sediments have not been sufficiently removed, the removal action contractor will perform additional cleaning of the 36-inch RCP until it is determined that the pipe has been adequately cleaned.

3.2 SAMPLE HANDLING

Sample handling includes the field-related considerations concerning the selection of sample containers, preservatives, allowable holding times and analysis requested. In addition, sample identification, packaging and shipping will be addressed.

The EPA User's Guide to the Contract Laboratory Program (EPA, December 1986), and the Federal Register (EPA, October 26, 1984) address the topics of containers and sample preservations.

3.2.1 Sampling Identification System

Each sample collected will be assigned a unique sample tracking number. This three to four segment alpha-numeric code will identify the location and QA designation, if required.

The alpha-numeric code to be used in the sampling event is as follows:

(AAN)	-	(A)	-	(NN)	-	(A)
[Sample Location]		[Sample Area]		[Sample Number]		[QA/QC Sample Designation]

Sample Location:

TS1 = Transect number (i.e., TS1, TS2, TS3, etc.)

Sample Area:

R = Right side of excavated area (facing downstream)
B = Bottom of excavated area
L = Left side at excavated area (facing down stream)
W = Water sample collected at pipe

Sample Number:

01 through 27 = A sequential ordering of samples taken

QA/QC Sample Designation:

E = Equipment Rinse

Duplicate samples will be "blind." The duplicate samples will be recorded in the field book. For example, the first sediment/soil sample collected from the middle of the excavated area by the 36" RCP outlet would be designated as:

TS1-B-01 (Transect 1 - Bottom - First sample taken)

A rinsate blank for that location would be:

TS1-B-02-E (Transect 1 - Bottom - Second sample taken - Equipment Rinse)

Information regarding sample labels and tags to be attached before shipment to a laboratory is contained in of the Halliburton NUS SOP SA-6.1.

3.2.2 Sample Packaging and Shipping

Samples will be packaged and shipped in accordance with Halliburton NUS SOP SA-6.2. The FOL will be responsible for completion of the following forms:

- Sample Labels
- Chain-of-Custody Forms
- Appropriate labels applied to shipping coolers
- Chain-of-Custody Labels

It is anticipated that the laboratory to be used for the analytical work will provide delivery of sample bottles and pickup of samples on a daily basis.

3.3 SAMPLE CUSTODY

Custody of samples must be maintained and documented at all times. Chain-of-custody begins with the collection of the samples in the field. Halliburton NUS SOP SA-6.1 provides a description of the chain-of-custody procedures to be followed. A sample chain-of-custody form is contained in Appendix A.

4.0 QUALITY ASSURANCE/QUALITY CONTROL

This section provides technical guidelines and procedures for conducting the field work. This document references the Halliburton NUS Standard Operating Procedures (SOPs) for specific protocols pertaining to individual procedures discussed.

4.1 QUALITY ASSURANCE OBJECTIVES

The objective of the post-removal verification sampling is to provide sufficient data to demonstrate that the cleanup goals developed in the Removal Plan have been achieved.

Achieving the above described objectives requires that the data collected from the field conform to an appropriate level of quality. The quality of a data set is measured by certain characteristics of the data, namely the PARCC parameters, precision and accuracy, representativeness, completeness, and comparability. Some of the parameters are expressed quantitatively, while others are expressed qualitatively. The PARCC goals for a particular project are determined by the intended use of the data, usually referred to as Data Quality Objectives (DQOs).

4.1.1 Data Quality Objectives

The intended use of the data resulting from a field investigation is a determining factor in defining the Data Quality Objective (DQO) for that data. As described by the Naval Energy and Environmental Support Activity (NEESA), the Navy has adopted three analytical quality levels (C, D, and E) corresponding to EPA quality Levels III, IV, and V described in the EPA document "Data Quality Objectives for Remedial Response Activities Development Process."

The analytical data reports generated as a result of the verification sampling discussed in this document will conform with NEESA data quality Level D requirements. The data will serve engineering considerations in addition to determining if cleanup goals have been met.

4.1.2 PARCC Parameters

The precision and accuracy, representatives, completeness, and comparability (PARCC) goals for the work covered by this FSP are discussed in the following sections.

4.1.2.1 Precision and Accuracy

Field and laboratory precision and accuracy performance can affect the attainment of project objectives, particularly when compliance with established criteria is based on laboratory analysis of environmental samples.

Analytical precision and accuracy will be evaluated upon receipt of the laboratory data. Analytical precision will be measured as the relative percent difference (RPD) of the data from the laboratory (internal) duplicates. Analytical accuracy measures the bias as the percent recovery from matrix spike and matrix spike duplicate samples. The quality control criteria identified int he Contract Laboratory Program (CLP) Statement of Work (SOW) will be used to evaluate analytical accuracy.

Field sampling precision and accuracy are not easily measured. Field contamination, sample preservation, and sample handling will affect precision and accuracy. By following the appropriate Halliburton NUS Standard Operating Guidelines, precision and accuracy errors associated with field activities can be minimized. Field duplicates and rinsate blanks will be used to estimate field sampling precision and accuracy.

No project resources will be expended to develop precision and accuracy data for method (field or analytical) validation except those commonly applied for collection of routine QA/QC data. Routine QA/QC data will include analyses from field duplicates and equipment rinsate blanks based on the existing NEESA guidance that specifies the type and frequency of QA/QC sample preparation.

Validity of data with respect to its intended use will be assessed based on laboratory-supplied QA/QC data and protocols routinely employed for validation of CLP analytical results. In general, results that are rejected by the validation process will be disqualified from application to the intended use. Qualified data will be used to the greatest extent practicable.

4.1.2.2 Representativeness

Representativeness describes the degree to which analytical data accurately and precisely define the population being measured. Several elements of the sampling and sample handling process must be controlled to maximize the representativeness of the analytical data (e.g., appropriate number of samples collected, physical state of the samples, site-specific factors, sampling equipment, containers, sample preservation and storage, holding times, sample identify and chain-of-custody will be defined to assure that

the samples analyzed represent the population being measured). The sampling program is designed to provide analytical data that is representative of the existing contaminant levels.

Representativeness of data is also affected by sampling techniques. Sampling techniques are described in the Halliburton NUS Standard Operating Procedures included in Appendix B.

4.1.2.3 Completeness

Completeness describes the amount of data generated that meets the objectives for precision, accuracy, and representativeness versus the amount of data expected to be obtained. For relatively clean, homogeneous matrices, 100 percent completeness is expected. However, as matrix complexity and heterogeneity increase, completeness may decrease. Where analysis is precluded or where data quality objectives are compromised, effects on the overall investigation must be considered. Whether or not any particular sample is critical to the investigation will be evaluated in terms of the sample location, the parameter in question, the intended data use, and the risk associated with the error.

The sampling and analysis program for the site is sufficiently broad in scope to prevent a single data point or parameter from jeopardizing attainment of the monitoring objectives. Each medium is critical to the assessment of contaminant migration. Consequently, there exists some critical data requirement below which the objectives of the monitoring program will be compromised.

Critical data points may not be evaluated until all the analytical results are evaluated. Additionally, several sampling points, in aggregate, may be considered to be critical either by location or by analysis. If in the evaluation of laboratory results it becomes apparent that the data for a specific medium are of insufficient quality, either with respect to the number of samples or an individual analysis, a subsequent sampling event may be necessary.

For the purposes of this effort, 90 percent is established as the minimum acceptable level of completeness. A data point shall be determined to contribute to the completeness of the data set if the information provided is meaningful, useful and contributes to the project objectives.

4.1.2.4 Comparability

One of the objectives of this FSP is to provide analytical data that is characterized by a level of quality that is comparable between sampling points. By specifying the use of standard analytical procedures in addition

to standardizing field sampling procedures by employing Halliburton NUS SOPs, the potential for variables to affect the final data quality will have been effectively minimized.

4.1.3 Quality Control Samples

Quality control (QC) samples to be collected during the sampling effort are identified below. QC samples include field duplicates or replicates, laboratory duplicates or replicates, and rinsate blanks. Each type of quality control sample defined below will undergo the same controls. The samples are to be submitted for total mercury analysis with a 48-hour turnaround.

4.1.3.1 Field Duplicates

Field Duplicates will be submitted at the rate of one for every 10 samples, per matrix. Field personnel will note in the remarks block on the Chain-of-Custody Form which of the duplicate samples is to be used for internal laboratory duplicate analysis. Field duplicates are collected by mixing a double portion of the required volume of sample and dividing it into two sample containers. Field duplicates provide precision information regarding homogeneity, handling, shipping, storing, preparation, and analysis.

4.1.3.2 Trip Blanks

Trip blanks are customarily analyzed for volatile organic compounds. Because post-removal verification samples will be analyzed for total mercury only, no trip blanks will be utilized during the verification sampling.

4.1.3.3 Rinsate Blanks

Rinsate blanks are obtained under representative field conditions by running analyte-free deionized water through sample collection equipment after decontamination, and placing it in the appropriate sample containers for analysis. These samples are used to assess the effectiveness of decontamination procedures. Rinsate blanks will be prepared at the rate of one per day and will be analyzed for the same parameters as the related samples.

4.1.3.4 Field Blanks

Field blanks are obtained by sampling the potable and analyte-free water used for decontamination during the field investigation. Field blanks are used to determine if the source waters used for decontamination

could potentially contribute to sample contamination. Per Navy request, field blank analysis will not be performed in this sampling event.

4.2 SAMPLING PROCEDURES

Field sampling will be conducted in accordance with Section 3.1 of this document and the Halliburton NUS SOPs in Appendix B. Allowable sample holding times and preservation requirements are shown in Table 4-1.

4.3 SAMPLE CUSTODY

The custody of field samples will be maintained, recorded and tracked in accordance with Halliburton NUS SOPs in Appendix B.

4.4 CALIBRATION PROCEDURES

Monitoring equipment which requires calibration will only be required if entrance into a confined area is required. This is not expected to occur with this project. Field equipment normally requiring calibration will be calibrated and operated in accordance with the manufacturer's instructions and manuals. A log will be kept documenting the calibration results for each field instrument.

Calibration procedures for laboratory equipment used in the analysis of environmental samples will be performed in accordance with NEESA requirements (i.e., CLP requirements for NEESA Level D).

4.5 ANALYTICAL PROCEDURES

Environmental samples collected during the field investigations covered by this Post-Removal Action Verification Field Sampling Plan will be analyzed by a laboratory previously approved by NEESA. The analytical procedures will conform to NEESA quality Level D as described in Section 4.1.1. Data validation will be performed by Halliburton NUS personnel.

4.6 DATA REDUCTION, VALIDATION, AND REPORTING

Data reduction, validation, and reporting will be conducted in accordance with Region III Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses, March 1990.

TABLE 4-1

FIELD QUALITY CONTROL SAMPLE SUMMARY
REMOVAL ACTION
INDIAN HEAD, MARYLAND

Media	Analysis	Quantity of Field Samples	Field Duplicates (1 per 10 samples)	Equipment Rinsate Blanks (1 per day)	Total Quantity
Post-Removal Solids Samples	Total Mercury	21[1]	3	3	27

[1] Initial sampling will occur at seven transects, three samples from each transect.

4.7 INTERNAL QUALITY CONTROL

Section 4.1.3 discussed the types and frequency of quality control samples that will be prepared during the field investigation activities. The quantities of the various types of the QA/QC samples are shown in Table 4-2.

Laboratory analyses will be conducted in accordance with CLP SOW ILM02.1.

4.8 PERFORMANCE AND SYSTEM AUDITS

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

- The FOL will supervise and check on a daily basis that the equipment is thoroughly decontaminated, samples are collected and handled properly, and the field work is accurately and neatly documented.

- The data validator will review the data packages submitted by the laboratory. The data validator will check that the data was obtained through the approved methodology, that the appropriate level of QC effort and reporting was conducted, that holding times were met, and that the results are in conformance with the QC criteria. On the basis of these factors, the data validator will evaluate the data quality and limitations.

- The project manager will oversee the FOL and data validator, and check that management of the acquired data proceeds in an organized and expeditious manner.

- A formal audit of the field sampling procedures may be conducted in addition to the auditing that is an inherent part of the daily project activities. The auditors will check that sample collection, sample handling, decontamination protocols, and instrument calibration and use are in accordance with the approved project SOPs. The auditors will also check that the field documentation logs and chain-of-custody forms are being filled out properly.

TABLE 4-2

SUMMARY OF ANALYSIS, BOTTLE REQUIREMENTS, PRESERVATION REQUIREMENTS AND HOLDING TIMES INDIAN HEAD, MARYLAND

Media	Analysis	Number of Samples	Number of Containers per Sample	Container Type	Preservation Requirements	Holding Times*
Sediment/Soil	Mercury (Total Recoverable)	24	1	4-oz. wide-mouth glass jar	Cool to 4°C	28 days
Water Samples/Equipment Rinsate Blanks	Mercury (Total Recoverable)	4	1	500-mL polyethylene bottle	Cool to 4°C; pH <2; HNO_3	28 days

* Samples are to be submitted for 48-hour turnaround analysis.

4.9 PREVENTATIVE MAINTENANCE

Halliburton NUS has established a program for the maintenance of field equipment to ensure the availability of equipment in good working order when and where it is needed. This program consists of the following elements:

- The equipment manager keeps an inventory of the equipment in terms of items (model and serial number) quantity and condition. Each item of equipment is signed out when in use, and its operating condition and cleanliness checked upon return.

- The equipment manager conducts routine checks on the status of equipment and is responsible for the stocking of spare parts and equipment readiness.

- The equipment manager maintains the equipment manual library and trains field personnel in the proper use and care of equipment.

- The FOL is responsible for working with the equipment manager to make sure that the equipment is tested, cleaned, charged, and calibrated in accordance with the manufacturer's instructions before being taken to the job site.

4.10 DATA ASSESSMENT PROCEDURES

4.10.1 Representativeness, Accuracy, and Precision

All data generated in the investigation will be assessed for its representativeness, accuracy, and precision. The completeness of the data will also be assessed by comparing the valid acquired data to the project objectives to see that these objectives are being addressed and met. The specific procedures used to determine data precision, accuracy, and completeness will be provided in the analytical data packages and data validation reports. Accuracy will be determined using laboratory spiked samples and laboratory method blanks.

The representativeness of the data will be assessed by determining if the data are consistent with known or anticipated chemical conditions and accepted principles. Field measurements will be checked for completeness of procedures and documentation of procedures and results.

Precision and accuracy will be determined using replicate samples, blanks and spiked samples. PARCC parameters are addressed in more detail in Section 4.1.2.

4.10.2 Validation

Data validation is addressed in Section 4.6.

4.10.3 Data Evaluation

The evaluation of the data collected during the field investigation will include a comparison of cleanup goals and chemical concentrations in soil samples collected from the field. Evidence of the presence of contamination at concentrations greater than the cleanup goals will indicate the need for additional excavation and removal.

4.11 CORRECTIVE ACTION

The QA program will enable problems to be identified, controlled, and corrected. Potential problems may involve nonconformance with the SOPs and/or analytical procedures established for the project or other unforeseen difficulties. Any person identifying an unacceptable condition will notify the project manager. The project manager, with the assistance of the project QA/QC officer, will be responsible for developing and initiating appropriate corrective action and verifying that the correction action has been effective. Corrective actions may include resampling and/or reanalysis of samples or modifying project procedures. If warranted by the severity of the problem (for example, if a change in the approved work plan is required), the Navy will be notified in writing and their approval will be obtained prior to implementing any change. Additional work that is dependent on a nonconforming activity will not be performed until the source of the problem has been addressed.

4.12 QUALITY ASSURANCE REPORTS/DOCUMENTS

A bound/weatherproof field notebook shall be maintained by the SSO, and by the FOL. The FOL or designee shall record all information related to sampling or field activities. This information may include sampling time, weather conditions, unusual events, field measurements, description of photographs, etc. The site logbook maintained by the FOL will contain a summary of the day's activities and will reference the field notebooks when applicable.

Custody of samples must be maintained and documented at all times. Chain-of-custody begins with the collection of the samples in the field. Section 4.3 addresses the topic of chain-of-custody.

At the completion of field activities, the FOL shall submit to the Project Manager all field records, data, field notebooks, logbooks, chain-of-custody receipts, sample log sheets, daily activities records, weekly field summary reports, etc. The Project Manager shall ensure that these materials are entered into the project file.

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

- The FOL notifies the Project Manager of the need for the change.
- If necessary, the Project Manager will discuss the change with the pertinent individuals (e.g., Navy personnel) and will provide a verbal approval or denial to the FOL for the proposed change.
- The FOL will document the change on a Field Modification Record and forward the form to the Project Manager at the earliest convenient time (e.g., end of the workweek).
- The Project Manager will sign the form and distribute copies to the Program Manager, Quality Assurance Manager, Field Operations Leader, and the project file.
- A copy of the completed Field Modification Record form will also be attached to the field copy of the affected document (i.e., Work Plan and Sampling and Analysis Plan).

5.0 SITE MANAGEMENT PLAN

Halliburton NUS will perform this project with support from the Navy. This section of the Post Removal Action Verification Field Sampling Plan describes the site organization, Navy contacts and support, and a project contingency plan.

5.1 PROJECT ORGANIZATION

The project organization is shown on Figure 5-1. The project will be staffed with personnel from the Pittsburgh office of Halliburton NUS. Support personnel from other offices may be utilized on an as-needed basis.

Mr. Anthony P. Klimek, P.E. is the project manager for CTO 99 and will be the primary point of contact. He manages the day-to-day aspects of this project and is responsible for cost and schedule control as well as technical performance. Mr. Klimek is a registered professional engineer in the state of Maryland and has more than 14 years of experience on civil and environmental engineering projects.

Mr. Robert Simcik is the project engineer. He provides technical continuity through development of the contract drawings and specifications, field sampling plan, and field work. Mr. Simcik will act as the Field Operations Leader (FOL).

As shown on Figure 5-1, other technical expertise will be provided as required by the project.

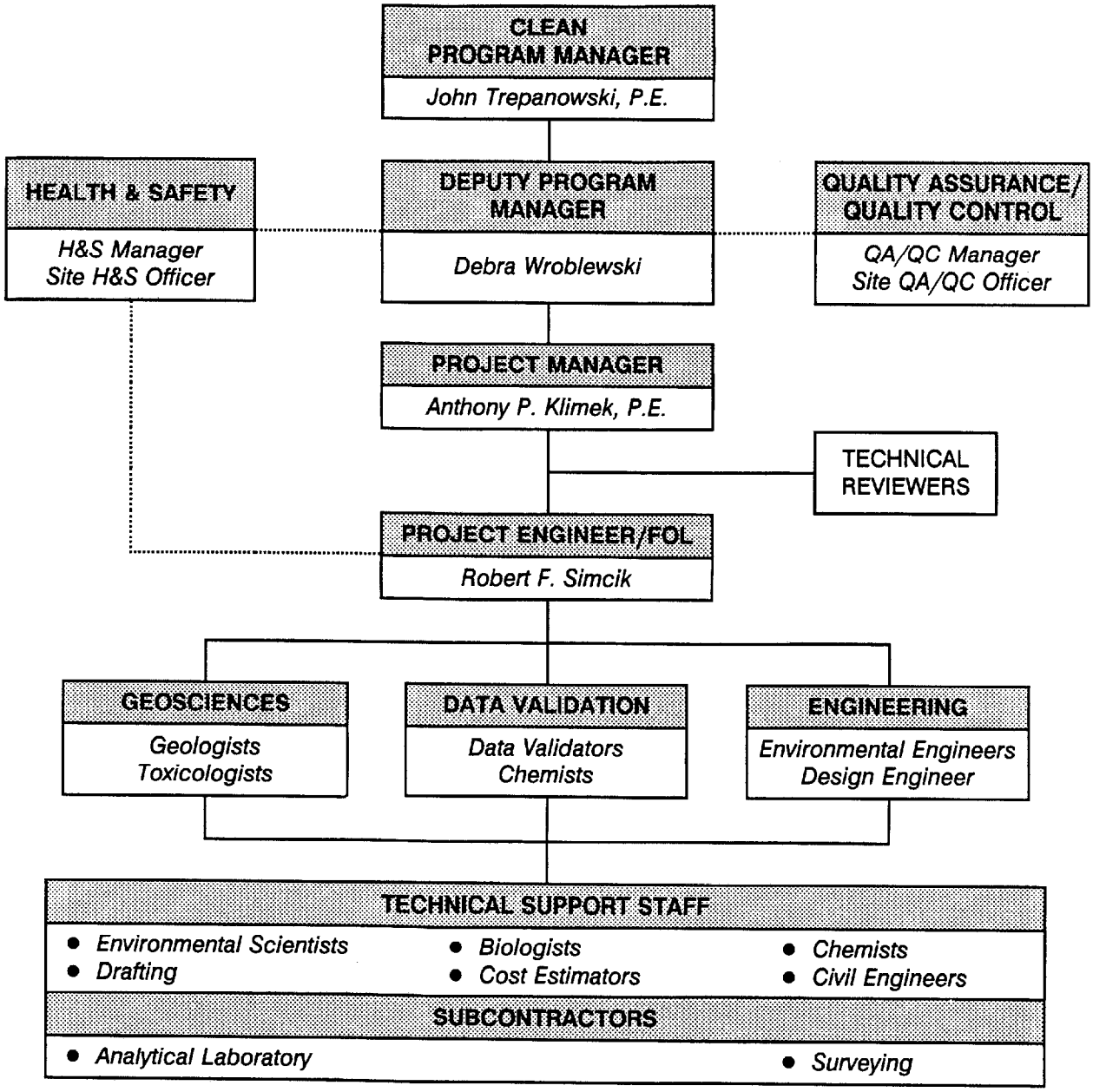
5.2 NAVY SUPPORT

Mr. Shawn Phillips will be the EFA Chesapeake Remedial Project Manager (RPM). He will be the primary Navy point-of-contact for the project. All project activities, including reporting and field activities, will be coordinated through Mr. Phillips. Any changes in scope will be approved through Mr. Phillips prior to implementation.

Mr. Allen Wilson will be the EFA Chesapeake Engineer-in-charge. He will review the technical aspects of the project and be the primary contact on the technical production of this project.

FIGURE 5-1

PROJECT ORGANIZATION



Mr. Shawn Jorgensen will be the primary point of contact at the NSWC. All field work will be coordinated through Mr. Jorgensen; he will notify appropriate NSWC personnel (security, etc.).

Mr. James Behrend will be the resident officer in charge of construction (ROICC) at the NSWC-Indian Head. He will be responsible for the day to day activities associated with the implementation of this project.

Mr. Phillips may be contacted at the following address:

Mr. Shawn Phillips, Code 18
Engineering Field Activity, Chesapeake
Naval Facilities Engineering Command
Washington Naval Yard, Building 212
Washington, D.C. 20374-2121
(202) 433-3760
Fax: (202) 433-6193

Mr. Wilson may be contacted at the following address:

Mr. Allen Wilson, Code 403
Engineering Field Activity, Chesapeake
Naval Facilities Engineering Command
Washington Naval Yard, Building 212
Washington, D.C. 20374-2121
(202) 433-3318
Fax: (202) 433-6202

Mr. Jorgensen may be contacted at the following address:

Mr. Shawn Jorgensen, Code 0965
Indian Head Division
Naval Surface Warfare Center, Building D28
Indian Head, MD 20640-5000
(301) 743-6745

Mr. Behrend may be contacted at the following address:

Mr. James Behrend, Code 09RB3
Indian Head Division
Naval Surface Warfare Center, Building 351
Indian Head, MD 20640-5000
(301) 743-4112

5.3 CONTINGENCY PLAN

In the event major problems are encountered during field activities, the Halliburton NUS Project Manager will be notified immediately, followed by the Halliburton NUS Point of Contact and the EFA Chesapeake RPM. Halliburton NUS will then develop an alternate course of action and discuss it with the Navy. All alternate courses of action that impact the project and schedule and budget will be approved through the NSWC Point of Contact and the EFA Chesapeake RPM before being enacted.

REFERENCES

Initial Assessment Study, May 1983.

NACIP Confirmation Study, September 1985

NIRP Feasibility Study and Remedial Design, Task 2, Supplemental Site Characterization Report, December 1987.

NIRP Feasibility Study and Remedial Design, Task 3, Remedial Alternatives Evaluation Report, February 1988.

Draft Data Evaluation Report, November 1989.

Draft Mercury Speciation Study Report, July 1990.

Technical Memoranda, Draft Final, May 1991.

Interim Remedial Action, Final Bid Specification Document, June 1991.

Biomonitoring Work Plan, Draft Final, January 1992.

Engineering Evaluation and Cost Analysis Report, March 1992.

AFSP, Final, August 1993.

APPENDIX A

FORMS FOR FIELD ACTIVITIES

SAMPLE LOG SHEET

HALLIBURTON NUS
Environmental Corporation

- ☐ Surface Soil
- ☐ Subsurface Soil
- ☐ Sediment
- ☐ Lagoon / Pond
- ☐ Other _____

Page _____ of _____

Case # _____

By _____

Project Site Name _____ Project Site Number _____

NUS Source No. _____ Source Location _____

Sample Method:	Composite Sample Data		
	Sample	Time	Color / Description
Depth Sampled:			
Sample Date & Time:			
Sampled By:			
Signature(s):			
Type of Sample ☐ Low Concentration ☐ High Concentration ☐ Grab ☐ Composite ☐ Grab - Composite			

Sample Data	
Color	Description: (Sand, Clay, Dry, Moist, Wet, etc.)

Analysis:	PRESERV :

Observations / Notes

	Organic	Inorganic
Traffic Report #		
Tag #		
AB #		
Date Shipped		
Time Shipped		
Lab		
Volume		

HALLIBURTON NUS

CHAIN OF CUSTODY RECORD

PROJECT NO.:	SITE NAME:
SAMPLERS (SIGNATURE):	

STATION NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CON-TAINERS							REMARKS

RELINQUISHED BY (SIGNATURE):	DATE/TIME:	RECEIVED BY (SIGNATURE):	RELINQUISHED BY (SIGNATURE):	DATE/TIME:	RECEIVED BY (SIGNATURE):
RELINQUISHED BY (SIGNATURE):	DATE/TIME:	RECEIVED BY (SIGNATURE):	RELINQUISHED BY (SIGNATURE):	DATE/TIME:	RECEIVED BY (SIGNATURE):
RELINQUISHED BY (SIGNATURE):	DATE/TIME:	RECEIVED FOR LABORATORY BY (SIGNATURE):	DATE/TIME:	REMARKS:	

NUS 440 34 0484

APPENDIX B

HALLIBURTON NUS STANDARD OPERATING PROCEDURES

APPENDIX B

HALLIBURTON NUS STANDARD OPERATING PROCEDURES (SOP's)

SA-1.2, Surface Water and Sediment Sampling

SA-6.1, Sample Identification and Chain-of-Custody

SA-6.2, Sampling Packaging and Shipping

SA-6.3, Site Logbook

SF-1.1, On-Site Water Quality Testing

SF-1.2, Sample Preservation

SF-2.3, Deconntamination of Chemical Sampling and Field Analytical Equipment

NUS CORPORATION ENVIRONMENTAL MANAGEMENT GROUP	STANDARD OPERATING PROCEDURES	Number SA-1.2	Page 1 of 10
		Effective Date 05/04/90	Revision 2
		Applicability EMG	
		Prepared Earth Sciences	
Subject SURFACE WATER AND SEDIMENT SAMPLING		Approved D. Senovich	

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7.0 ATTACHMENTS

1.0 PURPOSE

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

2.0 SCOPE

The information presented in this guideline is generally applicable to all environmental sampling of surface waters (Section 5.3) and aquatic sediments (Section 5.4), except where the analyte(s) may interact with the sampling equipment. The collection of concentrated sludges or hazardous waste samples from disposal or process lagoons often requires methods, precautions and equipment different from those described herein.

3.0 GLOSSARY

Environmental Sample - low concentration sample typically collected offsite and not requiring DOT hazardous waste labeling or CLP handling as a high concentration sample.

Hazardous Waste Sample - medium to high concentration sample (e.g., source material, sludge, leachate) requiring DOT labeling and CLP handling as a high concentration sample.

4.0 RESPONSIBILITIES

Field Operations Leader - has overall responsibility for the correct implementation of surface water and sediment sampling activities, including review of the sampling plan with, and any necessary training of, the sampling technician(s). The actual collection, packaging, documentation (sample label and log sheet, chain-of-custody record, CLP traffic reports, etc.) and initial custody of samples will be the responsibility of the sampling technician(s).

5.0 PROCEDURES

5.1 INTRODUCTION

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

Representativeness is a qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important quality not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been taken. Regardless of scrutiny and quality control applied during laboratory analyses, reported data are not better than the confidence that can be placed in the representativeness of the samples.

5.2 DEFINING THE SAMPLING PROGRAM

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

5.2.1 Sampling Program Objectives

The objective of surface water sampling is to determine the surface water quality entering, leaving or remaining within the site. The scope of the sampling program must consider the sources and potential pathways for transport of contamination to or in a surface water body. Sources may include point sources (leaky tanks, outfalls, etc.) or nonpoint sources (e.g., spills). The major pathways for surface water contamination (not including airborne deposition are: (a) overland runoff; (b) leachate influx to the waterbody; (c) direct waste disposal (solid or liquid) into the water body; and groundwater flow influx from upgradient. The relative importance of these pathways, and therefore the design of the sampling program, is controlled by the physiographic and hydrologic features of the site, the drainage basin(s) which encompass the site, and the history of site activities.

Physiographic and hydrologic features to be considered include slopes and runoff direction, areas of temporary flooding or pooling, tidal effects, artificial surface runoff controls such as berms or drainage ditches (and when they were constructed relative to site operation), and locations of springs, seeps, marshes, etc. In addition, the obvious considerations such as the location of man-made discharge points to the nearest stream (intermittent or flowing), pond, lake, estuary, etc., shall be considered.

A more subtle consideration in designing the sampling program is the potential for dispersion of dissolved or sediment-associated contaminants away from the source. The dispersion could lead to a more homogeneous distribution of contamination at low or possibly non-detectable concentrations. Such dispersion does not, however, always readily occur. For example, obtaining a representative sample of contamination from a main stream immediately below an outfall or a tributary is difficult because the inflow frequently follows a stream bank with little lateral mixing for some distance. Sampling alternatives to overcome this situation are: (1) move the site far enough downstream to allow for adequate mixing, or (2) collect integrated samples in a cross section. Also, nonhomogeneous distribution is a particular problem with regard to sediment-associated contaminants, which may accumulate in low-energy environments (coves, river bends, deep spots, or even behind boulders) near or distant from the source while higher-energy areas (main stream channels) near the source may show no contaminant accumulation.

The distribution of particulates within a sample itself is an important consideration. Many organic compounds are only slightly water soluble and tend to be absorbed by particulate matter. Nitrogen, phosphorus, and the heavy metals may also be transported by particulates. Samples will be collected with a representative amount of suspended material; transfer from the sampling device shall include transferring a proportionate amount of the suspended material.

5.2.2 Location of Sampling Stations

Accessibility is the primary factor affecting sampling costs. The desirability and utility of a sample for analysis and description of site conditions must be balanced against the costs of collection as controlled by accessibility. Bridges or piers are the first choice for locating a sampling station on a stream because bridges provide ready access and also permit the sampling technician to sample any point across the stream. A boat or pontoon (with an associated increase in cost) may be needed to sample locations on lakes and reservoirs, as well as those on larger rivers. Frequently, however, a boat will take longer to cross a water body and will hinder manipulation of the sampling equipment. Wading for samples is not recommended unless it is known that contaminant levels are low so that skin contact will not produce adverse health effects. This provides a built in margin of safety in the event that wading boots or other protective equipment should fail to function properly. If it is necessary to wade into the water body to obtain a sample, the sampler shall be careful to minimize disturbance of bottom sediments and must enter the water body downstream of the sampling location. If necessary, the sampling technician shall wait for the sediments to settle before taking a sample.

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

Under ideal and uniform contaminant dispersion conditions in a flowing stream, the same concentrations of each would occur at all points along the cross section. This situation is most likely downstream of areas of high turbulence. Careful site selection is needed in order to ensure, as nearly as possible, that samples are taken where uniform flow or deposition and good mixing conditions exist.

The availability of streamflow and sediment discharge records can be an important consideration in choosing sampling sites in streams. Streamflow data in association with contaminant concentration data are essential for estimating the total contaminant loads carried by the stream. If a gaging station is not conveniently located on a selected stream, the project hydrologist shall explore the possibility of obtaining streamflow data by direct or indirect methods.

5.2.3 Frequency of Sampling

The sampling frequency and the objectives of the sampling event will be defined by the work plan. For single-event site- or area-characterization sampling, both bottom material and overlying water samples shall be collected at the specified sampling stations. If valid data are available on the distribution of the contaminant between the solid and aqueous phases it may be appropriate to sample only one phase, although this is not often recommended. If samples are collected primarily for monitoring purposes, consisting of repetitive, continuing measurements to define variations and trends at a given location, water samples shall be collected at a pre-established and constant interval as specified in the work plan (often monthly or quarterly) and during droughts and floods. Samples of bottom material shall be collected from fresh deposits at least yearly, and preferably during both spring and fall seasons.

The variability in available water-quality data shall be evaluated before deciding on the number and collection frequency of samples required to maintain an effective monitoring program.

5.3 SURFACE WATER SAMPLE COLLECTION

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

Methods for sampling streams, rivers, outfalls and drainage features at a single point vary from the simplest of hand sampling procedures to the more sophisticated multipoint sampling techniques known as the equal-width-increment (EWI) method or the equal-discharge-increment (EDI) methods (see below).

Samples from different depths or cross-sectional locations in the water course taken during the same sampling episode shall be composited. However, samples collected along the length of the watercourse or at different times may reflect differing inputs or dilutions and therefore shall not be composited. Generally, the number and type of samples to be taken depend on the river's width, depth, discharge and on the suspended sediment the river's transports. The greater number of individual points that are sampled, the more likely that the composite sample will truly represent the overall characteristics of the water.

In small streams less than about 20 feet wide, a sampling site can generally be found where the water is well-mixed. In such cases, a single grab sample taken at mid-depth in the center of the channel is adequate to represent the entire cross-section.

For larger streams, at least one vertical composite shall be taken with one sample each from just below the surface, at mid-depth, and just above the bottom. The measurement of DO, pH, temperature, conductivity, etc., shall be made on each aliquot of the vertical composite and on the composite itself. For rivers, several vertical composites shall be collected.

5.3.2 Lakes, Ponds and Reservoirs

Lakes, ponds, and reservoirs have as much greater tendency to stratify than rivers and streams. The relative lack of mixing requires that more samples be obtained.

The number of water sampling sites on a lake, pond, or impoundment will vary with the size and shape of the basin. In ponds and small lakes, a single vertical composite at the deepest point may be sufficient. Similarly, the measurement of DO, pH, temperature, etc., is to be conducted on each aliquot of the vertical composite. In naturally-formed ponds, the deepest point may have to be determined empirically; in impoundments, the deepest point is usually near the dam.

In lakes and larger reservoirs, several vertical composites shall be composited to form a single sample. These verticals are often taken along a transect or grid. In some cases, it may be of interest to form separate composites of epilimnetic and hypolimnetic zones. In a stratified lake, the epilimnion is the thermocline which is exposed to the atmosphere. The hypolimnion is the lower, "confined" layer which is only mixed with the epilimnion and vented to the atmosphere during seasonal "overturn" (when density stratification disappears). These two zones may thus have very different concentrations of contaminants if input is only to one zone, if the contaminants are volatile (and therefore vented from the epilimnion but not the hypolimnion), or if the epilimnion only is involved in short-term flushing (i.e., inflow from or outflow to shallow streams). Normally, however, a composite consists of several verticals with samples collected at various depths.

In lakes with irregular shape and with bays and coves that are protected from the wind, separate composite samples may be needed to adequately represent water quality since it is likely that only poor mixing will occur. Similarly, additional samples are recommended where discharges, tributaries, land use characteristics, and other such factors are suspected of influencing water quality.

Many lake measurements are now made in-situ using sensors and automatic readout or recording devices. Single and multiparameter instruments are available for measuring temperature, depth, pH, oxidation-reduction potential (ORP), specific conductance, dissolved oxygen, some cations and anions, and light penetration.

5.3.3 Estuaries

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

- Mixed estuary - characterized by the absence of a vertical halocline (gradual or no marked increase in salinity in the water column) and a gradual increase in salinity seaward. Typically this type of estuary is shallow and is found in major freshwater sheetflow areas. Being well mixed, the sampling locations are not critical in this type of estuary.

- Salt wedge estuary - characterized by a sharp vertical increase in salinity and stratified freshwater flow along the surface. In these estuaries the vertical mixing forces cannot override the density differential between fresh and saline waters. In effect, a salt wedge tapering inland moves horizontally, back and forth, with the tidal phase. If contamination is being introduced into the estuary from upstream, water sampling from the salt wedge may miss it entirely.

- Oceanic estuary - characterized by salinities approaching full strength oceanic waters. Seasonally, freshwater inflow is small with the preponderance of the fresh-saline water mixing occurring near, or at, the shore line.

Sampling in estuarine areas is normally based upon the tidal phases, with samples collected on successive slack tides (i.e. when the tide turns). Estuarine sampling programs shall include vertical salinity measurements at 1 to 5 foot increments coupled with vertical dissolved oxygen and temperature profiles.

5.3.4 Surface Water Sampling Equipment

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

- Open tube
- Dip sampler
- Hand pump
- Kemmerer
- Depth-Integrating Sampler

The dip sampler and the weighted bottle sampler are used most often.

The criteria for selecting a sampler include:

- Disposable and/or easily decontaminated
- Inexpensive (if the item is to be disposed of)
- Ease of operation
- Nonreactive/noncontaminating - Teflon-coating, glass, stainless steel or PVC sample chambers are preferred (in that order)

Each sample (grab or each aliquot collected for compositing) shall be measured for:

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

as soon as it is recovered. These analyses will provide information on water mixing/stratification and potential contamination.

5.3.4.1 Dip Sampling

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

5.3.4.2 Weighted Bottle Sampling

A grab sample can also be taken using a weighted holder that allows a sample to be lowered to any desired depth, opened for filling, closed, and returned to the surface. This allows discrete sampling with depth. Several of these samples can be combined to provide a vertical composite. Alternatively, an open bottle can be lowered to the bottom and raised to the surface at a uniform rate so that the bottle collects sample throughout the total depth and is just filled on reaching the surface. The resulting sample using either method will roughly approach what is known as a depth-integrated sample.

A closed weighted bottle sampler consists of a stopped glass or plastic bottle, a weight and/or holding device, and lines to open the stopper and lower or raise the bottle. The procedure for sampling is:

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

5.3.4.3 Kemmerer

If samples are desired at a specific depth, and the parameters to be measured do not require a Teflon coated sampler, a standard Kemmerer sampler may be used. The Kemmerer sampler is a brass, stainless steel or acrylic cylinder with rubber stoppers that leave the ends open while being lowered in a vertical position to allow free passage of water through the cylinder. "Messenger" is sent down the line when the sampler is at the designated depth, to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill sample bottles.

5.3.5 Surface Water Sampling Techniques

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

- The most representative samples are obtained from mid-channel at 0.6 stream depth in a well-mixed stream.
- Even though the containers used to obtain the samples are previously laboratory cleaned, it is suggested that the sample container be rinsed at least once with the water to be sampled before the sample is taken.
- For sampling running water, it is suggested that the farthest downstream sample be obtained first and that subsequent samples be taken as one works upstream. Work from zones suspected of low contamination to zones of high contamination.
- To sample a pond or other standing body of water, the surface area may be divided into grids. A series of samples taken from each grid is combined into one sample, or several grids are selected at random.
- Care should be taken to avoid excessive agitation of the water that results in the loss of volatile constituents.
- When obtaining samples in 40 ml septum vials for volatile organics, analysis, it is important to exclude any air space in the top of the bottle and to be sure that the Teflon liner faces in after the bottle is filled and capped. The bottle can be turned upside down to check for air bubbles.
- Do not sample at the surface, unless sampling specifically for a known constituent which is immiscible and on top of the water. Instead, the sample container should be inverted, lowered to the approximate depth, and held at about a 45-degree angle with the month of the bottle facing upstream.

5.4 SEDIMENT SAMPLING

5.4.1 General

Sediment samples are usually collected at the same verticals at which water samples were collected. If only one sediment sample is to be collected, the site shall be approximately at the center of water body. Generally, the coarser grained sediments are deposited near the headwaters of the reservoir. Bed sediments near the center will be composed of fine-grained materials which may, because of their lower porosity and greater surface area available for adsorption, contain greater concentrations

of contaminants. The shape, flow pattern, bathometry (depth distribution), and water circulation patterns must all be considered when selecting sediment sampling sites. In streams, areas likely to have sediment accumulation (bends, behind islands or boulders, quiet shallow areas or very deep, low-velocity areas) shall be sampled while areas likely to show net erosion (high-velocity, turbulent areas) and suspension of fine solid materials shall be avoided.

Chemical constituents associated with bottom material may reflect an integration of chemical and biological processes. Bottom samples reflect the historical input to streams, lakes, and estuaries with respect to time, application of chemicals, and land use. Bottom sediments (especially fine-grained material) may act as a sink or reservoir for adsorbed heavy metals and organic contaminants (even if water column concentrations are below detection limits). It is therefore important to minimize the loss of low-density "fines" during any sampling process.

5.4.2 Sampling Equipment and Techniques

A bottom-material sample may consist of a single scoop or core or may be a composite of several individual samples in the cross section. Sediment samples may be obtained using on-shore or off-shore techniques.

When boats are used for sampling, life preservers must be provided and two individuals must undertake the sampling. An additional person shall remain on-shore in visual contact at all times.

The following samplers may be used to collect bottom materials:

- Scoop sampler
- Dredge samplers

5.4.2.1 Scoop Sampler

A scoop sampler consists of a pole to which a jar or scoop is attached. The pole may be made of bamboo, wood or aluminum and be either telescoping or of fixed length. The scoop or jar at the end of the pole is usually attached using a clamp.

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

5.4.2.2 Dredges

Dredges are generally used to sample sediments which cannot easily be obtained using coring devices (i.e., coarse-grained or partially-cemented materials) or when large quantities of materials are required. Dredges generally consist of a clam shell arrangement of two buckets. The buckets may either close upon impact or be activated by use of a messenger. Most dredges are heavy (up to several hundred pounds) and require use of a winch and crane assembly for sample retrieval. There are three major types of dredges: Peterson, Eckman and Ponar dredges.

The Peterson dredge is used when the bottom is rocky, in very deep water, or when the flow velocity is high. The dredge shall be lowered very slowly as it approaches bottom, because it can force out and miss lighter materials if allowed to drop freely.

The Eckman dredge has only limited usefulness. It performs well where bottom material is unusually soft, as when covered with organic sludge or light mud. It is unsuitable, however, for sandy, rocky, and hard bottoms and is too light for use in streams with high flow velocities.

The Ponar dredge is a Peterson dredge modified by the addition of side plates and a screen on the top of the sample compartment. The screen over the sample compartment permits water to pass through the sampler as it descends thus reducing the "shock wave" and permitting direct access to the secured sample without opening the closed jaws. The Ponar dredge is easily operated by one person in the same fashion as the Peterson dredge. The Ponar dredge is one of the most effective samplers for general use on all types of substrates. Access to the secured sample through the covering screens permits subsampling of the secured material with coring tubes or Teflon scoops, thus minimizing the change of metal contamination from the frame of the device.

6.0 REFERENCES

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

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

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

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

7.0 ATTACHMENTS

None.

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1.0 PURPOSE

This purpose of this procedure is to provide information on chain-of-custody procedures to be used under the NUS Program.

2.0 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 all samples collected for on-site or off-site analysis (chemical or geotechnical) during program activities. 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 and its introduction as evidence. 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.

3.0 GLOSSARY

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. A Chain-of-Custody Record Form is a controlled document, provided by the regional office of EPA.

The chain-of-custody form is a two-page carbon-copy type form. The original form accompanies the samples during shipment, and the pink carbon-copy is retained in the project file.

Controlled Document - A consecutively-numbered form released by EPA or Program Management Office (PMO) for use on a particular work assignment. All unused forms must be returned or accounted for at the conclusion of the assignment.

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 your custody if:

- It is in your actual possession.
- It is in your view, after being in your physical possession.
- It was in your physical possession and then you 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.

4.0 RESPONSIBILITIES

Field Operations Leader - Responsible for determining that chain-of-custody procedures are implemented up to and including release to the shipper.

Field Samplers - Responsible for initiating the Chain-of-Custody Record and maintaining custody of samples until they are relinquished to another custodian, to the shipper, or to the common carrier.

Remedial Investigation Leader - Responsible for determining that chain-of-custody procedures have been met by the sample shipper and analytical laboratory.

5.0 PROCEDURES

5.1 OVERVIEW

The term "chain-of-custody" refers to procedures which ensure that evidence presented in a court of law is what it is represented to be. The chain-of-custody procedures track the evidence from the time and place it is first obtained to the courtroom and, secondly, provide security for the evidence as it is moved and/or passes 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.

5.2 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.

5.2.1 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 Plan. Each sample container is identified by a sample label (see Attachment B). Sample labels are provided by the PMO. The information recorded on the sample label includes:

- **Project:** EPA Work Assignment Number (can be obtained from the Sampling Plan).
- **Station Location:** The unique sample number identifying this sample (can be obtained from the Sampling Plan).
- **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.

- **Concentration:** The expected concentration (i.e., low, medium, high).
- **Sample Type:** Grab or composite.
- **Preservation:** Type of preservation added and pH levels.
- **Analysis:** VOA, BNAs, PCBs, pesticides, metals, cyanide, other.
- **Sampled By:** Printed name of the sampler.
- **Case Number:** Case number assigned by the Sample Management Office.
- **Traffic Report Number:** Number obtained from the traffic report labels.
- **Remarks:** Any pertinent additional information.

Using just 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 analysis from knowing the identity of the site (e.g., if the laboratory is part of an organization that has performed previous work on the site).

5.2.2 Sample Identification Tag

A Sample Identification Tag (Attachment F) must also be used for samples collected for CLP (Contract Laboratory Program) analysis. The Sample Identification Tag is a w[illegible]e, waterproof paper label, approximately 3-by-6 inches, with a reinforced eyelet, and string or w[illegible] or attachment to the neck of the sample bottle. The Sample Tag is a controlled document, and i[illegible]ovided by the regional EPA office. Following sample analysis, the Sample Tag is retained by the la[illegible]atory as evidence of sample receipt and analysis.

The following information is recorded on the tag:

- **Project Code:** Work Assignment Number.
- **Station Number:** The middle portion of the Station Location Number, (between the hyphens).
- **Month/Day/Year:** Same as Date on Sample Label.
- **Time:** Same as Time on Sample Label.
- **Designate - Comp/Grab:** Composite or grab sample.
- **Station Location:** Same as Station Location on Sample Label.
- **Samplers:** Same as Sampled By on Sample Label.
- **Preservative:** Yes or No.
- **Analyses:** Check appropriate box(es).

- **Remarks:** Same as Remarks on Sample Label (make sure the Case Number and Traffic Report numbers are recorded).

- **Lab Sample Number:** For laboratory use only.

The tag is then tied around the neck of the sample bottle.

If the sample is to be split, it is aliquoted into similar sample containers. Identical information is completed on the label attached to each split.

Blank, duplicate, or field spike samples shall not be identified as such on the label, as they may compromise the quality control function. Sample blanks, duplicates, spikes, and splits are defined in Procedure SA-6.6.

5.3 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 of.

5.3.1 Field Custody Procedures

- Samples are collected as described in the site-specific Sampling Plan. Care must be taken to record precisely the sample location and to ensure that the sample number on the label matches the sample log sheet and 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.

- 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 because a ballpoint pen would not function in freezing weather.

5.3.2 Transfer of Custody and Shipment

Samples are accompanied by a Chain-of-Custody Record Form. Chain-of-Custody Record Forms used in EPA Regions I-IV are shown in Attachments A through D. The appropriate form shall be obtained from the EPA Regional Office. 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 follows:

- Enter header information (project number, samplers, and project name -- project name can be obtained from the Sampling Plan).

- Sign, date, and enter the time under "Relinquished by" entry.

- Enter station number (the station number is the middle portion of the station location number, between the hyphens).
- Check composite or grab sample.
- Enter station location number (the same number as the station location on the tag and label).
- Enter the total number of containers per station number and the type of each bottle.
- Enter either the inorganic traffic report number, the organic traffic report number, or the SAS number for each station number in the remarks column.
- Enter the tag number from the bottom of the sample identification tag in the remarks column for each station location.
- Make sure that the person receiving the sample signs the "Received by" entry, or enter the name of the carrier (e.g., UPS, Federal Express) under "Received by." Receiving laboratory will sign "Received for Laboratory by" on the lower line and enter the date and time.
- Enter the bill-of-lading or Federal Express airbill number under "Remarks," in the bottom right corner, if appropriate.
- Place the original (top, signed copy) of the Chain-of-Custody Record Form in the appropriate sample shipping package. Retain the pink 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 G 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 are provided by ZPMO on an as-needed basis.
- Place the seal across the shipping container opening so that it would be broken if the container is opened.
- Complete other carrier-required shipping papers.

The custody record is completed using black 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 sample container (enclosed with other documentation in a plastic zip-lock bag). As long as custody forms are sealed inside the sample container and the custody seals are intact, commercial carriers are not required to sign off on the custody form.

If sent by mail, the package will be registered with return receipt requested. If sent by common carrier or air freight, proper documentation must be maintained.

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.

5.3.3 Receipt for Samples Form

Whenever samples are split with a private party or government agency, a separate Receipt for Samples Record Form is prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the party or agency shall require the signature of a representative of the appropriate party acknowledging receipt of the samples. If a representative is unavailable or refuses to sign, this is noted in the "Received by" space. When appropriate, as in the case where the representative is unavailable, the custody record shall contain a statement that the samples were delivered to the designated location at the designated time. This form must be completed and a copy given to the owner, operator, or agent-in-charge even if the offer for split samples is declined. The original is retained by the Field Operations Leader.

6.0 REFERENCES

U.S. EPA, 1984. User's Guide to the Contract Laboratory Program, Office of Emergency and Remedial Response, Washington, D.C.

7.0 ATTACHMENTS

Attachment A - Chain-of-Custody Record Form for use in Region I
Attachment B - Chain-of-Custody Record Form for use in Region II
Attachment C - Chain-of-Custody Record Form for use in Region III
Attachment D - Chain-of-Custody Record Form for use in Region IV
Attachment E - Sample Label
Attachment F - Sample Identification Tag
Attachment G - Chain-of-Custody Seal

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1.0 PURPOSE

This procedure provides instruction for sample packaging and shipping in accordance with U.S. Department of Transportation (DOT) regulations.

2.0 SCOPE

Samples collected at hazardous waste sites usually have to be transported elsewhere for analysis. This requires that the samples be appropriately preserved to prevent or minimize chemical alteration prior to analysis, and be transported to protect their integrity, as well as to protect against any detrimental effects from leakage or breakage. Regulations for packaging, marking, labeling, and shipping hazardous materials and wastes are promulgated by the U.S. Department of Transportation and described in the Code of Federal Regulations (49 CFR 171 through 177, in particular 172.402h, Packages Containing Samples). In general, these regulations were not intended to cover shipment of samples collected at controlled or uncontrolled hazardous waste sites or samples collected during emergency responses. However, the EPA has agreed through a memorandum of agreement to package, mark, label, and ship samples observing DOT procedures. The information presented here is for general guidance.

This procedure is applicable to all samples taken from uncontrolled hazardous substance sites for analysis at laboratories away from the site.

3.0 GLOSSARY

Carrier - A person or firm engaged in the transportation of passengers or property.

Hazardous Material - A substance or material in a quantity and form which may pose an unreasonable risk to health and safety or property when transported in commerce ("commerce" here to include any traffic or transportation). Defined and regulated by DOT (49 CFR 173.2) and listed in Attachment A of this guideline.

Hazardous Waste - Any substance listed in 40 CFR Subpart D (¥261.20 et seq) or otherwise characterized as ignitable, corrosive, reactive, or EP toxic as specified under 40 CFR Subpart C (¥261.20 et seq) that would be subject to manifest requirements specified in 40 CFR 262. Defined and regulated by EPA.

Marking - Applying the descriptive name, instruction, cautions, weight, or specification marks or combination thereof required to be placed outside containers of hazardous materials.

n.o.i. - Not otherwise indicated.

n.o.s. - Not otherwise specified.

ORM - Other regulated material.

Packaging - The assembly of one or more containers and any other components necessary to assure compliance with the minimum packaging requirements of 49 CFR 174, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, multiunit tank car tanks.

Placard - Color-coded, pictorial sign depicting the hazard class symbol and name to be placed on all four sides of a vehicle transporting certain hazardous materials.

Reportable Quantity (RQ) - A parenthetical note of the form "(RQ-1000/454)" following an entry in the DOT Hazardous Materials table (49 CFR 172.101) indicates the reportable quantity of the substance in pounds and kilograms. If a spill of that amount or more of the substance occurs during transit or storage, a report must be filed with DOT according to ¥171.15-15 concerning hazardous materials incidents reports. If the material spilled is a hazardous waste, a report must always be filed, regardless of the amount, and must include a copy of the manifest. If the RQ notation appears, it must be shown either immediately before or after the proper shipping name on the shipping paper (or manifest). Most shipping papers and manifests will have a column designated "HM" which may be used for this purpose.

4.0 RESPONSIBILITIES

Field Operations Leader or Team Sampling Leader - responsible for determining that samples are properly packaged and shipped.

Sampling Personnel - responsible for implementing the packaging and shipping requirements.

5.0 PROCEDURES

5.1 INTRODUCTION

Samples collected for shipment from a site shall be classified as either environmental or hazardous material (or waste) samples. In general, environmental samples are collected off-site (for example from streams, ponds, or wells) and are not expected to be grossly contaminated with high levels of hazardous materials. On-site samples (for example, soil, water, and materials from drums or bulk storage tanks, obviously contaminated ponds, lagoons, pools, and leachates from hazardous waste sites) are considered hazardous. A distinction must be made between the two types of samples in order to:

- Determine appropriate procedures for transportation of samples. If there is any doubt, a sample shall be considered hazardous and shipped accordingly.
- Protect the health and safety of laboratory personnel receiving the samples. Special precautions are used at laboratories when samples other than environmental samples are received.

5.2 ENVIRONMENTAL SAMPLES

5.2.1 Packaging

Environmental samples may be packaged following the procedures outlined in Section 5.4 for samples classified as "flammable liquids" or "flammable solids." Requirements for marking, labeling, and shipping papers do not apply.

Environmental samples may also be packed without being placed inside metal cans as required for flammable liquids or solids.

- Place sample container, properly identified and with a sealed lid, in a polyethylene bag, and seal the bag.
- Place sample in a fiberboard container or metal picnic cooler which has been lined with a large polyethylene bag.
- Pack with enough noncombustible, absorbent, cushioning materials to minimize the possibility of the container breaking.
- Seal large bag.
- Seal or close outside container.

5.2.2 Marking Labeling

Sample containers must have a completed sample identification tag and the outside container must be marked "Environmental Sample." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT marking or labeling are required.

5.2.3 Shipping Papers

No DOT shipping papers are required. However, the appropriate chain-of-custody forms must be included with the shipment.

5.2.4 Transportation

There are no DOT restrictions on mode of transportation.

5.3 DETERMINATION OF SHIPPING CLASSIFICATION FOR HAZARDOUS MATERIAL SAMPLES

Samples not determined to be environmental samples, or samples known or expected to contain hazardous materials, must be considered hazardous material samples and transported according to the requirements listed below.

5.3.1 Known Substances

If the substance in the sample is known or can be identified, package, mark, label and ship according to the specific instructions for that material (if it is listed) in the DOT Hazardous Materials Table, 49 CFR 172.101.

Unz and Company have published the following steps to help in locating a proper shipping name from the Hazardous Materials Table, 49 CFR 172.101.

1. Look first for the chemical or technical name of the material, for example, ethyl alcohol. Note that many chemicals have more than one technical name, for example, perchloroethylene (not listed in 172.101) is also called tetrachloroethylene (listed 172.101). It may be useful to consult a chemist for all possible technical names a material can have. If your material is not listed by its technical name then. . . . . .

2. Look for the chemical family name. For example, pentyl alcohol is not listed but the chemical family name is: alcohol, n.o.s. (not otherwise specified). If the chemical family name is not listed then. . . . . .

3. Look for a generic name based on end use. For example, Paint, n.o.s or Fireworks, n.o.s. If a generic name based on end use is not listed then. . . . . .

4. Look for a generic family name based on end use, for example, drugs, n.o.s. or cosmetics, n.o.s. Finally, if your material is not listed by a generic family name but you suspect or know the material is hazardous because it meets the definition of one or more hazardous classes, then. . . . . .

5. You will have to go the the general hazard class for a proper shipping name. For example, Flammable Liquid, n.o.s, or Oxidizer, n.o.s.

5.3.2 Unknown Substances

For samples of hazardous substances of unknown content, select the appropriate transportation category according to the DOT Hazardous Materials Classification (Attachment A), a priority system of transportation categories.

The correct shipping classification for an unknown sample is selected through a process of elimination, utilizing Attachment A. Unless known or demonstrated otherwise (through the use of radiation survey instruments), the sample is considered radioactive and appropriate shipping regulations for "radioactive material" followed.

If a radioactive material is eliminated, the sample is considered to contain "Poison A" materials (Attachment B), the next classification on the list. DOT defines "Poison A" as extremely dangerous poisonous gases or liquids of such a nature that a very small amount of gas, or vapor of the liquids, mixed with air is dangerous to life. Most Poison A materials are gases or compressed gases and would not be found in drum-type containers. Liquid Poison A would be found only in closed containers; however,all samples taken from closed drums do not have to be shipped as Poison A, which provides for a "worst case" situation. Based upon information available, a judgment must be made whether a sample from a closed container is a Poison A.

If Poison A is eliminated as a shipment category, the next two classifications are "flammable" or "nonflammable" gases. Since few gas samples are collected, "flammable liquid" would be the next applicable category. With the elimination of radioactive material, Poison A, flammable gas, and nonflammable gas, the sample can be classified as flammable liquid (or solid) and shipped accordingly. These procedures would also suffice for shipping any other samples classified below flammable liquids in the DOT classification table (Attachment A). For samples containing unknown materials, categories listed below flammable liquids/solids on Attachment A are generally not used because showing that these materials are not flammable liquids (or solids) requires flashpoint testing, which may be impractical and possibly dangerous at a site. Thus, unless the sample is known to consist of materials listed as less hazardous than flammable liquid (or solid) on Attachment A, it is considered a flammable liquid (or solid) and shipped as such.

For any hazardous material shipment, utilize the shipping checklist (Attachment C) as a guideline to ensure that all sample-handling requirements are satisfied.

5.4 PACKAGING AND SHIPPING OF SAMPLES CLASSIFIED AS FLAMMABLE LIQUID (OR SOLID)

5.4.1 Packaging

Applying the word "flammable" to a sample does not imply that it is in fact flammable. The word prescribes the class of packaging according to DOT regulations.

1. Collect sample in the prescribed container with a nonmetallic, Teflon-lined screw cap. To prevent leakage, fill container no more than 90 percent full.

2. Complete sample label and sample identification tag and attach securely to sample container.

3. Seal container and place in 2-mil thick (or thicker) polyethylene bag, one sample per bag. Position sample identification tag so that it can be read through bag. Seal bag.

4. Place sealed bag inside metal can and cushion it with enough noncombustible, absorbent material (for example, vermiculite or diatomaceous earth) between the bottom and sides of the can and bag to prevent breakage and absorb leakage. Pack one bag per can. Use clips, tape, or other positive means to hold can lid securely, tightly and permanently. Mark can as indicated in Paragraph 1 of Section 5.4.2, below.

5. Place one or more metal cans (or single 1-gallon bottle) into a strong outside container, such as a metal picnic cooler or a DOT-approved fiberboard box. Surround cans with noncombustible, absorbent cushioning materials for stability during transport. Mark container as indicated in Paragraph 2 of Section 5.4.2.

5.4.2 Marking/Labeling

1. Use abbreviations only where specified. Place the following information, either hand-printed or in label form, on the metal can (or 1-gallon bottle):

   - Laboratory name and address.

   - "Flammable Liquid, n.o.s. UN1993" or "Flammable Solid, n.o.s. UN1325."

Not otherwise specified (n.o.s) is not used if the flammable liquid (or solid) is identified. Then the name of the specific material is listed before the category (for example, Acetone, Flammable Liquid), followed by its appropriate UN number found in the DOT Hazardous Materials table (49 CFR 172.101).

2. Place all information on outside shipping container as on can (or bottle), specifically:

   - Proper shipping name.
   - UN or NA number.
   - Proper label(s).
   - Addressee and sender.

   Place the following labels on the outside shipping container: "Cargo Aircraft Only" and "Flammable Liquid" (or "Flammable Solid"). "Dangerous When Wet" label shall be used if the solid has not been exposed to a wet environment. "Laboratory Samples" and "THIS SIDE UP" or "THIS END UP" shall also be marked on the top of the outside container, and upward-pointing arrows shall be placed on all four sides of the container.

5.4.3 Shipping Papers

1. Use abbreviations only where specified. Complete the carrier-provided bill of lading and sign certification statement (if carrier does not provide, use standard industry form, see Attachment D). Provide the following information in the order listed (one form may be used for more than one exterior container).

   - "Flammable Liquid, n.o.s. UN1993" or "Flammable Solid, n.o.s. UN1325."
   - "Limited Quantity" (or "Ltd. Qty.").
   - "Cargo Aircraft Only."
   - Net weight (wt) or net volume (vol), just before or just after "Flammable Liquid, n.o.s." or "Flammable Solid, n.o.s.," by item, if more than one metal can is inside an exterior container.
   - "Laboratory Samples" (if applicable).

2. Include Chain-of-Custody Record, properly executed in outside container.

3. "Limited Quantity" of "Flammable Liquid, n.o.s." is limited to one pint per inner container. For "Flammable Solid, n.o.s.," net weight of inner container plus sample shall not exceed one pound; total package weight shall not exceed 25 pounds.

5.4.4 Transportation

1. Transport unknown hazardous substance samples classified as flammable liquids by rented or common carrier truck, railroad, or express overnight package services. Do not transport by any passenger-carrying air transport system, even if they have cargo-only aircraft. DOT regulations permit regular airline cargo-only aircraft, but difficulties with most suggest avoiding them. Instead, ship by airline carriers that only carry cargo.

2. For transport by government-owned vehicle, including aircraft, DOT regulations do not apply. However, procedures described above, with the exception of execution of the bill of lading with certification, shall still be used.

6.0 REFERENCES

U.S. Department of Transportation, 1983. Hazardous Materials Regulations, 49 CRF 171-177.

NUS Standard Operating Procedure SA-6.1 - Sample Identification and Chain-of-Custody

NUS Standard Operating Procedure SA-1.2 - Sample Preservation

NUS Standard Operating Procedure SF-1.5 - Compatibility Testing

7.0 ATTACHMENTS

Attachment A - DOT Hazardous Material Classification (49 CFR 173.2)
Attachment B - DOT List of Class "A" Poisons (49 CFR 172.101)
Attachment C - Hazardous Materials Shipping Checklist
Attachment D - Standard Industry Certification Form

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1.0 PURPOSE

This procedure describes the process for keeping a site logbook.

2.0 SCOPE

The site logbook is a controlled document which records all major on-site activities during a Remedial Investigation/Feasibility Study. At a minimum, the following activities/events shall be recorded in the site logbook:

- Arrival/departure of site visitors
- Arrival/departure of equipment
- Sample pickup (chain-of-custody form numbers, carrier, time)
- Sampling activities/sample logsheet numbers
- Start or completion of borehole/trench/monitoring well installation or sampling activities
- Health and Safety issues

The site logbook is initiated at the start of the first on-site activity (e.g., initial reconnaissance survey). Entries are made for every day that on-site activities take place which involve RI/FS contractor personnel. One current site logbook is maintained per site.

The site logbook becomes part of the permanent site file maintained in the RI contractor's office. Because information contained in the site logbook may be admitted as evidence in cost recovery or other legal proceedings, it is critical that this document be properly maintained.

3.0 GLOSSARY

Site Logbook - The logbook is a bound notebook with consecutively numbered pages that cannot be removed. Upon entry of data, the logbook requires signature by the responsible site leader (see Section 5.1).

4.0 RESPONSIBILITIES

The site logbook is issued by the Regional Manager (or his designee) to the Site Manager for the duration of the project. The Site Manager releases the site logbook to the Field Operations Leader or other person responsible for the direction of on-site activities (e.g., Reconnaissance Survey Team Leader, Sampling Team Leader). It is the responsibility of this person (or his designee) to keep the site logbook current while in his possession, and return it to the Site Manager or turn it over to another field team. Following the completion of all fieldwork, the site logbook is returned to the Site Manager for inclusion in the permanent site files.

5.0 PROCEDURES

5.1 GENERAL

The cover of each site logbook contains the following information:

- Project Name
- NUS Project Number
- RI/FS Contractor and Site Manager's Name
- Sequential Book Number

- Start Date
- End Date

Daily entries into the logbook may contain a variety of information. At the beginning of each day the following information must be recorded:

- Date
- Start time
- Weather
- All field personnel present
- Any visitors present

During the day, a summary of all site activities and level of personal protection shall be recorded in the logbook. The information need not duplicate that recorded in other field notebooks (e.g., sample logbook, Site Geologist's notebook, Health and Safety Officer's notebook, etc.), but shall summarize the contents of these other notebooks and refer to the page locations in these notebooks for detailed information. An example of a site logbook page is shown in Attachment A.

The sample logsheet for each sample collected (see Procedure SA-6.6) must be referenced. If measurements are made at any location, the measurements and equipment used must either be recorded in the site logbook or reference must be made to the notebook and page number(s) on which they are recorded (see Attachment A).

All entries shall be made in black pen. No erasures are permitted. If an incorrect entry is made, the data shall be crossed out with a single strike mark, and initialed and dated. At the completion of entries by any individual, the logbook must be signed. It must also be signed by the Field Operations Leader or responsible site leader at the end of each day.

5.2 PHOTOGRAPHS

When movies, slides, or photographs are taken of a site or any monitoring location, they are numbered to correspond to logbook entries. The name of the photographer, date, time, site location, site description, and weather conditions are entered in the logbook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook. If possible, such techniques shall be avoided, since they can adversely affect the admissibility of photographs as evidence. Chain-of-custody procedures depend upon the subject matter, type of film, and the processing it requires. Film used for aerial photography, confidential information, or criminal investigation require chain-of-custody procedures. Adequate logbook notation and receipts may be used to account for routine film processing. Once processed, the slides of photographic prints shall be serially numbered and labeled according to the logbook descriptions.

6.0 REFERENCES

None.

7.0 ATTACHMENTS

Attachment A - Typical Site Logbook Entry

ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: _____ DATE: _____

SITE LEADER: _____
PERSONNEL:

NUS	DRILLER	EPA
_____	_____	_____
_____	_____	_____
_____	_____	_____

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenney and fire hoses were set up.

2. Drilling activities at well _____ resumes. Rig geologist was _____. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4 inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well _____.

3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well _____.

4. Well _____ drilled. Rig geologist was _____. See Geologist's Notebook, No. 2, page _____ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.

5. Well _____ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."

6. EPA remedial project manger arrives on-site at 14:25 hours.

7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit _____.

8. Test pit _____ dug with cuttings placed in dump truck. Rig geologist was _____. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit _____ resulted in a very soft and wet area. A mound was developed and the area roped off.

9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel offsite, gate locked.

_____
Field Operations Leader

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1.0 PURPOSE

This procedure describes the procedures and equipment required to measure the following parameters of an aqueous sample in the field:

- pH
- Specific Conductance
- Temperature
- Dissolved Oxygen (DO) Concentration
- Oxidation Reduction Potential
- Certain Dissolved Constituents Using Specific Ion Elements

2.0 SCOPE

This procedure is applicable for use in an on-site groundwater quality monitoring program to be conducted during a remedial investigation or site investigation program at a hazardous or non-hazardous site. The procedures and equipment described are applicable to nearly all aqueous samples, including potable well water, monitoring well water, surface water, leachate and drummed water, etc. and are not, in general, subject to solution interferences from color, turbidity and colloidal material, or suspended matter.

This procedure provides generic information for measuring the parameters listed above with instruments and techniques in common use. Since instruments from different manufacturers may vary, review of the manufacturer's literature pertaining to the use of a specific instrument is required before use.

3.0 GLOSSARY

3.1 pH MEASUREMENT

pH - The negative logarithm (base 10) of the hydrogen ion activity. The hydrogen ion activity is related to the hydrogen ion concentration, and, in relatively weak solution, the two are nearly equal. Thus, for all practical purposes, pH is a measure of the hydrogen ion concentration.

pH Paper - Paper that turns different colors depending on the pH of the solution to which it is exposed. Comparison with color standards supplied by the manufacturer will then give an indication of the solution pH.

3.2 SPECIFIC CONDUCTANCE MEASUREMENT

Ohm - Standard unit of electrical resistance (R). A siemen (or umho) is the standard unit of electrical conductance, the inverse of the ohm.

Resistance - A measure of the solution's ability to oppose the passage of electrical current. For metals and solutions, resistance is defined by Ohm's law, $E = IR$, where E is the potential difference, I is the current, and R is the resistance.

Conductance - The conductance of a conductor 1 centimeter long and 1 square centimeter in cross-sectional area. Conductivity and specific conductance are used synonymously.

3.3 TEMPERATURE MEASUREMENT

None.

3.4 DISSOLVED OXYGEN MEASUREMENT

Galvanic Cell - An electrochemical cell in which chemical energy is spontaneously converted to electrical energy. The electrical energy produced is supplied to an external circuit.

Electrolytic Cell - An electrochemical cell in which electrical energy is supplied from an external source. This cell functions in much the same way as a galvanic cell, only in the opposite direction due to the external source of applied voltage.

3.5 OXIDATION-REDUCTION POTENTIAL MEASUREMENT

Oxidation - The process in which an atom or group of atoms loses electrons to achieve an increasing positive charge.

Reduction - The gaining of electrons by an atom or group of atoms and subsequent increase in negative charge.

Oxidation-Reduction Potential (ORP) - A measure of the activity ratio of oxidizing and reducing species as determined by the electromotive force developed by a noble metal electrode, immersed in water, as referenced against a standard hydrogen electrode.

3.6 SPECIFIC ION ELECTRODES MEASUREMENT

Specific Ion Electrode - An electrode which develops a potential difference across a membrane in response to the concentration differences for selected ions on either side of that membrane.

4.0 RESPONSIBILITIES

Site Manager - in consultation with the Project Geochemist, is responsible for determining which on-site water quality measurements can contribute to the RI, when these measurements shall be made, and the data quality objectives (DQOs) for these measurements. The Project Operations Plan (POP) shall contain details of type, frequency and locations of the desired measurements.

Project Geochemist - primarily responsible for determining the type, frequency and locations for on-site water quality measurements as presented in the POP and for interpreting the results, including determination of which measurements are unrepresentative.

Field Operations Leader - responsible for implementing the POP, and also for deciding under what field conditions a particular on-site measurement will be unrepresentative or unobtainable.

Field Samplers/Analysts - responsible for the actual analyses that take place, including calibration, quality control and recording of results, as well as for the care and maintenance of the equipment in the field.

5.0 GUIDELINES

5.1 MEASUREMENT OF pH

5.1.1 General

Measurement of pH is one of the most important and frequently used tests in water chemistry. Practically every phase of water supply and wastewater treatment such as acid-base neutralization, water softening, and corrosion control, is pH dependent. Likewise, the pH of leachate can be correlated with other chemical analyses to determine the probable source of contamination. It is therefore important that reasonably accurate pH measurements be taken.

Measurements of pH can also be used to check the quality and corrosivity of soil and solid waste samples. However, these samples must be immersed in water prior to analysis, and specific techniques are not described.

Two methods are given for pH measurement: the pH meter and pH indicator paper. The indicator paper is used when only a rough estimate of the pH is required, and the pH meter when a more accurate measurement is needed. The response of a pH meter can be affected to a slight degree by high levels of colloidal or suspended solids, but the effect is usually small and generally of little significance. Consequently, specific methods to overcome this interference are not described. The response of pH paper is unaffected by solution interferences from color, turbidity, colloidal or suspended materials unless extremely high levels capable of coating or masking the paper are encountered. In such cases, use of a pH meter is recommended.

5.1.2 Principles of Equipment Operation

Use of pH papers for pH measurement relies on a chemical reaction caused by the acidity or basicity of the solution with the indicator compound on the paper. Depending on the indicator and the pH range of interest, a variety of different colors can be used. Typical indicators are weak acids or bases, or both. Process chemistry and molecular transformations leading to the color change are variable and complex.

Use of a pH meter relies on the same principle as other ion-specific electrodes. Measurement relies on establishment of a potential difference across a glass or other type of membrane in response to hydrogen ion concentration across that membrane. The membrane is conductive to ionic species and, in combination with a standard or reference electrode, a potential difference proportional to hydrogen ion concentration can be generated and measured.

5.1.3 Equipment

The following equipment is needed for taking pH measurements:

- Accumet 150 portable pH meter, or equivalent.
- Combination electrode with polymer body to fit the above meter (alternately a pH electrode and a reference electrode can be used if the pH meter is equipped with suitable electrode inputs.
- pH indicator paper, such as Hydrion or Alkacid, to cover the pH range 2 through 12.
- Buffer solutions of pH 4, 7 and 10, or other buffers which bracket the expected pH range.

5.1.4 Measurement Techniques for Field Determination of pH

1. pH Meter

The following procedure is used for measuring pH with a pH meter (Standardization is according to manufacturers instructions):

a. The instrument and batteries shall be checked and calibrated prior to initiation of the field effort.

b. The accuracy of the buffer solutions used for field and laboratory calibration shall be checked. Buffer solutions need to be changed often due to degradation upon exposure to the atmosphere.

c. Immerse the tip of the electrodes in water overnight. If this is not possible due to field conditions, immerse the electrode tip in water for at least an hour before use. The electrode tip may be immersed in a rubber or plastic sack containing buffer solution for field transport or storage. This is not applicable for all electrodes as some must be stored dry.

d. Make sure all electrolyte solutions within the electrode(s) are at their proper levels and that no air bubbles are present within the electrode(s).

e. Immerse the electrode(s) in a pH-7 buffer solution.

f. Adjust the temperature compensator to the proper temperature (on models with automatic temperature adjustment, immerse the temperature probe into the buffer solution). Alternately, the buffer solution may be immersed in the sample and allowed to reach temperature equilibrium before equipment calibration. It is best to maintain buffer solution at or near expected sample temperature before calibration.

g. Adjust the pH meter to read 7.0.

h. Remove the electrode(s) from the buffer and rinse well with demineralized water. Immerse the electrode(s) in pH-4 or 10 buffer solution (depending on the expected pH of the sample) and adjust the slope control to read the appropriate pH. For best results, the standardization and slope adjustments shall be repeated at least once.

i. Immerse the electrode(s) in the unknown solution, slowly stirring the probe until the pH stabilizes. Stabilization may take several seconds to minutes. If the pH continues to drift, the sample temperature may not be stable, a chemical reaction (e.g., degassing) may be taking place in the sample, or the meter or electrode may be malfunctioning. This must be clearly noted in the logbook.

j. Read and record the pH of the solution, after adjusting the temperature compensator to the sample temperature. pH shall be recorded to the nearest 0.1 pH unit. Also record the sample temperature.

k. Rinse the electrode(s) with deionized water.

l. Keep the electrode(s) immersed in deionized water when not in use.

The sample used for pH measurement shall never be saved for subsequent conductivity or chemical analysis. All pH electrodes leak small quantities of electrolytes (e.g., sodium or potassium chloride) into the solution. Precipitation of saturated electrolyte solution, especially at colder temperatures, or in cold water, may result in slow electrode response. Any visual observation of conditions which may interfere with pH measurement, such as oily materials, or turbidity, shall be noted.

2. pH Paper

Use of pH paper is very simple and requires no sample preparation, standardization, etc. pH paper is available in several ranges, including wide-range (indicating approximately pH 1 to 12), mid-range (approximately pH 0 to 6, 6 to 9, 8 to 14) and narrow-range (many available, with ranges as narrow as 1.5 pH units). The appropriate range of pH paper shall be selected. If the pH is unknown the investigation shall start with wide-range paper.

5.2 MEASUREMENT OF SPECIFIC CONDUCTANCE

5.2.1 General

Conductance provides a measure of dissolved ionic species in water and can be used to identify the direction and extent of migration of contaminants in groundwater or surface water. It can also be used as a measure of subsurface biodegradation or to indicate alternate sources of groundwater contamination.

Conductivity is a numerical expression of the ability of a water sample to carry an electric current. This value depends on the total concentration of the ionized substances dissolved in the water and the temperature at which the measurement is made. The mobility of each of the various dissolved ions, their valences, and their actual and relative concentrations affect conductivity.

It is important to obtain a specific conductance measurement soon after taking a sample, since temperature changes, precipitation reactions, and absorption of carbon dioxide from the air all affect the specific conductance.

5.2.2 Principles of Equipment Operation

An aqueous system containing ions will conduct an electric current. In a direct-current field, the positive ions migrate toward the negative electrode, while the negatively charged ions migrate toward the positive electrode. Most inorganic acids, bases and salts (such as hydrochloric acid, sodium carbonate, or sodium chloride, respectively) are relatively good conductors. Conversely, organic compounds such as sucrose or benzene, which do not disassociate in aqueous solution, conduct a current very poorly, if at all.

A conductance cell and a Wheatstone Bridge (for the measurement of potential difference) may be used for measurement of electrical resistance. The ratio of current applied to voltage across the cell may also be used as a measure of conductance. The core element of the apparatus is the conductivity cell containing the solution of interest. Depending on ionic strength of the aqueous solution to be tested, a potential difference is developed across the cell which can be converted directly or indirectly (depending on instrument type) to a measurement of specific conductance.

5.2.3 Equipment

The following equipment is needed for taking specific conductance measurements:

- YSI Model 33 portable conductivity, meter, or equivalent
- Probe for above meter

A variety of conductivity meters are available which may also be used to monitor salinity and temperatures. Probe types and cable lengths vary, so equipment may be obtained to meet the specific requirement of the sampling program.

5.2.4 Measurement Techniques for Specific Conductance

The steps involved in taking specific conductance measurements are listed below (standardization is according to manufacturers instructions):

- Check batteries and calibrate instrument before going into the field.
- Calibrate the instrument daily when used. Potassium chloride solutions with a specific conductance closest to the values expected in the field shall be used. Attachment A may be used for guidance.
- Rinse the cell with one or more portions of the sample to be tested or with deionized water.
- Immerse the electrode in the sample and measure the conductivity. Adjust the temperature setting to the sample temperature.
- Read and record the results in a field logbook or sample log sheet.

If the specific conductance measurements become erratic, or inspection shows that any platinum black has flaked off the electrode, replatinization of the electrode is necessary. See the manufacturer's instructions for details.

Note that specific conductance is occasionally reported at temperatures other than ambient.

5.3 MEASUREMENT OF TEMPERATURE

5.3.1 General

In combination with other parameters, temperature can be a useful indicator of the likelihood of biological action in a water sample. It can also be used to trace the flow direction of contaminated groundwater. Temperature measurements shall be taken in-situ, or as quickly as possible in the field. Collected water samples may rapidly equilibrate with the temperature of their surroundings.

5.3.2 Equipment

Temperature measurements may be taken with alcohol-toluene, mercury filled or dial-type thermometers. In addition, various meters such as specific conductance or dissolved oxygen meters, which have temperature measurement capabilities, may also be used. Using such instrumentation along with suitable probes and cables, in-situ measurements of temperature at great depths can be performed.

5.3.3 Measurement Techniques for Water Temperature

If a thermometer is used on a collected water sample:

- Immerse the thermometer in the sample until temperature equilibrium is obtained (1-3 minutes). To avoid the possibility of contamination, the thermometer shall not be inserted into samples which will undergo subsequent chemical analysis.
- Record values in a field logbook or sample log sheet.

If a temperature meter or probe is to be used, the instrument shall be calibrated according to manufacturer's recommendations with an approved thermometer before each measurement or group of closely spaced measurements.

5.4 MEASUREMENT OF DISSOLVED OXYGEN CONCENTRATION

5.4.1 General

Dissolved oxygen (DO) levels in natural water and wastewater depend on the physical, chemical and biochemical activities in the water body. Conversely, the growth of many aquatic organisms as well as the rate of corrosivity, are dependent on the dissolved oxygen concentration. Thus, analysis for dissolved oxygen is a key test in water pollution and waste treatment process control. If at all possible, DO measurements shall be taken in-situ, since concentration may show a large change in a short time if the sample is not adequately preserved.

The method monitoring discussed herein is limited to the use of dissolved oxygen meters only. Chemical methods of analysis (i.e., Winkler methods) are available, but require more equipment and greater sample manipulation. Furthermore, DO meters, using a membrane electrode, are suitable for highly polluted waters, because the probe is completely submersible, and are free from interference caused by color, turbidity, colloidal material or suspended matter.

5.4.2 Principles of Equipment Operation

Dissolved oxygen probes are normally electrochemical cells that have two solid metal electrodes of different nobility immersed in an electrolyte. The electrolyte is retained by an oxygen-permeable membrane. The metal of highest nobility (the cathode) is positioned at the membrane. When a suitable potential exists between the two metals, reduction of oxygen to hydroxide ion (OH) occurs at the cathode surface. An electrical current is developed that is directly proportional to the rate of arrival of oxygen molecules at the cathode.

Since the current produced in the probe is directly proportional to the rate of arrival of oxygen at the cathode, it is important that a fresh supply of sample always be in contact with the membrane. Otherwise, the oxygen in the aqueous layer along the membrane is quickly depleted and false low readings are obtained. It is therefore necessary to stir the sample (or the probe) constantly to maintain fresh solution near the membrane interface. Stirring, however, shall not be so vigorous that additional oxygen is introduced through the air-water interface at the sample surface. To avoid this possibility, some probes are equipped with stirrers to agitate the solution near the probe, but to leave the surface of the solution undisturbed.

Dissolved oxygen probes are relatively free of interferences. Interferences that can occur are reactions with oxidizing gases (such as chlorine) or with gases such as hydrogen sulfide which are not

easily depolarized from the indicating electrode. If the gaseous interference is suspected, it shall be noted in the field log book and checked if possible. Temperature variations can also cause interference because probes exhibit temperature sensitivity. Automatic temperature compensation is normally provided by the manufacturer.

5.4.3 Equipment

The following equipment is needed to measure dissolved oxygen concentration:

- YSI Model 56 dissolved oxygen monitor or equivalent.
- Dissolved oxygen/temperature probe for above monitor.
- Sufficient cable to allow the probe to contact the sample.

5.4.4 Measurement Techniques for Dissolved Oxygen Determination

Probes differ as to specifics of use. Follow the manufacturer's instructions to obtain an accurate reading. The following general steps shall be used to measure the dissolved oxygen concentration:

- The equipment shall be calibrated and have its batteries checked in the laboratory before going to the field.

- The probe shall be conditioned in a water sample for as long a period as practical before use in the field. Long periods of dry storage followed by short periods of use in the field may result in inaccurate readings.

- The instrument shall be calibrated in the field before each measurement or group of closely spaced measurements by placing the probe in a water sample of known dissolved oxygen concentration (i.e., determined by Winkler method) or in a freshly air-saturated water sample of known temperature. Dissolved oxygen values for air-saturated water can be determined by consulting a table listing oxygen solubilities as a function of temperature and salinity (see Attachment B).

- Immerse the probe in the sample. Be sure to provide for sufficient flow past the membrane, either by stirring the sample, or placing the probe in a flowing stream. Probes without stirrers placed in wells can be moved up and down.

- Record the dissolved oxygen content and temperature of the sample in a field logbook or sample log sheet.

- Recalibrate the probe when the membrane is replaced, or as needed. Follow the manufacturer's instructions.

Note that in-situ placement of the probe is preferable, since sample handling is not involved. This however, may not always be practical. Be sure to record whether the liquid was analyzed in-situ, or if a sample was taken.

Special care shall be taken during sample collection to avoid turbulence which can lead to increased oxygen solubilization and positive test interferences.

5.5 MEASUREMENT OF OXIDATION-REDUCTION POTENTIAL

5.5.1 General

The oxidation-reduction potential (ORP) provides a measure of the tendence of organic or inorganic compounds to exist in an oxidized state. The technique therefore provides evidence of the likelihood of anaerobic degradation of biodegradable organics or the ratio of activities of oxidized to reduced species in the sample.

5.5.2 Principles of Equipment Operation

When an inert metal electrode, such as platinum, is immersed in a solution, a potential is developed at that electrode depending on the ions present in the solution. If a reference electrode is placed in the same solution, an ORP electrode pair is established. This electrode pair allows the potential difference between the two electrodes to be measured and will be dependent on the concentration of the ions in solution. By this measurement, the ability to oxidize or reduce species in solution may be determined. Supplemental measurements, such as dissolved oxygen, may be correlated with ORP to provide a knowledge of the quality of the solution, water, or wastewater.

5.5.3 Equipment

The following equipment is needed for measuring the oxidation-reduction potential of a solution:

- Accumet 150 portable pH meter or equivalent, with a millivolt scale.
- Platinum electrode to fit above pH meter.
- Reference electrode such as a calomel, silver-silver chloride, or equivalent.

5.5.4 Measurement Techniques for Oxidation-Reduction Potential

The following procedure is used for measuring oxidation-reduction potential:

- The equipment shall be calibrated and have its batteries checked before going to the field.

- Check that the platinum probe is clean and that the platinum bond or tip is unoxidized. If dirty, polish with emery paper or, if necessary, clean the electrode using aqua regia, nitric acid, or chromic acid, in accordance with manufacturer's instructions.

- Thoroughly rinse the electrode with demineralized water.

- Verify the sensitivity of the electrodes by noting the change in millivolt reading when the pH of the test solution is altered. The ORP will increase when the pH of the test solution decreases and the ORP will decrease if the test solution pH is increased. Place the sample in a clean glass beaker and agitate the sample. Insert the electrodes and note the ORP drops sharply when the caustic is added, the electrodes are sensitive and operating properly. If the ORP increases sharply when the caustic is added, the polarity is reversed and must be corrected in accordance with the manufacturer's instructions. If the ORP does not respond as above when the caustic is added, the electrodes shall be cleaned and the above procedure repeated.

- After the assembly has been checked for sensitivity, wash the electrodes with three changes of water or by means of a flowing stream of water from a wash bottle. Place the sample in a clean glass beaker or sample cup and insert the electrodes. Set temperature

compensator throughout the measurement period. Read the millivolt potential of the solution, allowing sufficient time for the system to stabilize and reach temperature equilibrium. Measure successive portions of the sample until readings on two successive portions differ by no more than 10 mV. A system that is very slow to stabilize properly will not yield a meaningful ORP. Record all results in a field logbook, including ORP (to nearest 10 mV), sample temperature and pH at the time of measurement.

5.6 SPECIFIC ION ELECTRODE MEASUREMENTS

5.6.1 General

Use of specific ion electrodes can be beneficial in the field for determining the presence and concentration of dissolved inorganic species which may be associated with contaminant plumes or leachate. Thus, electrodes can be used for rapid screening of water quality and determination of water migration pathways.

This procedure provides generic information for specific ion electrodes commonly used in groundwater quality monitoring programs and describes the essential elements of a field investigation program. Analytical methods using some specific ion electrodes have not been approved by the USEPA. In addition, calibration procedures and solutions, interferences and conditions and requirements for use for various electrodes vary greatly. Consequently, review of manufacturer's literature is mandatory prior to use.

5.6.2 Principles of Equipment Operation

All specific ion electrode measurements involve the use of a reference electrode, a pH meter, and a specific ion electrode (SIE). When the SIE and the reference electrode are immersed in a solution of the ion to be measured, a potential difference is developed between the two electrodes. This potential can be measured by a pH meter and related to the concentration of the ion of interest through the use of standard solutions and calibration curves.

Several different types of SIEs are in use: glass, solid-state, liquid-liquid membrane, and gas-sensing. All of the electrodes function using an ion exchange process as the potential determining mechanism. Glass electrodes are used for pH measurement. The glass in the tip of the electrode actually acts as a semi-permeable membrane to allow solution. Solid-state electrodes replace the glass membrane with an ionically-conducting membrane, (but act in essentially the same manner) while liquid-liquid membrane electrodes have an organic liquid ion exchanger contained in the pores of a hydrophobic membrane. Maintenance of the conducting interface, in combination with a reference electrode, allows completion of the electrical circuit and subsequent measurement of the potential difference. Gas-sensing electrodes have a membrane that permits the passage of gas only, thus allowing for the measurement of gas concentration. Regardless of the mechanism involved in the electrode, most SIEs are easy to use under field conditions. The sensitivity and applicable concentration range for various membranes and electrodes will vary.

5.6.3 Equipment

The following equipment is required for performing quantitative analyses using a specific ion electrode:

- A pH meter with a millivolt scale, or equivalent.
- The specific ion electrode for the parameter to be measured. A partial list of ions which can be measured includes cyanide, sulfide, ammonia, lead, fluoride and chloride.
- A suitable reference electrode to go with the above SIE.

Specific electrodes for other ions have also been developed, but are not widely used for field investigation efforts at this time. Note that of the specific electrodes referenced above, only fluoride and ammonia have analytical methods approved by the U.S. EPA.

5.6.4 Measurement Techniques for Inorganic Ions Using Specific Ion Electrodes

Different types of electrodes are used in slightly different ways and are applicable for different concentration ranges. Following the manufacturer's instructions, the general steps given below are usually followed:

- Immerse the electrode in water for a suitable period of time prior to sample analysis.

- Standardize the electrode according to the manufacturer's instructions, including necessary chemical additions for ionic strength adjustment, etc. Standard solutions normally differ by factors of ten in concentration. Constant stirring is needed for accurate readings.

- Immerse the electrode in the sample. Allow the reading to stabilize and record the results in a site logbook. Stir the sample at the same rate as the standards. Air bubbles near the membrane shall be avoided, since this may cause interference in millivolt readings.

(NOTE: Each SIE has substances which interfere with proper measurement. These may be eliminated using pretreatment methods as detailed by the manufacturer. It is important to know if interferences are present so that suspect readings may be noted as such.)

- If the pH meter does not read out directly, plot millivolts versus concentration for the standards and then determine sample concentration.

6.0 REFERENCES

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

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

U.S. Geological Survey, 1984. National Handbook of Recommended Methods for Water Data Acquisition, Chapter 5: Chemical and Physical Quality of Water and Sediment. U.S. Department of the Interior, Reston, Virginia.

7.0 ATTACHMENTS

Attachment A - Specific Conductance of KC1 Solutions at 25 degrees Centigrade

Attachment B - Variation of Dissolved Oxygen Concentration in Water as a a Function of Temperature and Salinity.

ATTACHMENT A

SPECIFIC CONDUCTANCE OF M KCl AT VARIOUS TEMPERATURES[1]

Temperature (°C)	Specific Conductance (µmhos/cm)
15	1,147
16	1,173
17	1,199
18	1,225
19	1,251
20	1,278
21	1,305
22	1,332
23	1,359
24	1,368
25	1,413
26	1,441
27	1,468
28	1,496
29	1,524
30	1,552

1 Data derived from the International Critical Tables 1-3-8.

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1.0 PURPOSE

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

2.0 SCOPE

Different types of chemicals react differently with sample containers made of various materials. For example, trace metals adsorb more strongly to glass than to plastic, while many organic chemicals may dissolve various types of plastic containers. It is therefore critical to select the correct container in order to maintain the quality of the sample prior to analysis.

Many water and soil samples are unstable, and therefore require preservation when the time interval between field collection and laboratory analysis is long enough to produce changes in either the concentration or the physical condition of the constituent(s) requiring analysis. While complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological changes that inevitably take place after the sample is collected.

Preservation techniques are usually limited to pH control, chemical addition(s) and refrigeration/ freezing. Their purpose is to (1) retard biological activity, (2) retard hydrolysis of chemical compounds/complexes, (3) reduce constituent volatility, and (4) reduce adsorption effects.

3.0 GLOSSARY

HCl - Hydrochloric Acid
H_2SO_4 - Sulfuric Acid
HNO_3 - Nitric Acid
NaOH - Sodium Hydroxide

Normality (N) - Concentration of a solution expressed as equivalent per liter, an equivalent being the amount of a substance containing one gram-atom of replaceable hydrogen or its equivalent. Thus, a one molar solution of HCl, containing one gram-atom of H, is "one-normal," while a one molar solution of H_2SO_4 containing two gram-atoms of H, is "two-normal."

4.0 RESPONSIBILITIES

Field Operations Leader - retains overall responsibility for the proper storage and preservation of samples. During the actual collection of samples, the sampling technician(s) will be directly responsible for the bottling, preservation, labeling, and custody of the samples they collect until released to another party for storage or transport to the analytical laboratory.

5.0 PROCEDURES

5.1 SAMPLE CONTAINERS

For most samples and analytical parameters either glass or plastic containers are satisfactory. In general, if the analyte(s) to be determined is organic in nature, the container shall be made of glass. If the analyte(s) is inorganic, then the container shall be plastic. Since container specification will depend on the analyte and sample matrix types (as indicated in Attachment A) duplicate samples shall be taken when both organic and inorganic analyses are required. Containers shall be kept in the

dark (to minimize biological or photooxidation/photolysis breakdown of constituent) until they reach the analytical laboratory. The sample container shall allow approximately 5-10 percent air space ("ullage") to allow for expansion/vaporization if the sample is heated during transport (1 liter of water at 4°C expands by 15 ml if heated to 130°F/55°C), however, head space for volatile organic analyses shall be omitted.

For CLP laboratories, containers will be obtained through the CLP Sample Management Office. For Responsible party actions or non-CLP laboratories, the laboratory shall provide containers that have been cleaned according to U.S. EPA procedures. Sufficient lead time shall be allowed. Shipping containers for samples, consisting of sturdy ice chests, are provided by the laboratory of the remedial investigation contractor.

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

General sample container and sample volume requirements are listed in Attachment A. Specific container requirements are listed in Attachment B.

5.2 PRESERVATION TECHNIQUES

The preservation techniques to be used for various analytes are listed in Attachments A and B. Reagents required for sample preservation will either be added to the sample containers by the laboratory prior to their shipment to the Field or added in the Field. In general, aqueous samples of low concentration organics (or soil samples of low or medium concentration organics) are cooled to 4°C. Medium concentration aqueous samples and high hazard organics sample are not preserved. Low concentration aqueous samples for metals are acidified with HNO_3, while medium concentration and high hazard aqueous metal samples are not preserved. Low or medium concentration soil samples for metals are cooled to 4°C while high hazard samples are not preserved.

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

5.2.1 Addition of Acid (H_2SO_4, HCl, or HNO_3) or Base

Addition of the following acids or bases may be specified for sample preservation; these reagents shall be analytical reagent (AR) grade and shall be diluted to the required concentration with double-distilled, deionized water in the laboratory, before Field sampling commences:

Acid Base	Concentration	Normality	Amount for Acidification*
HCl	1:1 dilution of concentrated HCl	6N	5-10 ml
H_2SO_4	1:1 dilution of concentrated H_2SO_4	18N	2-5 ml
HNO_3	Undiluted concentrated HNO_3	16N	2-5 ml
NaOH	400 grams solid NaOH in 870 ml water	10N	2 ml**

* Amount of acid to add (at the specified strength) per liter of water to reduce the sample pH to less than 2, assuming that the water is initially at pH 7, and is poorly buffered and does not contain particulate matter.

** To raise pH of 1 liter of water to 12.

The approximate volumes needed to acidify one liter of neutral water to a pH of less than 2 (or raise the pH to 12) are shown in the last column of the above table. These volumes are only approximate; if the water is more alkaline, contains inorganic or organic buffers, or contains suspended particles, more acid may be required. The final pH must be checked using narrow-range pH paper.

Sample acidification or base addition shall proceed as follows:

- Check initial pH of sample with wide range (0-14) pH paper.
- Fill sample bottle to within 5-10 ml of final desired volume and add about 1/2 of estimated acid or base required, stir gently and check pH with medium range pH paper (pH 0-6 or pH 7.5-14, respectively).
- Add acid or base a few drops at a time while stirring gently. Check for final pH using narrow range (0-2.5 or 11-13, respectively) pH paper; when desired pH is reached, cap sample bottle and seal.

  Never dip pH paper into the sample; apply a drop of sample to the pH paper using the stirring rod.

5.2.2 Cyanide Preservation

Pre-sample preservation is required if oxidizing agents such as chlorine are suspected to be present. To test for oxidizing agents, place a drop of the sample on KI-starch paper; a blue color indicates the need for treatment. Add ascorbic acid to the sample, a few crystals at a time, until a drop of sample produces no color on the KI-starch paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume. Add NaOH solution to raise pH to greater than 12 as described in 5.2.1. If oxidizing agents are not suspected, add NaOH as directed.

5.2.3 Sulfide Preservation

Samples for sulfide analysis must be preserved by addition of 4 drops (0.2 ml) of 2N zinc acetate solution per 100 ml sample. The sample pH is then raised to 9 using NaOH. The 2N zinc acetate solution is made by dissolving 220 g of zinc acetate in 870 ml of distilled water to make 1 liter of solution.

5.2.4 Preservation of Organic Samples Containing Residual Chlorine

Some organic samples containing residual chlorine must be treated to remove this chlorine upon collection (See Attachment A). Test the samples for residual chlorine using EPA methods 330.4 or 330.5 (Field Test Kits are available for this purpose). If residual chlorine is present, add 0.008% sodium thiosulfate (80 mg per liter of sample).

5.2.5 Field Filtration

When the objective is to determine concentration of dissolved inorganic constituents in a water system, the sample must be filtered through a non-metallic 0.45 micron membrane filter immediately after collection. A filtration system is recommended if large quantities of samples must be filtered in the field. The filtration system shall consist of a Büchner funnel inserted into a single-hole rubber stopper, sized to form a seal when inserted into the top of a vacuum filter flask equipped with a single side arm. Heavy-wall Tygon tubing shall be attached to the single side arm of the vacuum filter flask and the suction port of a vacuum pump. The stem of the Büchner funnel shall extend below the level of the side arm of the vacuum filter flask to prevent any solvent from entering the tubing leading to the vacuum pump. Before filtration, the filter paper, which shall be of a size to lay flat on the funnel plate, shall be wetted with the solvent in order to "seal" it to the funnel. Slowly pour the solvent into the funnel and monitor the amount of solvent entering the vacuum filter flask. When the rate of solvent entering the flask is reduced to intermittent dripping and the added aliquot of solvent in the funnel has passed through the filter, the used filter paper shall be replaced with new filter paper. If the solvent contains a high percentage of suspended solids, a coarser-sized nonmetallic membrane filter may be used prior to usage of the 0.45 micron membrane filter. This "prefiltering" step may be necessary to expedite the filtration procedure. Discard the first 20 to 50 ml of filtrate from each sample to rinse the filter and filtration apparatus to minimize the risk of altering the composition of the samples by the filtering operation. For analysis of dissolved metals, the filtrate is collected in a suitable bottle (see Section 5.1) and is immediately acidified to pH 2.0 or less with nitric acid whose purity is consistent with the measurement to be made. Inorganic anionic constituents may be determined using a portion of the filtrate that has not been acidified.

Samples used for determining temperature, dissolved oxygen, Eh, and pH should not be filtered. Do not use vacuum filtering prior to determining carbonate and bicarbonate concentration because it removes dissolved carbon dioxide and exposes the sample to the atmosphere. Pressure filtration can be done using water pressure from the well. If gas pressure is required, use an inert gas such as argon or nitrogen.

Do not filter samples for analysis of volatile organic compounds. If samples are to be filtered for analyzing other dissolved organic constituents, use a glass-fiber or metal-membrane filter and collect the samples in a suitable container (see Section 5.1). Because most organic analyses require extraction of the entire sample, do not discard any of it. After filtering, the membrane containing the suspended fraction can be sealed in a glass container and analyzed separately as soon as practicable. Total recoverable inorganic constituents may be determined using a second, unfiltered sample collected at the same time as the sample for dissolved constituents.

6.0 REFERENCES

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

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

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

7.0 ATTACHMENTS

Attachment A - General Sample Container and Preservation Requirements CERCLA/RCRA Samples

Attachment B - Required Containers, Preservation Techniques, and Holding Times (3 sheets)

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1.0 PURPOSE

The purpose of these procedures is to provide a general methodology, protocol, and reference information on the proper decontamination procedures to be used on chemical sampling and field analytical equipment.

2.0 SCOPE

This procedure addresses chemical sampling and field analytical equipment only, and should be consulted when equipment decontamination procedures are being developed as part of project-specific plans.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Site Manager - responsible for ensuring that project-specific plans and the implementation of field investigations are in compliance with these guidelines.

Field Operations Leader - responsible for ensuring that decontamination procedures for all chemical sampling and field analytical equipment are programmed prior to the actual field effort and that personnel required to accomplish the task have been briefed and trained to execute the task.

5.0 PROCEDURES

In order to assure that chemical analysis results are reflective of the actual concentrations present at sampling locations, chemical sampling and field analysis equipment must be properly decontaminated prior to the field effort, during the sampling program (i.e., between sample points) and at the conclusion of the sampling program. This will minimize the potential for cross-contamination between sample points and the transfer of contamination offsite.

This procedure incorporates only those aspects of decontamination not addressed in other procedures. Specifically it incorporates those items involved in decontamination of chemical sampling and field analytical equipment.

5.1 ACCESS FOR SAMPLING

5.1.1 Bailers and Bailing Line

The potential for cross-contamination between sampling points via the use of common bailer, or its attached line, is high unless strict procedures for decontamination are followed. It is preferable, for the aforementioned reason, to dedicate an individual bailer and its line to each sample point, although this does not eliminate the need for decontamination of dedicated bailers. For non-dedicated sampling equipment, the following conditions and/or decontamination procedures should be followed.

Before the initial sampling and after each succeeding sampling point, the bailer must be decontaminated. The following steps should be followed if sampling for organic contaminants:

- Potable water rinse
- Alconox or Liquinox detergent wash
- Scrubbing of the line and bailer with a scrub brush may be required if the sample point if heavily contaminated with heavy or extremely viscous compounds
- Potable water rinse
- Rinse with 10 percent nitric acid solution*
- Deionized water rinse
- Acetone or methanol rinse
- Hexane rinse**
- Distilled/Deionized water rinse
- Air dry

If sampling for organics only, the nitric acid, acetone, methanol, and hexane rinses may be omitted. Contract-specific requirements may permit alternative procedures.

Braided nylon or polypropylene lines may be used with a bailer, however, the same line must not come in contact with the sample medium, otherwise, the line must be discarded in an approved receptacle and replaced. Prior to use, the bailer should be wrapped in aluminum foil or polyethylene sheeting.

5.1.2 Sampling Pumps

Most sampling pumps are normally low volume (less than 2 gpm) pumps. These include peristaltic, diaphragm, air-lift, pitcher and bladder pumps, to name a few. If these pumps are used for sampling from more than one sampling point, they must be decontaminated.

The procedures to be used for decontamination of sampling pumps compare to those used for a bailer except the 10 percent nitric acid solution is omitted. Each of the liquid factions is to be pumped through the system. The amount of pumping is dependent upon the size of the pump and the length of the intake and discharge hoses. Certain types of pumps are unacceptable for sampling purposes.

An additional problem is introduced when the pump relies on absorption of water via an inlet or outlet hose. For organic sampling, this hose should be Teflon. Other types of hoses leach organics into the water being sampled (especially the phthalate esters) or adsorb organics from the sampled water. For all other sampling, the hose should be Viton, polyethylene, or polyvinyl chloride (in order of preference). Whenever possible, dedicated hoses should be used.

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* Due to the leaching ability of nitric acid, on stainless steel, this step is to be omitted if a stainless steel sampling device is being used and metals analysis is required with detection limits less than approximately 50 ppb; or the sampling equipment is dedicated.

** If sampling for pesticides, PCBs, or fuels.

5.1.3 Filtering Equipment

Part of the sampling plan may incorporate the filtering of groundwater samples, and subsequent preservation. This should occur as soon after sample retrieval as possible; preferably in the field as soon as the sample is obtained. To this end, three basic filtration systems are most commonly used - the in-line disposable Teflon filter, the inert gas over-pressure filtration system, and the vacuum filtration system.

For the in-line filter, decontamination is not required since the filter cartridge is disposable, however, the cartridge must be disposed of in an approved receptacle and the intake and discharge lines must still be decontaminated.

For the over-pressure and the vacuum filtration systems, the portions of the apparatus which come in contact with the sample must be decontaminated as outlined in the paragraphs describing the decontamination of bailers. (Note: Varieties of both of these systems come equipped from the manufacturer with Teflon-lined surfaces for those that would come into contact with the sample. These filtration systems are preferred when decontamination procedures must be employed.)

5.2 FIELD ANALYTICAL EQUIPMENT

5.2.1 Water Level Indicators

Water level indicators that come into contact with groundwater must be decontaminated using the following steps:

- Rinse with potable water
- Rinse with deionized water
- Acetone or methanol rinse
- Rinse with deionized water

Water level indicators that do not come in contact with the groundwater but may encounter incidental contact during installation or retrieval need only undergo the first and last steps stated above.

5.2.2 Probes

Probes, e.g., pH or specific ion electrodes, geophysical probes, or thermometers which would come in direct contact with the sample, will be decontaminated using the procedures specified above unless manufacturer's instructions indicate otherwise; in those cases, the methods of decontamination must be clearly described in the FSAP. Probes that contact a volume of groundwater not used for laboratory analyses can be rinsed with deionized water. For probes which make no direct contact, e.g., OVA equipment, the probe will be wiped with clean paper-towels or cloth wetted with alcohol.

6.0 REFERENCES

None.

7.0 RECORDS

None.

APPENDIX C

SITE-SPECIFIC HEALTH AND SAFETY PLAN (HASP)